



EuroCell

Operator's Manual



Part number 988-0002

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Revision 1.22
October 5, 2016

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Telephone	(215) 682-9330 9:00 AM - 5:00 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. Optional parts may be available from Gamry Instruments, or from 3rd party vendors. Contact your local Gamry sales representative to discuss any special requirements or accessories that you need.

Disclaimer

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Introduction

Gamry Instruments designed the EuroCell™ kit for electrochemical corrosion testing. Like Gamry's Multiport, it is used with cylindrical metal specimens. The EuroCell is more versatile and less expensive than the Multiport, and is an alternative to the Multiport.

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

Alabama Specialty Products.
P.O. Box 6
Munford, AL 36268 USA
Telephone (256) 358-4202 fax (256) 358-4515

Your Euro Corrosion Cell kit was shipped with one Alabama Specialty Products P/N 410 Series sample. The sample material is C1018 mild steel. Most of the pieces required to run routine experiments are included in EuroCell kit.

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/Hg₂SO₄) that are suitable for use with your EuroCell kit. Your reference electrode should be ordered separately.

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

Too often, temperature control is neglected in designing electrochemical experiments. Temperature is an important variable in the rate of 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 EuroCell cell body. When this cell is connected to a circulating water bath, accurate temperature control of your experiments becomes possible.

Chemical Compatibility of the EuroCell

The components in the EuroCell 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 glass frit,
- 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 an 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 fittings' 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 EuroCell was not designed for use in electrolytes that dissolve glass (extremely basic solutions or HF containing solutions).

CAUTION: The glass components in the cell and the glass frits used in the reference bridge tube are not suitable for use with extremely basic solutions or solutions containing hydrofluoric acid. We 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 EuroCell 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 a Cell Kit

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

Checking for Shipping Damage

Your new EuroCell 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 EuroCell 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 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 EuroCell kit packing list in Table 1. When shipped, all of the EuroCell 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 Figure 1 later in this manual.

Table 1
EuroCell Kit Packing List

Quantity	P/N	Description
2	820-00001	Teflon Compression Gasket
2	820-00004	Sample Rod Centering Washer
1	820-00005	C1018 Sample, Cylinder
1	820-00036	Sample Rod, Threaded
1	850-00005	Standoff, Hex 8-32 Threaded
1	930-00030*	EuroCell Cell Body (incl. 2 #7 bushing with O-ring)
1	930-00041**	EuroCell Cell Body, jacketed (incl. 2 #7 bushing with O-ring)
1	930-00033	Gas Flow Adapter
1	930-00039	Tube, Glass, Elect Holder, EuroCell
1	930-00040	Bubbler, Ace to Frit
3	930-00042	Glass Frit w/ Teflon Heat Shrink Tubing
1	930-00045	Reference Electrode Bridge Tube, EuroCell w/ glass frit
1	935-00005	Adapter, 24/40 to 8 mm Tube
1	935-00014	Graphite counter electrode
1	935-00053	#11 ACE-Thred Bushing, Nylon with 2 O-rings
1	935-00054	Adapter, #7 ACE-Thred to ¼" hose
5	935-00059	Polyethylene Stoppers for 14/20 Joint
1	988-00002	Manual, EuroCell

* Only included in the Standard EuroCell configuration, 990-00196 (no stand).

** Only included in the Jacketed EuroCell configuration, 990-00203.

Contact us as soon as possible if any of the parts are missing. The contact information for our US facility can be found on the first pages of this manual. If you're located outside the US, you may want to contact your local Gamry representative.

Assembly of and Use of Your Cell Kit

This section of the manual tells you how to assemble and use a EuroCell kit in its standard configuration.

This "standard" cell configuration has:

- a standard cell body for use at room temperature
- a corrosion sample in the central 24/40 standard taper port
- a 3-way gas purge/blanket/vent adapter in one 14/20 standard taper port
- a graphite-rod counter electrode in one #7 ACE-Thred port
- a reference electrode and bridge tube in one #7 ACE-Thred port
- one unused 14/20 standard taper port (may be closed with a stopper)

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

Cell Assembly- General Information

A picture of an assembled cell can be seen in Figure 1.

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

If you are assembling your EuroCell kit for the first time, you may want to assemble 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.

