

# **MultiPort**

**Electrochemical Cell Kit** 

**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

(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

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# MultiPort Cell Kit Operator's Manual

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# Introduction

The Gamry Instruments MultiPort<sup>TM</sup> Cell Kit was designed for simple, reliable operation. The cell is normally used to run electrochemical tests on standard cylindrical metal specimens. You can also customize the cell for use with other sample types.

A commercial source of suitable cylindrical specimens is the P/N 410 series from Metal Samples, Inc. They have cylindrical samples available in several hundred different metals. They can be contacted at:

Metal Samples Co., Inc. P.O. Box 6 Munford, AL 36268 USA Telephone (205) 358-4202

fax (205) 358-4515

Your Corrosion Cell kit was shipped with one Metal Samples, Inc. P/N 410 Series samples. The sample material is C1018 mild steel.

NOTE: A reference electrode is <u>not</u> 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<sub>2</sub>SO<sub>4</sub>) that are suitable for use with your MultiPort kit. Your reference electrode should be ordered separately.

The MultiPort uses four of the seven 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 MultiPort cell body. When this cell is connected to a circulating water bath, accurate temperature control of your experiments becomes possible.

# Chemical Compatibility of the MultiPort Cell Kit

The components in the MultiPort Cell Kit were selected to be as chemically inert as possible. In normal use the only materials in contact with the test solution are:

- the corrosion sample,
- borosilicate glass (Pyrex® or equivalent),
- unfired Vycor®,
- PTFE (Teflon®),
- polyethylene
- ACE Glass's FETFE o-ring material

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 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.

Gamry's MultiPort 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 Vycor™ frits used in the reference bridge tube are not suitable for use with extremely basic solutions or solutions containing hydrofluoric acid. We don't know of any substitute cell components that will completely overcome this limitation. If you need to work with solutions that will damage the 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, don't forget the need to vent purge gas from the cell.

# **Unpacking and Checking Your MultiPort Cell Kit**

This section is primarily intended for the user who has just received a new MultiPort Cell Kit.

# **Checking for Shipping Damage**

Your MultiPort Cell Kit is shipped 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 MultiPort 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 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 Corrosion Cell packing list in Table 1. When shipped, all of the Corrosion Cell Kit 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 MultiPort Cell Kit Packing List

Quantity	Gamry Part Number	Description	
1	988-13	Manual, MultiPort Cell Kit	
1	930-53/930-54	Cell Bottom, (standard/jacketed w/hose barb adapters through #11 bushings)	
1	930-52	Cell Top, (incl. 3 #7 bushing w/o-ring)	
1	935-18	Cell Flange Clamp	
1	935-77	Teflon encapsulated silicone O-ring for flange	
1	930-55	Reference Electrode Bridge tube w/ Vycor Tip(incl. #11 bushing w/ o-ring)	
1	930-50	Adapter SJ28 to #7 (w/ bushing & o-ring)	
1	935-73	Clamp, SJ28 Ball Joint	
1	930-48	Gas Dispersion Tube	
1	935-3	12" Graphite Rod, Counter Electrode	
1	935-5	Adapter, 24/40 to 8 mm Tube, Teflon	
1	930-51	Tube, Cylindrical Specimen Holder, Pyrex	
1	820-51	Rod, Cylindrical Specimen Holder, S.Steel	
1	820-1	Compression Gasket, Teflon	
1	820-4	Centering Washer, Polypropylene	
1	850-5	Standoff, Hex 8-32 x 1/2", S. Steel	
1	820-5	Cylindrical Specimen, C1018 Mild Steel	
5	935-76	Polyethylene Stoppers for 24/40 Joint	
2	935-74	Polyethylene Stoppers for 24/40 Joint	
1	935-53	#11 Bushing, w/ o-Ring	
3	930-42	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 MultiPort Cell. The descriptions are based on a "standard" cell configuration consisting of a cylindrical metal sample working electrode, a graphite rod counter electrode, a single junction reference electrode in a reference bridge tube and a gas dispersion tube.

Your kit was shipped to you with one C1018 mild steel metal samples. Please use this sample to try out the assembly of your cell as you read this section of the manual.

Feel free to customize your cell configuration. 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 diagram of an assembled cell can be seen in Figure 1. The basic setup shown makes use of four of the seven available ports (five are clearly visible in the figure). A more detailed diagram of the working electrode assembly is shown in Figure 2.

If you are assembling your MultiPort Cell for the first time, you may want to assembly the entire cell dry first. Once you are comfortable with the cell's assembly, you can fill the cell with a test solution and run real tests.

