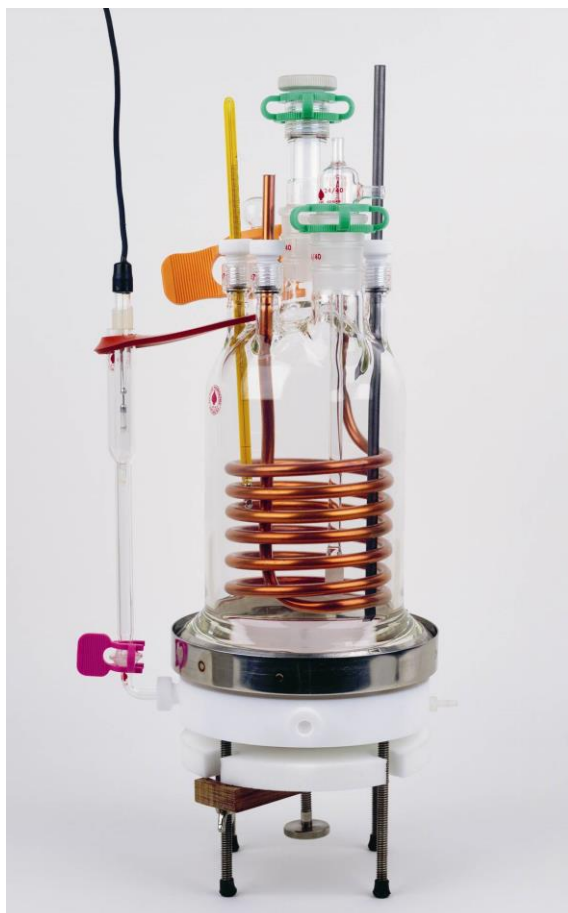




Flexcell Cell Kit

Operator's Manual



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Flexcell 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. Optional parts may be available from Gamry Instruments, or from third-party vendors. Contact your local Gamry sales representative to discuss any special requirements or accessories that you need.

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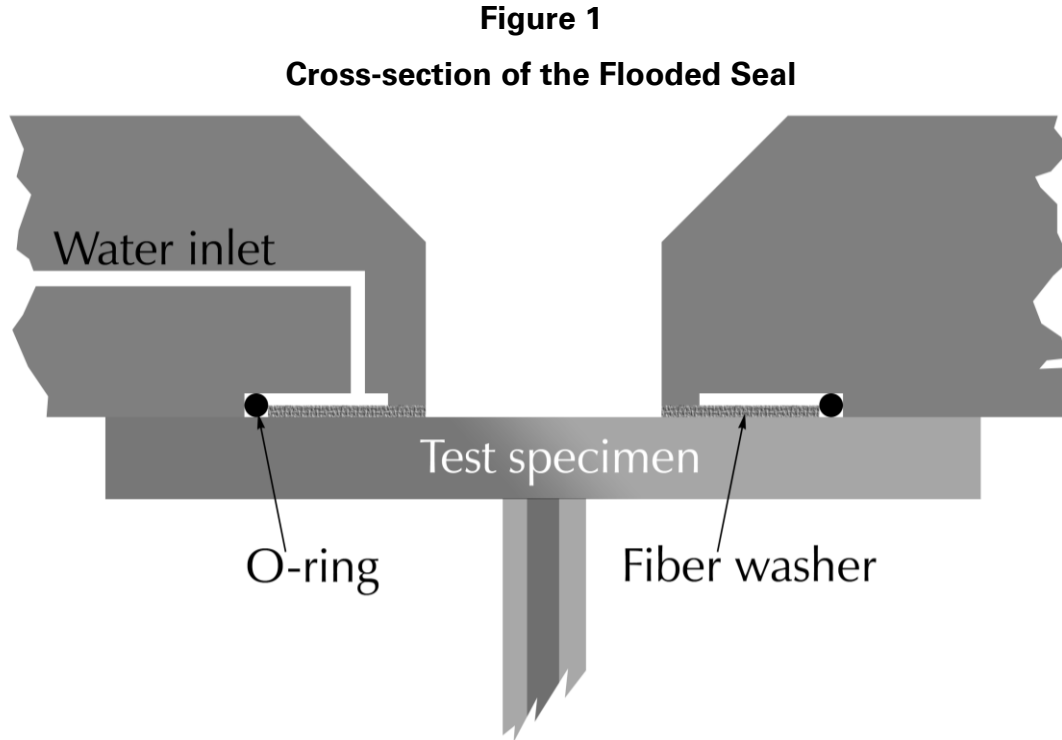
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Chapter 1: Introduction

The Flexcell is used to perform electrochemical tests on flat metal specimens. Unlike traditional cells, the crevice-free cell avoids localized corrosion at the seal between the sample and its holder. Following a design popularized by Avesta Steel, the cell uses a flooded-gasket seal. This simple, yet elegant, design results in an easy-to-use, reliable, crevice-free system.