We recommend that you start the assembly of your cell by clamping the cell body in a three-fingered clamp attached to a ring stand. Gamry sells a ring stand kit which can be purchased using part number 990-00202. Alternatively, these can be purchased from any supplier of laboratory equipment. The cell body is not stable when laid on a flat surface such as a lab bench and can easily roll off a bench onto the floor with cell-shattering results. Gamry Instruments can always use additional sales, but we'd hate to have them come in the form of replacement cell bodies!

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

Three of the ports on EuroCell are standard taper ground glass joints. These joints should not be greased.

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

Figure 1
The Standard Configuration of a EuroCell



You should pay careful attention to cell cleanliness. In most electrochemical testing situations, contaminants in the cell and test solution can lead to poorly reproducible results.

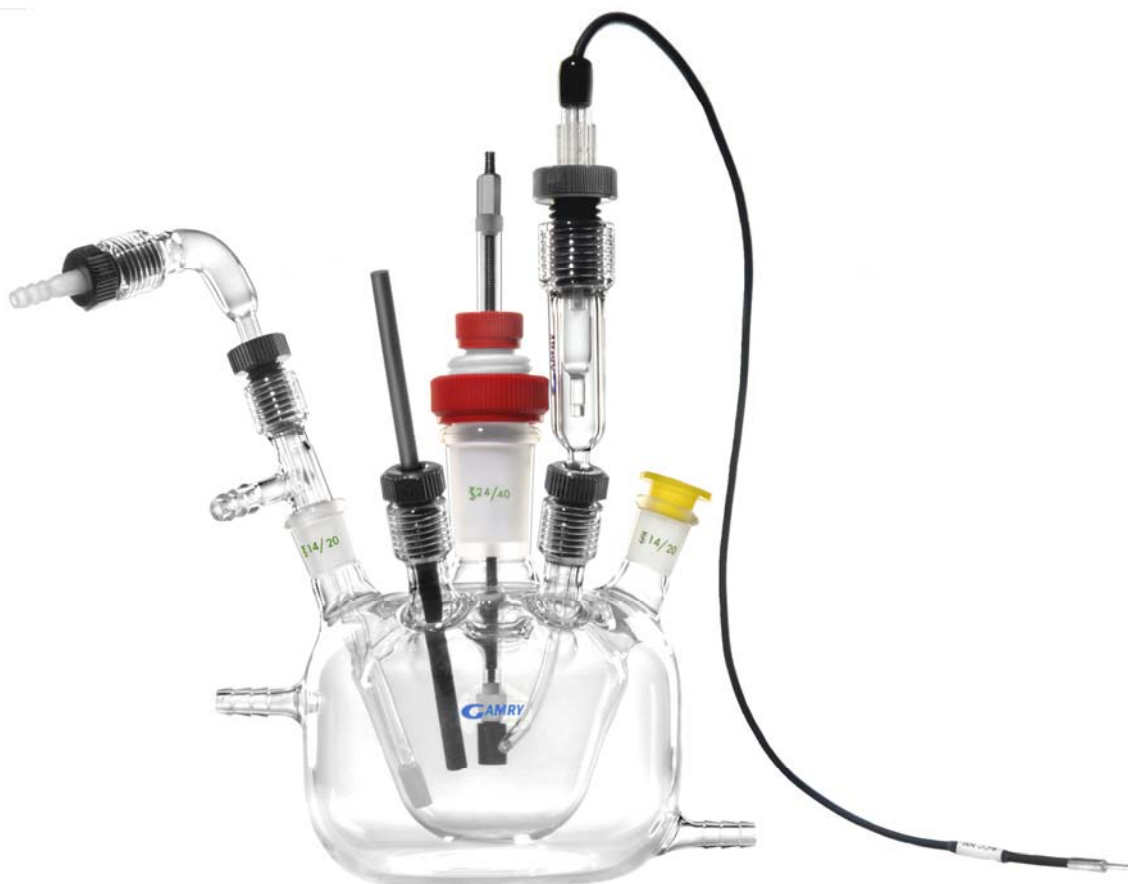
If you touch the cell components with your fingers, you can inadvertently add salts and organic compounds to your cell solution. We recommend that you carefully clean the cell components using good laboratory practice. After the components are clean avoid touching their wetted surfaces.

EuroCell 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 not use tools such as a wrench or pliers to tighten an ACE-Thred fitting. Overtightening 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 EuroCell kit, make sure you are aware of this restriction.