Three of the ports on the cell top are 24/40 ground glass female joints, one is a 15/28 ground glass female ball joint and the other three are #7 glass thread joints. The central (24/40) port is used for the Sample Holder (the Working Electrode). The ball joint is intended for use with the included adapter and bridge tube for the reference electrode. The included gas dispersion tube (bubbler) fits either of the remaining 24/40 joints and the counter electrode is intended for one of the #7 glass thread joints.

CAUTION: <u>Do not grease</u> any of the ground glass joints on your MultiPort. This is unnecessary with the MultiPort, and may even cause problems if the grease gets into the test solution or onto the Metal Sample.

The base of the MultiPort is flat. The cell will stand up on a lab bench as long as it is not subject to accidental jostling. If you are concerned that it might be knocked over, clamp the cell to a ring stand, available from laboratory supply houses.

CAUTION: The cell can be damaged and/or valuable data can be lost if the cell topples over. Take care that this cannot occur.

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: Standard and Jacketed Versions

The MultiPort 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, glass 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". Do <u>not</u> 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 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 MultiPort kit, make sure you are aware of this restriction, as this joint type is two of the three "spare" ports.

# **Main Cell Assembly**

Assembly of the cell starts with placing the Cell Top onto the Cell Bottom, with the o-ring in the o-ring grove on the Cell Bottom flange.

The Cell Clamp holds the top and bottom together. This clamp is a draw type. With the draw portion released, fit the clamp around the flange, then fit and secure the clamp on the hooks and push the bar back to bring the ends together. If necessary the clamp can be loosend or tightened either by fingers or using a flat head screwdriver.

CAUTION: The cell requires very modest clamping force. If you are having difficulty closing the clamp loosen the bar and try again. Do not force the clamp or tighten it beyond what is necessary to keep the top from sliding/rotating against the bottom (and even that degree is unnecessary in many cases). Overtightening can result in damage to the cell.



## **Bridge Tube and Reference Electrode**

The Bridge tube will fit any of the #7 ports on the top, but the designed use is via the #7 to 15/28 adapter included. It can be adjusted in its depth and position and should generally be positioned with the tip near the sample but not touching. Always use the included ball joint clamp as the joint is not stable without it.

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 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

#### **Counter Electrode**

The Counter Electrode is a rod of very high density graphite.

To fit the counter electrode to the cell, first roll on one of the small black washers included. With the washer on, feed the counter through any of the available #7 ports on the top and secure with a bushing. Leave at least half an inch exposed for electrical contact.

The graphite rod that is shipped with your MultiPort Cell Kit is spectroscopic grade. It is very pure and is therefore unlikely to be a significant source of contamination in your initial experiment. However, the rod is somewhat porous and can adsorb substances present in your test solution. If you reuse a graphite rod, it can contaminate your test solution. The effect is small, and you are unlikely to see it unless the test solution changes drastically between tests. If this is a concern to you, consider a platinum counter, possibly with a fritted glass tube (available from us) to isolate it further from the test solution.

# **Gas-flow Overview and Terminology**

The Gas Dispersion Tube 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 corrosion 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. 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 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 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.

The MultiPort does not include a gas washing bottle, since they are already available in many laboratories.

#### **Gas Dispersion Tube**

In the MultiPort gas flow control is via a gas dispersion tube that fits a 24/40 standard taper. At Gamry, we often refer to the gas bubbler assembly as a 3-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.

CAUTION: If you use purge and/or blanket gas, you <u>must</u> provide a vent for the gas to escape the cell. The MultiPort 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**

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. 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 severe 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.

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. Bubbling should not be vigorous enough to splash large quantities of electrolyte on the cell walls .

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, we recommend that you:

- Connect the purge gas to the top adapter on of the Gas Dispersion Tube
- Connect the blanket gas to the lower adapter of the Gas Dispersion Tube
- Use a 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 3-way gas control valve, you can switch from purge mode to blanket mode manually by switching the tubing from the top to bottom hose barb.

# Sample Holder Assembly

The Sample Holder is generally the last part inserted into the cell. If you are deoxygenating your test solution, you should do so prior to placing the Test Sample into the 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 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.

A diagram of an assembled Sample Holder can be seen in Figure 5. A threaded rod is used to hold the assembly together and to provide electrical contact with the metal sample. The Teflon Compression Gasket is used to prevent leakage of solution into the interior of the glass rod. The sealing surfaces of this gasket must be smooth or a good seal will not be obtained.

