Limited Warranty

Gamry Instruments, Inc. warrants to the original user of this product that it shall be free of defects resulting from faulty manufacture of the product or its components for a period of one year from the original shipment date of your purchase.

Gamry Instruments, Inc. makes no warranties regarding either the satisfactory performance of this Corrosion Test Cell or the fitness of the product for any particular purpose. The remedy for breach of this Limited Warranty shall be limited solely to repair or by replacement, as determined by Gamry Instruments, Inc., and shall not include other damages.

Gamry Instruments, Inc. reserves the right to make revisions to the product at any time without incurring any obligation to install same on systems previously purchased. All product specifications are subject to change without notice.

There are no warranties which extend beyond the description herein. This warranty is in lieu of, and excludes any and all other warranties or representations, expressed, implied or statutory, including merchantability and fitness, as well as any and all other obligations or liabilities of Gamry Instruments, Inc.; including but not limited to, special or consequential damages.

This Limited Warranty gives you specific legal rights and you may have others which vary from state to state. Some states do not allow for the exclusion of incidental or consequential damages.

No person, firm or corporation is authorized to assume for Gamry Instruments, Inc., any additional obligation or liability not expressly provided herein except in writing duly executed by an officer of Gamry Instruments, Inc.

If You have Problems

Contact us at your earliest convenience. We can be contacted via:

Telephone	(215) 682-9330 8:00 AM - 6:00 PM US Eastern Standard Time
Fax	(215) 682-9331
Email	techsupport@gamry.com
Mail	Gamry Instruments, Inc. 734 Louis Drive Warminster, PA 18974 USA

If you write or fax us about a problem, provide as much information as possible.

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.

Copyrights

Corrosion Cell Kit

Operator's Manual

Revision 1.1
August 15, 1994

Table of Contents

--

Introduction

The Gamry Instruments Corrosion 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 two Metal Samples, Inc. P/N 410 Series samples. The sample material is C1018 mild steel.

The cell components were selected to be as chemically inert as possible. In normal use the only materials in contact with the test solution are glass and TFE (teflon). The cell is therefore usable with a wide variety of test solutions, including nonaqueous media.

Unpacking and Checking Your Corrosion Cell Kit

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

Checking for Shipping Damage

Your Corrosion 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 Corrosion 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.

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
Corrosion Cell Kit Packing List

Quantity	Part Number	Description
1	990-00028	Manual, Corrosion Cell
1	930-00006	Corrosion Cell Bottom, Pyrex
1	930-00007	Corrosion Cell Top, Pyrex
1	935-00006	Corrosion Cell Clamp, Metal
1	930-00003	Reference Electrode, SCE
1	930-00002	Lugin Capillary, Glass
1	930-00004	Gas Dispersion Tube, Pyrex
2	935-00003	Graphite Rod, Counter Electrode
1	935-00004	Adapter, 24/40 to 6 mm Tube, Teflon
2	935-00005	Adapter, 24/40 to 8 mm Tube, Teflon
1	930-00001	Tube, Cylindrical Specimen Holder, Pyrex
1	820-00002	Rod, Cylindrical Specimen Holder, S.Steel
2	820-00001	Compression Gasket, Teflon
2	820-00004	Centering Washer, Polypropylene
1	850-00005	Standoff, Hex 8-32 x 1/2", S. Steel
2	820-00005	Cylindrical Specimen, C1018 Mild Steel

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 Corrosion 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 simple Lugin capillary and a gas bubbling tube.

Your kit was shipped to you with two C1018 mild steel metal samples. Please use these samples to try out the assembly of your cell as your 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. Three of the four ports in the cell top are shown in this figure. In the interests of clarity, the fourth port, which holds a gas dispersion tube in the normal cell configuration, is not shown. A more detailed diagram of the working electrode assembly is shown in Figure 2.

If you are assembling your Corrosion Cell kit 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.

All of the ports on the cell top are 24/40 ground glass female joints. The central port is used for the Sample Holder (the Working Electrode). The remaining three ports are identical, and any cell component can be placed in any port.

> CAUTION: In vacuum work in a chemical laboratory, ground glass joints are often greased. This is unnecessary with the Corrosion Cell, and may even cause problems if the grease gets into the test solution or onto the Metal Sample. Do not grease any of the ground glass joints on your Corrosion Cell.