Figure 1 is an expanded, cross-sectional diagram of the cell in the area of the flooded seal.



As in conventional cells, the active area of the test specimen is in contact with an electrolyte. Unlike other cells, the active area of the test specimen is not bounded by a plastic barrier or O-ring.

De-ionized water is slowly forced through a fiber washer placed on the test-solution-side of the metal sample being tested. After passing through the washer, the water diffuses into the electrolyte. The fluid in the fiber washer is nearly pure deionized water, which is not conductive enough to support corrosion processes. Thus, instead of a plastic barrier with its resultant crevice, a conductivity gradient between the electrolyte and the deionized water defines the test specimen's active area.

The Flexcell components were selected to be as chemically inert as possible. In normal use, the only materials in contact with the test solution are glass, filter paper, and PTFE. The cell is therefore usable with a wide variety of test solutions, including non-aqueous media.

--Checking for Shipping Damage

Chapter 2: Metal Specimens

A commercial source for suitable flat metal specimens is Metal Samples Co., Inc. Their P/N CO0130 series works well.

The dimensions of this part are 2" × 2" × 1/8" (approximately 5 cm × 5 cm × 0.31 cm). Metal Samples offers the CO0130 series in several hundred different metals. When ordering, make sure that you append a metal code to the part number. For example, the full part number for a cell specimen made from 316 Stainless Steel is CO0130-316.

Metal Samples Co., Inc. can be contacted at:

Metal Samples Co., Inc.
152 Metal Samples Road
P.O. Box 8
Munford, AL 36268 USA

Telephone: (256) 358-4202
Fax: (256) 358-4515
Email: msc@alspi.com

You can also make your own specimens. The specimen must be flat, and sufficiently smooth so that an O-ring can make a leak-free seal to the metal. The minimum specimen size is a square 4 cm on a side. Smaller specimens will not seal on the O-ring outside of the fiber washer.

Metal specimens that are 3.2 mm (1/8 inch) or thicker can be mounted on the cell without any sample support. They are held in place with a metal thumbscrew. You can also use this thumbscrew to make electrical contact with the specimen.

Thin specimens require a support that prevents distortion as the thumbscrew is tightened. The minimum specimen thickness is 0.8 mm (1/32 inch).

On thin metal samples, we recommend that you bend one corner at a right angle to the rest of the specimen. Electrical contact is made with this bent tab on the sample. The thumbscrew cannot be used to make contact, because the sample support is made from electrically insulating plastic (metal would be too thermally conductive).

Chapter 3: Unpacking and Assembling Your Flexcell Kit

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

Checking for Shipping Damage

Your new Flexcell 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 Flexcell kit, please check it for any signs of shipping damage. Be especially careful if the shipping container shows signs of rough handling.