Figure 2
The Jacketed Configuration of the EuroCell



Gas-flow Overview and Terminology

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

Oxygen is an electrochemically active gas. 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 can remove oxygen from the test solution by bubbling nitrogen, or another electrochemically inert gas, through the solution. This process is often (imprecisely) called de-aeration. It is more correctly called de-oxygenation. Other commonly used terms for the process of oxygen removal are gas purging or sparging. At least 15 minutes of vigorous bubbling with nitrogen 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' Pulse Voltammetry and Physical Electrochemistry 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 its Purge and Stir option, provides a complete solution for purge gas control.

Pre-saturation of the Purge Gas

Bubbling dry purge gas through your cell electrolyte can cause significant evaporation of the electrolyte's solvent during the purge process. This can be a 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.

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

EuroCell Gas Bubbler Assembly

The standard gas bubbler assembly consists of four pieces, the Gas Flow Adapter, the Gas Bubbler Tube, a #7 ACE-Thred nut, and an ACE-Thred To Hose adapter. A photograph of these pieces can be seen in Figure 3.

Figure 3. Gas Bubbler Assembly



Figure 3, is representative of the Gas Bubbler used for purge only (no blanketing). A plastic hose barb is shown connected to the Gas Bubbler Tube using the ACE-Thred To Hose adapter. The other end of this hose is connected to a source of purge gas.

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 or blanket gas, you must provide a vent for the gas to escape the cell. EuroCell 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.

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

Attaching Gas Tubing to the Cell

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

Your gas flow system should include a needle valve to control the gas flow rate. All gas tubing connections to the cell should be made 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.

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

If you do not have a 3-way gas control valve, you can switch from purge mode to blanket mode manually. Connect the gas flow to the ACE-Thred To Hose adapter. When you want to purge, loosen the ACE-Thred nut holding the Gas Bubbler tube in place, slide the Gas Bubbler Tube deeply into the cell so its orifice is in the cell solution, then retighten the ACE-Thred fitting. When you want to blanket, slide the Gas Bubbler Tube far out of the cell, so its orifice is above the solution. When you use the cell in this way, the hose barb port on the Gas Flow Adapter provides a convenient vent.

Counter Electrode Assembly

Counter Electrode Overview and Terminology

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

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

High Density Graphite Counter Electrodes

Your EuroCell kit comes with a high density graphite rod counter electrode.

The rod is 150 mm long and approximately 6.3 mm in diameter. It fits nicely in a #7 ACE-Thred port. The Gamry Instruments part number for replacement graphite rods is 935-00014.

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

The graphite rod that is shipped with your EuroCell 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.

EuroCell Counter Electrode – Optional Isolated Assembly

In the standard configuration of the EuroCell, the counter electrode is a graphite rod, which is immersed directly in the test solution.

In some cases, you may want your counter electrode isolated from the bulk solution by a glass frit. Gamry Instruments sells an Isolated Counter Electrode kit (Part Number 990-00194) that works with both the EuroCell and the PTC1 Paint Test Cell kit.

There are two pieces in this isolated counter electrode kit:

- a fritted isolation tube
- 150 mm of platinum wire.

A complete counter electrode assembly (without the platinum wire) is shown in Figure 4. Note the placement of the required O-ring between the nut and the fritted end of the tube.

Figure 4. Counter Electrode Isolation Tube



The fritted tube is slid into a #7 ACE-Thred port on the cell. Slide the platinum wire into the isolation tube. The lower end of the platinum wire must be in contact with electrolyte. A sharp bend in the platinum wire at the upper rim of the isolation tube can help prevent loss of contact between the electrolyte and the platinum. When the counter electrode wire is clipped to the platinum wire below this bend, gravity helps keep the counter electrode in place.

Reference Electrode and Bridge Tube

Overview and Terminology

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

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

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

To avoid these problems, EuroCell always uses a bridge tube to locate the reference electrode above the cell. The bridge tube is a glass tube filled with electrolyte that provides a conductive path from the cell electrolyte to the tip of the reference electrode. The electrolyte in the bridge tube is usually identical to the cell electrolyte, but in some cases a different electrolyte may be called for.

A bridge tube is similar, but not identical, to a Luggin capillary. A Luggin capillary attempts to move the "sensing point" of the reference electrode close to the working electrode surface. The bridge tube in EuroCell has too large a tip for it to qualify as a true Luggin capillary.