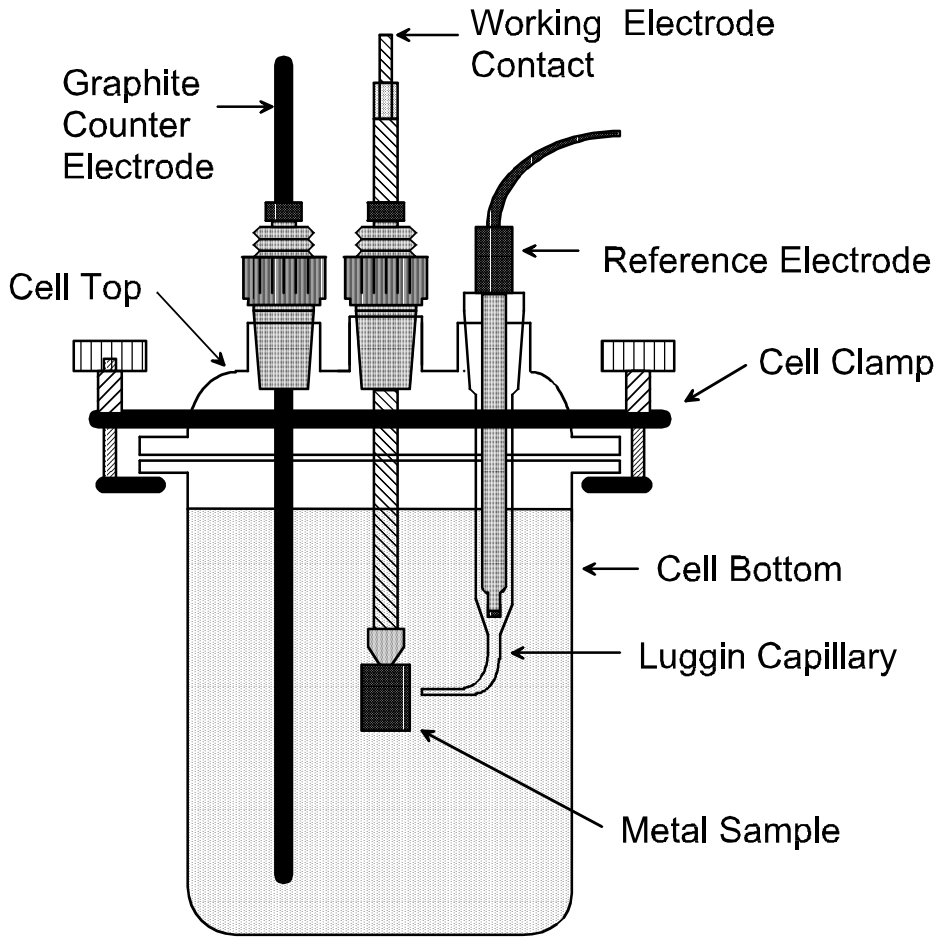
The Cell Bottom is flat on the bottom. The cell will stand up on a lab bench as long as it is not subject to accidental jostling. If you are concerned that it might be knocked over, apparatus to clamp the cell to a ring stand is available from laboratory supply houses.

> 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



Main Cell Assembly

Assembly of the cell starts with placing the Cell Top onto the Cell Bottom.

The Cell Clamp holds the top and bottom together. This clamp has three spring loaded clips. Push down on the knob on top of each clip, then rotate the clip so that its metal tab is under the lip of the cell. Gently raise the knob.

> CAUTION: The springs on the Cell Clamp clips are strong enough to chip the glass cell Do not fully depress a clip and let in snap back into place. Gently release the spring tension.

Lugin Capillary and Reference Electrode

The Lugin Capillary is placed in any of the three available ports in the cell top (remember that the central port is reserved for the Sample Holder). It can be rotated so that the tip of the capillary points towards the corrosion sample.

The test solution can fill the Lugin Capillary through the hole in its tip. Unless your test solution is unusually viscous, the capillary will quickly fill with the test solution, so that the liquid level in the cell and the capillary are the same.