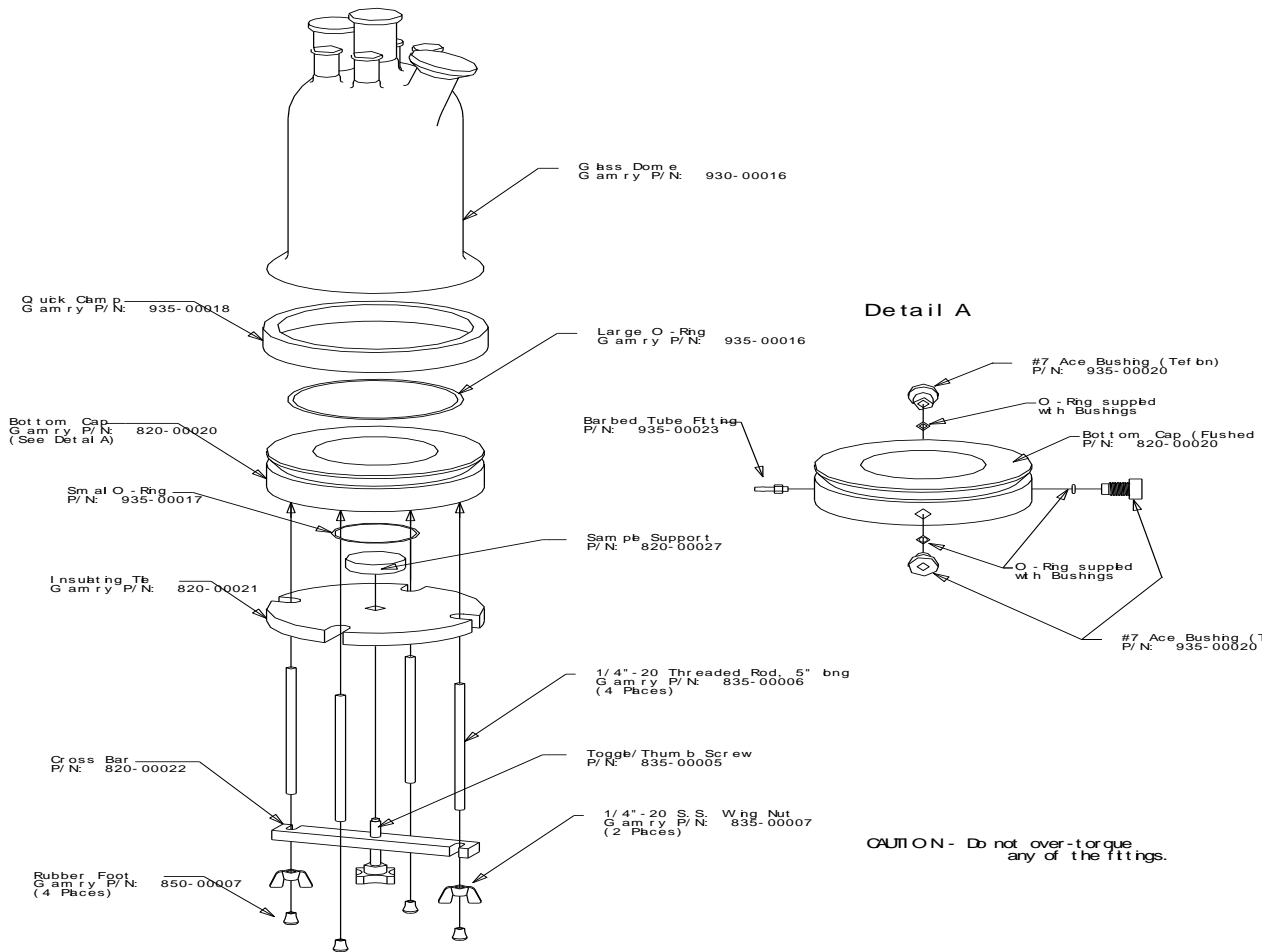
Obviously, the glass pieces are the most susceptible to damage. Check the glass pieces for chipping and small cracks as well as for major damage.

If any parts were broken in shipment, please contact our US facility or your local Gamry representative as soon as possible. In most cases, Gamry should have replacement parts in stock. Please retain the shipment's packaging material for a possible claim against the shipping company.



Warning: Do not use any glass parts that are chipped or cracked. Any damage to glass increases the probability of additional damage. Broken glass can have extremely sharp edges that represent a significant safety hazard. Injuries from broken glass can be quite severe.