The bridge tube in EuroCell is particularly convenient, because it terminated in an unfired glass frit, which makes filling the bridge tube easy yet doesn't add a lot of resistance to the reference electrode circuit. Most other bridge tube or Luggin capillary designs either have excessive resistance causing noisy data, potentiostat oscillation or are difficult to fill with electrolyte.

Bridge Tube and Reference Electrode Assembly

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

Figure 5. Reference Electrode Bridge Tube



EuroCell does not include a reference electrode, because different tests may call for different reference electrode types. The reference electrode shown in Figure 5 is Gamry's standard saturated calomel reference electrode (SCE) P/N 930-00003. Gamry also offers an Ag/AgCl Reference (P/N 930-00015) and a Hg/Hg₂SO₄ reference (P/N 930-00029). Contact your local Gamry sales representative if you need a new or a replacement electrode.

EuroCell can be used with third-party reference electrodes, if the reference electrode diameter is between 9 and 10.5 mm.

These are the steps required to fill, assemble, and install the bridge tube and reference electrode:

- 1) Place an O-ring and #7 ACE-Thred fitting on the middle section of the bridge tube (see Figure 5).
- 2) Place an O-ring and #11 ACE-Thred fitting on the reference electrode (see Figure 5).
- 3) Pour electrolyte into the larger diameter end of the bridge tube, leaving about 2.5 cm between the bottom of the ACE-Thred and the top of the electrolyte.
- 4) Place the reference electrode, tip first, into the larger end of the bridge tube and tighten the #11 ACE-Thred fitting until it is finger tight and the reference electrode is held in place.
- 5) Place the smaller end of the bridge tube into one of ACE-Thred ports on your EuroCell.
- 6) Adjust the height of the bridge tube so that its tip will be in the electrolyte and close to the working electrode.
- 7) Tighten the bridge tube's #7 ACE-Thred fitting until it is finger-tight and the bridge tube is held firmly in place.

Bridge Tube and Reference Electrode – Maintenance and Care

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

CAUTION: Do not allow the glass frits on the reference electrode or the bridge tube to dry out. They are likely to crack, greatly increasing the flow of bridge tube or reference filling solution into your cell. If the frit does dry out, we recommend that you replace it. Your EuroCell kit contains three replacement frits with Teflon heat-shrinkable TFE sleeves. Use one of these whenever you need to replace a damaged frit.

Whenever possible the bridge tube tip should be stored in the electrolyte used in your system. The bridge tube can simply be placed tip down, in a flask containing the electrolyte you use in your tests.

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

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

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

The reference electrode is also supplied with a clear plastic sleeve covering a fill hole in its glass body. This fill hole serves two purposes. It allows you to refill the electrode with saturated KCl if the liquid level in the electrode drops. It also provides a vent, so the filling solution flows slowly out of the electrode. In normal use, we recommend that the fill hole be kept slightly open. When storing the electrode, the fill hole should be closed.

Note: If the electrode or the bridge tube has been stored in a pure, poorly conductive solvent, the glass-frit tip must refill with ions prior to use. In the case of the reference electrode, ½ hour with the vent hole open should suffice. In the case of the Bridge Tube, ½ hour with a vented reference in place should also be sufficient.

Assembly of and Use of Your Cell Kit--Reference Electrode and Bridge Tube

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

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

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. Many 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 6. 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.