Insert the reference electrode into the wide opening at the upper end the Lugin Capillary. It will contact the test solution that has risen into the tube. Each Corrosion Cell Kit comes equipped with an SCE (Saturated Calomel Electrode) reference. This is a single junction device, utilizing a porous Vycor tip to isolate the electrode's saturated KCl from the test solution.

Reference electrodes with the same outside dimensions, but of different types (Ag/AgCl, double junction, etc.) may be available from our supplier. Contact us for details.

The reference electrode is shipped with a small plastic cap protecting the Vycor tip of the electrode. The electrode tip must be kept wet or covered by this cap at all times. The Vycor tip can be damaged if the electrode tip is exposed to dry air for a significant period of time.

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 liquid level in the electrode drops. It also provides a vent. In normal use, we recommend that the fill hole be kept slightly open so the electrode is vented.

Counter Electrode

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

The Graphite Rod Counter Electrode is placed in the 6 mm (the smallest) Teflon tubing adapter. Loosen the smaller of the two knurled nuts on the adapter. Slide the graphite rod into the adapter's central hole. About two thirds of the rod should extend below the adapter. You may need a little pressure to get by the o-ring. When you have the rod at the desired position, tighten the smaller knurled nut again.

The adapter is then slid into one of the ports in the Cell Top. It can be pressed firmly into place. 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.

The graphite rod that is shipped with your Corrosion Cell Kit is spectroscopic grade. It is very pure and is therefore is 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.

Gas Dispersion Tube

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 a polarization curve. To avoid this noise, don't bubble gas through the solution during the data acquisition phase of your experiment. Instead flow the gas over the top of the test solution.

Regardless of whether the nitrogen is going through or over the test solution, you must provide a way for it leave the cell. We recommend that you avoid firmly seating the Gas Dispersion Tube's Teflon adapter firmly into the cell top.

The Gas Dispersion Tube is placed in one of the 8 mm (larger size) Teflon tubing adapters. Loosen the smaller of the two knurled nuts on the adapter. From the bottom of the adapter, slide the top of the Gas Dispersion Tube into the adapter's central hole. About two thirds of the tube should extend below the adapter. You may need a little pressure to get by the o-ring. When you have the tube at the desired position, tighten the smaller knurled nut again.

The adapter is then slid into one of the ports in the Cell Top. It should not be sealed in place. A small piece of wire or other obstruction can be helpful in making sure that it does not form a good seal with the cell top.

You can adjust the depth of the fritted portion of the Gas Dispersion Tube in the test solution. Simply slide it up or down in the adapter. With the Gas Dispersion Tube slid all the way up, you can flow nitrogen over the top of the test solution without actually removing the tube or its adapter from the cell.

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 2. A threaded rod is used to hold the assembly together and to provide electrical contact with the metal sample. The Teflon Compression Gasket is