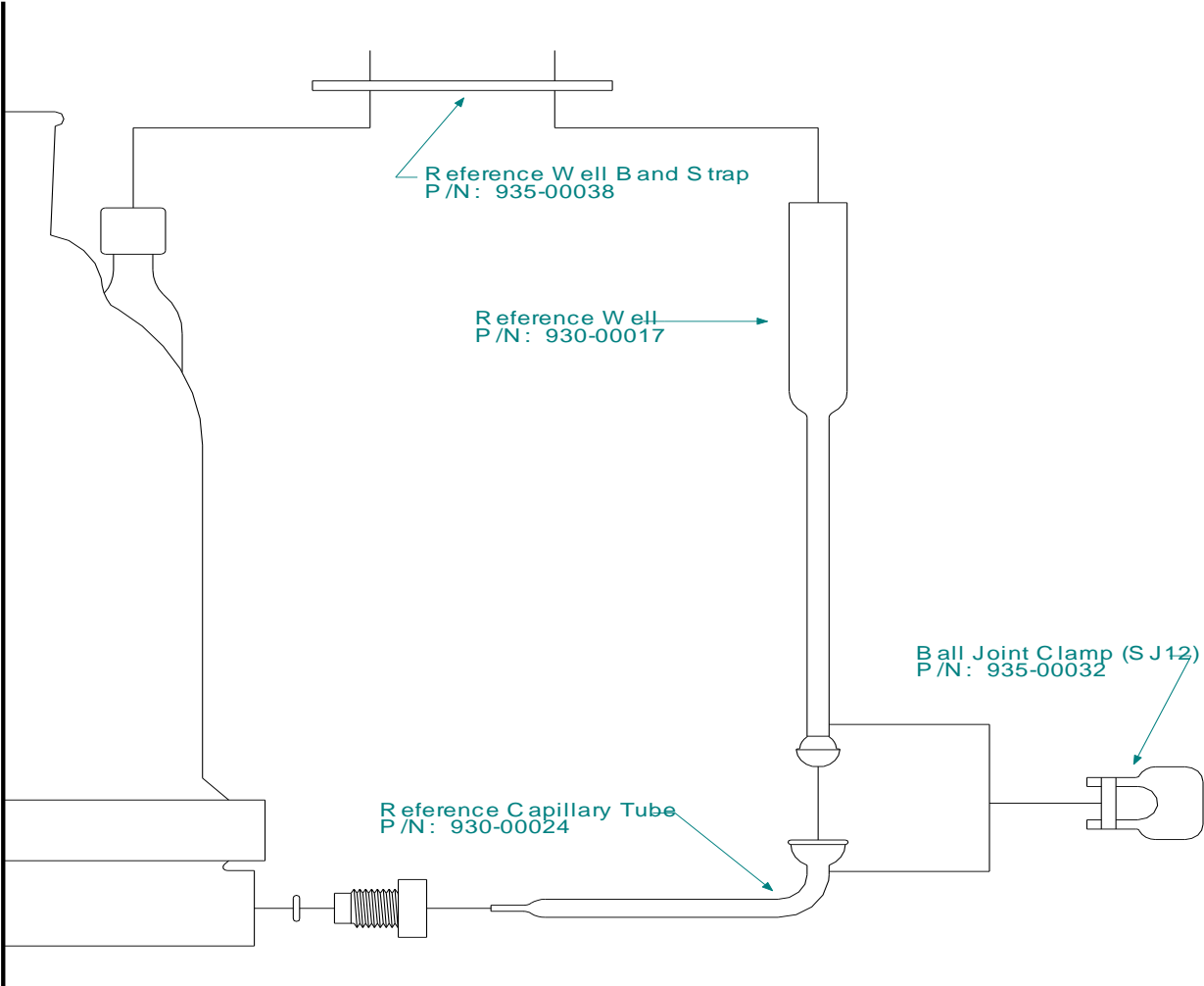
**Figures 3-1
Flexcell Assembly**



CAUTION - Do not over-torque any of the fittings.

Any unused #7 Bushing port can be plugged using the glass rod/plug (930-00016) supplied with the kit. Just slip the O-ring over the rod like any other 6-7 mm probe or electrode. Insert the rod into the port. Then use the bushing to tighten the seal.

Figure 3-2
Flexcell: Reference Well Assembly



CAUTION - Do not over-torque any of the fittings.

Checking Parts against the Packing List

Please check the contents of your Flexcell Kit versus the Packing List that accompanied the shipment. In most cases, individually wrapped Flexcell components are labeled with their Gamry Instruments, Inc. part number (P/N). Be careful, for some of the Flexcell components are small enough to be lost in the cell's packing material.

If you are in doubt about the identity of a component, refer to Figures 3-1 and 3-2. These figures are also useful if you must check the completeness of an older kit.

Assemble the cell.

Assembly of the cell requires only minimal mechanical aptitude and no tools. The rest of this chapter describes the initial assembly process in detail.

This chapter does not cover assembly steps that occur during routine use of the cell. An example is the procedure for changing the test sample. Routine use of the cell is the subject of the next chapter.