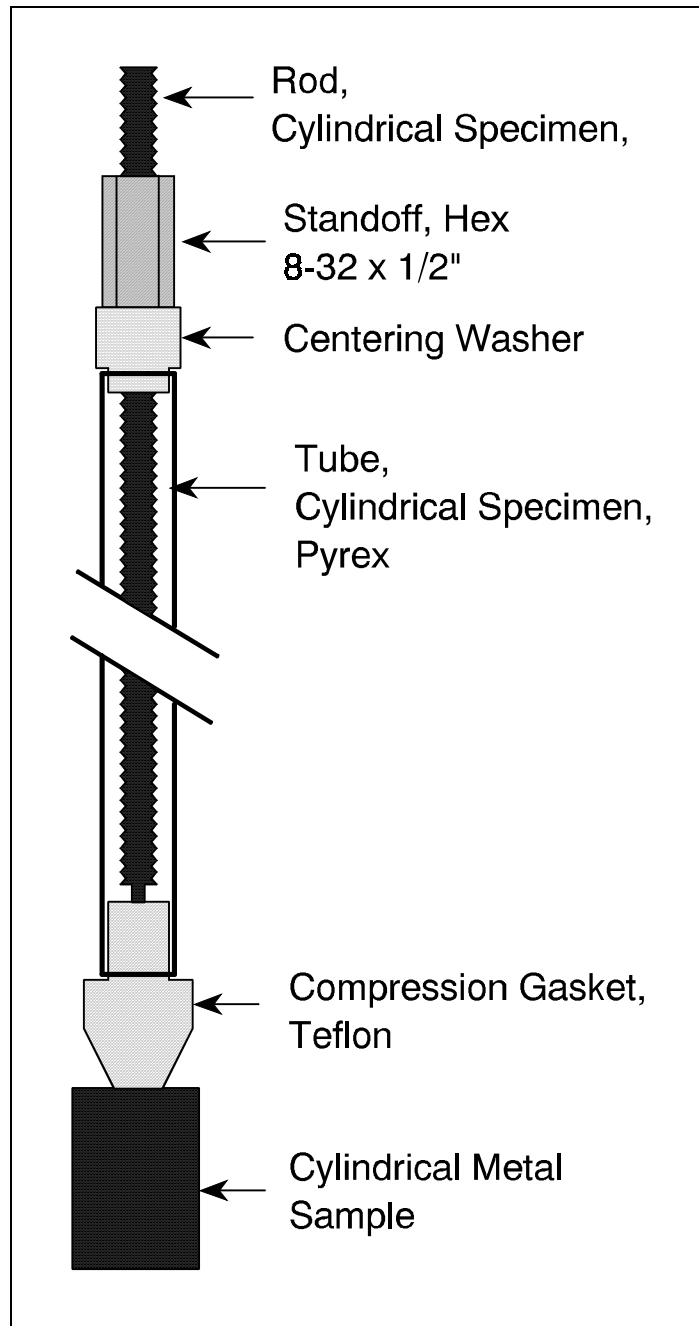
The assembly should be loosely put together before tightening the Hex Standoff. Consult the diagram in Figure 6 for the position of the Sample Holder components. Notice that the centering washer has a small protrusion on one surface. The other surface is flat. The side with the protrusion side faces 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 6
Sample Holder Assembly



Spare Port

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

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

Addition of 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.

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

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.

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

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

- you purchase the jacketed option for the EuroCell. This is a special cell body (Gamry part number 930-00041) 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 the spare port of the cell. This may require purchase of another option (for example an RTD to 14/20 port adapter).

In some cases, Gamry's software supports automatic temperature setting. The controlled temperature is 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.

Moving the Pieces About

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

Assembly of and Use of Your Cell Kit--Sample Holder Assembly

There is no requirement that any electrode or adapter mount in any specific port. However, the main port (24/40 joint) is the largest diameter of all the ports.

Electrode Connections

If you are using your EuroCell with a Gamry Instruments Potentiostat, the following connections are 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. Cell current in a EuroCell experiment are often large enough that the Blue and Green leads should be separately clipped to the working electrode.

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 long Black lead on the cell cable cannot touch any other cell connection. You may find that connection of this lead to a source of earth ground, such as a water pipe, 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, should be connected to an earth ground. The Black lead from the cell cable is then connected to the Faraday shield.

Gamry's VistaShield is a versatile, easy to use Faraday cage that was designed to work well with the EuroCell kit. When equipped with a Purge and Stir option, it provides a complete electrochemical corrosion test stand, well integrated with Gamry's cells, potentiostats, and software systems.

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

Troubleshooting

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

One very common and often embarrassing error is to forget to connect one of the cell leads! Always double check your cell connections.

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

- Purge gas or gaseous reaction products collect on the face of the working electrode.
- The counter electrode wire is not in the solution.
- If you have a counter isolation tube, it can fail to fill with electrolyte.
- The Reference Bridge tube contains a bubble between the reference electrode and the electrolyte.
- A bubble collects on the glass frit at the end of the reference electrode.

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

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

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 Luggin Capillary.

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 vent 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.

Specifications

Cell

Volume 175 ml maximum
 125 ml minimum

Port Types One 24/40 standard taper ground glass joint
 Two 14/20 standard taper ground glass joint
 Two #7 ACE-Thred ports

Reference Electrode

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

Working Electrode

Diameter 7 to 9 mm (using the adapter supplied with the cell)
Length 50 mm minimum (using large cell volumes)

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