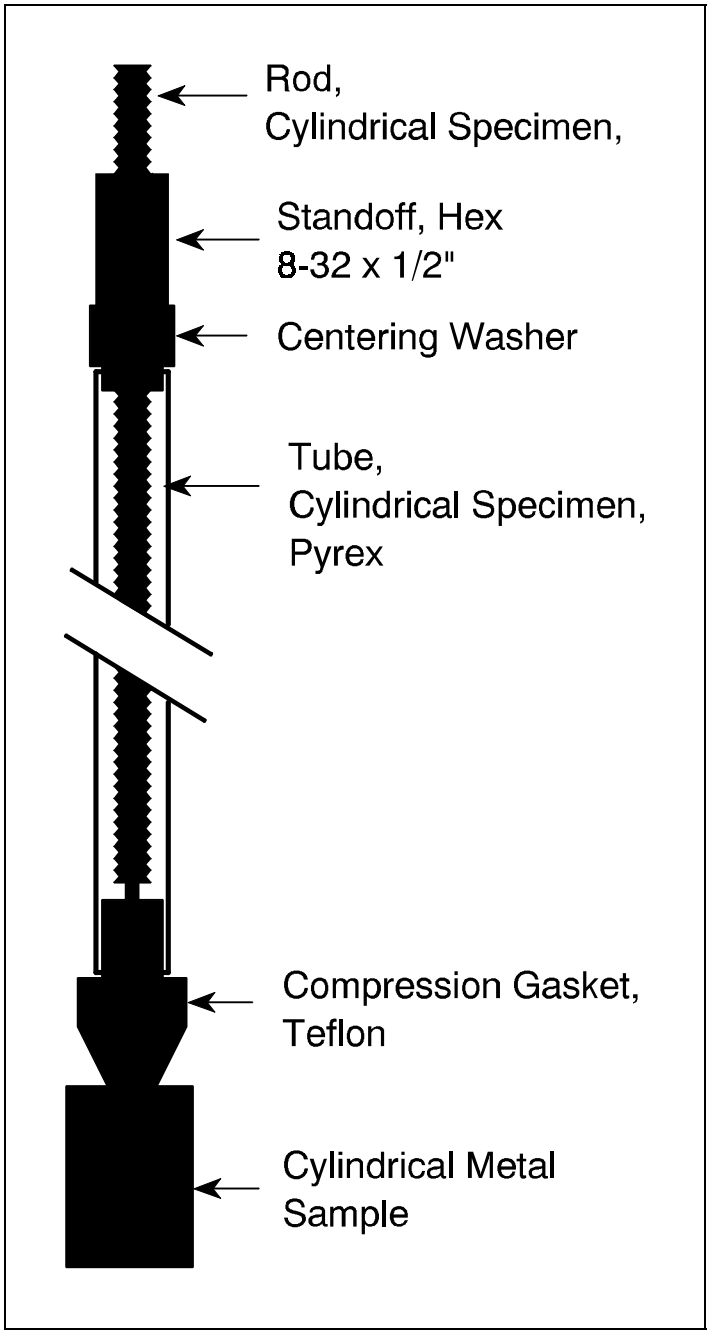
used to prevent leakage of solution into the interior of the glass rod. The sealing surfaces of this gasket must be smooth or a good seal will not be obtained.

The assembly should be loosely put together before tightening the Hex Standoff. Consult the diagram in Figure 2 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 2
Sample Holder Assembly



Inserting the Sample Holder into the Cell

The assembled Sample Holder is placed one of the 8 mm (larger size) Teflon tubing adapters. 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 Lugin Capillary. If you drop the sample holder into place, you can actually break the Lugin Capillary.

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 Corrosion Cell 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 shield, should be connected to an earth ground. The Black lead from the cell cable is then connected to the Faraday shield.

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

Customizing Your Cell

There are many things that you can do to customize your cell. If you do not need to deaerate your test solution, you can leave out the Gas Dispersion Tube. This gives you a free port in the cell top. This port can be used to hold a thermometer or other temperature measuring device. It can also be used for other purposes.

You can isolate your Counter Electrode from the test solution by use of a fritted glass tube in place of the Graphite Rod. A 6 mm tube will fit in the Graphite Rod's adapter. You can then use a platinum wire counter electrode inserted into the fritted tube as the new counter electrode.

You can also use other non-cylindrical sample geometries. It is helpful if the samples used with this cell fit through a 24/20 ground glass joint. Don't discount epoxy or other coatings as a means of defining the active area of your metal sample and covering up the region where the sample contacts the Sample Holder Support Rod.

Selected Specifications

Cell

Volume	1000 ml maximum
	800 ml approximate in use
Port Type	24/40 standard taper
Number of Ports	4

Reference Electrode

Type	Saturated Calomel
Isolation	Porous Vycor

Working Electrode

Threaded Hole	US 3-48 Coarse Thread
Area under Gasket	0.03 square inches nominal 0.20 cm^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

- The counter electrode (red) lead in the cell cable is not connected to the cell properly.
- The working electrode (green) lead in the cell cable is not connected to the cell properly.

Full scale current when you run an experiment

- 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).
- There is a gas bubble in the Lugin Capillary.
- Solution has leaked into the Sample Holder Tube.
- Two or more of the cell leads are shorted together.

Noisy Cell Current

- Your deoxygenation gas is still bubbling through the solution.
- The corrosion system is naturally noisy. Either pits are forming randomly or gas bubbles are randomly blocking some of the working electrode surface.
- There is a gas bubble in the Lugin Capillary.
- Solution has leaked under the Teflon Compression Gasket. It does not have to actually reach the inside of the sample holder tube to have an effect.

Excess back pressure required to bubble deoxygenation gas

- No path is available for the gas to escape.

Poor Experimental Reproducibility

- 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.

Index