Install the Bottom Cap – Leg.

The bottom cap is a large machined disk made from white PTFE. It is a complex part that needs to be equipped with a number of additional parts and fittings prior to its use.

1. Attach legs.
 - a) Lay the bottom cap (P/N 820-00020) on a table with the face containing the conical depression facing the table top.
 - b) Find the four stainless steel rods that form the legs of the cell (P/N 835-00006). There are four threaded holes on the visible face of the bottom cap. Thread one rod into each hole until some resistance is felt. Do not over tighten.



Caution: Do not use tools to screw the rods in place. **Hand-tighten the rods only.**
Over-tightening the rods can damage the bottom cap.

2. Install wingnuts.
 - a) Find the two wingnuts (P/N 835-00007). Screw one wingnut, with the flat surface facing down, about halfway down one of the cell's threaded legs. The exact position on the leg is not important.
 - b) Install the second wingnut the same distance down on the diagonally opposite leg.
3. Press one rubber foot (P/N 850-00007) on the exposed end of each cell leg.
4. Invert the cell and place it on a flat surface, with the four rubber feet touching the surface.
5. Check that the top surface of the bottom cap is approximately level. The assembly should be stable, with only a few millimeters of wobble. If the assembly is not stable, the length of one or more legs can be adjusted by unscrewing them slightly.

Assemble the Sample Holder.

The sample holder mounts underneath the cell. It is held in place by the wingnuts mounted on the cell's legs.

1. Place the bottom cap on a table top with the conical depression facing the table.
2. Install the thumbscrew.

Locate the phenolic crossbar (P/N 820-00022) and the thumbscrew (P/N 835-00005). Screw the thumbscrew through the central hole in the crossbar, starting from the rough side (if any) of the bar. Stop when about 5 mm of the screw extends from the smooth side of the bar.
3. Install the PTFE-coated silicone-rubber O-ring (P/N 935-00017).

Press the O-ring into the groove on the visible side of the bottom cap.
4. Install the Crossbar and Sample Support.
 - a. Locate the sample support (P/N 820-00027). It looks like a small hockey puck. Note the depression in one face of the support.
 - b. Place the sample support, depression up, on the O-ring installed in step 3.
 - c. Hold the crossbar with the pointed end of the thumbscrew pointing downwards. Slide the crossbar between the legs on the cell, below the wingnuts.
 - d. Rotate the crossbar so that each slot in the crossbar engages one of the legs with a wingnut. A glance at Figures 3-1 may be helpful.
 - e. Hold the sample support in one hand. Turn the thumbscrew with your other hand, poking the end of the thumbscrew into the depression in the sample support. When properly installed, the crossbar is pressed against the wingnuts on the legs and the thumbscrew presses the sample support against the cell.



Caution: Do not over tighten the thumbscrew. Excessive tightening can damage the cell's bottom cap. Tighten the thumbscrew about 90° (1/4 revolution) beyond the point at which some resistance is felt.

Install the Bottom-Cap Fittings

The rounded outer surface of the bottom cap contains four threaded ports. Three ports are designed to accept #7 ACE-Thred™ bushings. During normal cell operation these ports are used for the reference-electrode Luggin capillary, a temperature probe, and an optional drain. There is also a smaller port that accepts a 1/4"-28 fitting. During normal cell operation, this port is used for injection of de-ionized water into the flooded gasket seal.



On early versions of the cell, one of the #7 ACE-Thred™ ports is replaced with a 1/8" NPT-threaded port. This port is capped with a plastic plug (P/N 935-00022). This plug can leak. If you see a leak, drain the cell and remove the plug. Apply a piece of PTFE thread-sealant tape to the threads on the plug and replace the plug in the bottom cap.

1. Install the Temperature Probe.

The Flexcell is often used with a Gamry Instruments TDC4 in controlled-temperature studies. In this system, use the RTD probe provided with the TDC4 to sense the electrolyte temperature as near as possible to the working electrode. One of the #7 ACE-Thred™ ports in the bottom cap holds this probe.