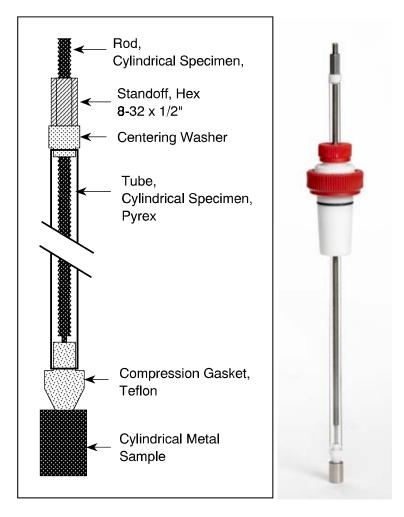
The assembly should be loosely put together before tightening the Hex Standoff. Consult the diagram in Figure v5 for the position of the Sample Holder components. Notice that the centering washer has a small protrusion on one surface. The other surface is flat. The side with the protrusion side faces into the glass tube.

The Hex Standoff at the top of the assembly is tightened while the metal sample is held on the other end. Only tighten the assembly finger tight.

CAUTION: Never use a wrench to tighten the Sample Holder Assembly. Doing so can damage the Teflon Compression Gasket or even break the glass tube.

NOTE: A small amount of bending in the glass tube may occur when you tighten the assembly. This is normal and no cause for concern.

Figure 5
Sample Holder Assembly (pictured with 24/40 port adapter)



# Inserting the Sample Holder into the Cell

The assembled Sample Holder is placed in the 8 mm Teflon tubing adapter. Loosen the smaller of the two knurled nuts on the adapter. From the bottom of the adapter, slide the top of the Sample Holder into the adapter's central hole. The Hex Standoff and Centering Washer should easily pass through the adapter.

Leave approximately 8 cm of the of the glass tube extending below the adapter. When you have the tube at the desired position, tighten the smaller knurled nut again.

Insert the Teflon adapter into the central hole in the Cell Top. Be careful that the sample doesn't hit the bridge tube.

Once you insert the sample holder, watch the glass tube to see that it does not fill with the test solution. You are interested in the electrochemical behavior of your sample, not that of the stainless steel rod holding the sample in place. Leaks can be the result of:

- A damaged Teflon Compression Gasket,
- A scratch or other imperfection in the sealing surface on the sample,
- A chip on the end of the Glass Tube.

The larger knurled nut on the adapter is used when it is time to remove the adapter. Without this nut, it is difficult to grab the adapter firmly enough pull it out of the Cell Top. By turning the nut so that it presses against the top of the cell, you can easily remove the adapter, no matter how deeply it is in the cell top.

# **Electrode Connections**

If you are using your MultiPort 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 and Blue leads from the cell cable are attached to the Working Electrode. Use an alligator clip on each of the leads. Clip each lead to the threaded portion of the Sample Holder Rod, above the Hex Standoff. Do not clip one lead to the rod and the second clip to the first clip.

NOTE: 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.

Make sure that the 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, 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 (such as Gamry's VistaShield™, must be connected to the potentiostat's ground lead and may be connected to earth ground as well.

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 MultiPort with a potentiostat sold by a different manufacturer, consult that potentiostat's documentation for electrode connection information.

# **Customizing Your Cell**

The MultiPort was designed to have options available even while fully accommodating the ASTM G5 standard. There are seven total ports and only three or four necessary for most experiments (5 or 6 for full ASTM G5 compliance with opposed counter electrodes and a thermometer or temperature probe). With anywhere from 1 to 4 spare access ports and a two-piece cell design, customization options are myriad. Some common modifications to experiments are mentioned below.

## **Addition of Corrosive Agents**

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.

In either case the additional ports will allow for making changes to the solution easily.

#### **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 purchase the jacketed option for the corrosion cell. 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 will be 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 using the spare port.

# **Selected Specifications**

#### Cell

1000 ml standard operating volume Volume

Port Type 24/40 standard taper #7 glass thread 15/28 socket

Number of Ports 3 1 3

# **Working Electrode**

Threaded Hole US 3-48 Coarse Thread

0.03 square inches nominal  $0.20~\mathrm{cm}^2$  nominal Area under Gasket

# **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.
- If your cell includes a counter electrode isolation tube it 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, many overloads

- 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).
- 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**

#### Troubleshooting--

- A variable amount of test solution is leaking under the Teflon Compression Gasket. It does not have to actually reach the inside of the sample holder tube to have an effect.
- 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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