- If you do not use the temperature probe port, you must plug it. Use a solid glass rod (P/N 930-00027) and a #7 ACE-Thred™ bushing (P/N 935-00020) with its associated O-ring.
 - If you are using the RTD probe, install it using a #7 ACE-Thred™ bushing (P/N 935-00020), a 2.5 cm long Viton® spacer (P/N 935-00044), and a special O-ring (P/N 935-00034). Do not use the O-ring provided with the #7 ACE-Thred™ bushing.
- a. Wet the spacer with a few drops of water, then slide it onto the RTD probe until you see only 2 cm of probe between the spacer and the probe's contact wire.
 - b. Slide the fitting onto the RTD probe, followed by the special O-ring.
 - c. Screw the fitting into any of the three ports on the bottom cap.
 - d. Adjust the position of the RTD probe until a few mm at the tip of the probe is seen extending into the conical well in the bottom cap.
 - e. Tighten the #7 ACE-Thred™ fitting finger tight.
You will be able to move the probe in and out within the fitting. However, the O-ring seal should remain watertight as you do so.

2. Install the Reference-Electrode Luggin Capillary.

The Flexcell uses an external reference electrode coupled to the cell via a Luggin capillary. Install the Luggin capillary into one of the #7 ACE-Thred™ ports in the bottom cap. 3-2 may be helpful.

- a) Find the reference capillary (P/N 930-00024). It may be packaged in a separate sub-assembly kit so you may have to hunt a bit for it. You also need one of the #7 ACE-Thred™ bushings (P/N 935-00020) and its associated O-ring.
- b) Slide the fitting on the capillary, followed by the O-ring.
- c) Insert the capillary into a #7 ACE-Thred™ port on the bottom cap.
- d) Tighten the fitting until it is finger-tight, then loosen about two turns. Adjust the position of the capillary tube until the tip is close to the edge of the circular hole on the bottom of the cap, and the ball joint is facing up (away from the cell's legs).
- e) Tighten the fitting finger-tight.

The rest of the reference-well assembly procedure is explained later.

3. Install the Drain Cap.

The Flexcell kit does not include drain plumbing. If you wish to add drain capability to the cell, you are responsible for providing any fittings, valves and tubing that may be required.

Any of the three #7 ACE-Thred™ ports may be used for the drain connection.

The cell kit includes a spare #7 ACE-Thred™ bushing (P/N 935-00020) and a solid glass rod (P/N 930-00027) for plugging this hole. Slide the O-ring that comes with the fitting over the rod, then insert the rod and the bushing into the spare hole. Tighten finger-tight.

4. Install the Flushed Gasket Seal Water Entry Fitting.

You should always use the Flexcell with de-ionized water flowing through the flooded gasket seal. Paradoxically, in the absence of this flow, the Flexcell suffers from crevice effects far worse than those in conventional cells. If you need to test flat-metal specimens and you don't need a true crevice-free cell, you should use a ParaCell.

Locate the 1/4"-28 barbed-hose fitting (Gamry P/N 935-00023). Screw this fitting into the smallest threaded hole on the outer edge of the bottom cap.

This fitting can leak if the threads on the barbed fitting do not exactly match those on the bottom cap. If you see a leak, drain the cell and remove the barbed fitting. Apply a tiny piece of PTFE thread-sealant tape to the threads on the fitting and replace the fitting into the bottom cap. You can find PTFE thread-sealant tape at hardware stores and plumbing-supply houses.

If you choose to disregard the warning and use the cell with no de-ionized water flow, you must cap this hole with a plastic 1/4"-28 screw.

Install the Cooling Coil.

If you need to make measurements at temperatures below ambient, you must install the cooling coil.



Your Flexcell kit may have either a glass cooling coil (P/N 930-00023) or a PTFE-coated copper cooling coil (P/N 935-00045). The glass version of the cooling coil makes the cell more fragile, so do not install it if cooling is not required.

Install the cooling coil in the glass dome (P/N 930-00016) that makes up the top of the cell. It is held in place with two #7 ACE-Thred™ bushings (P/N 935-00020) and their associated O-rings.

Install the Cooling Coil:

1. Take the glass dome in one hand and the cooling coil in the other. The coil has two straight tubes extending from its top.
2. Insert the coil, top end first, into the large open end of the dome. Aim the tubes on the coil at a pair of diagonally opposed #7 ACE-Thred™ ports on the top of the dome. Use either pair of ports. After the tubes extend from the ports, gently place the dome and coil together on a table top. When the large open end of the dome rests on the table, the tubes should still extend from the ports.
3. Carefully slide an O-ring several centimeters down each tube.
4. Place a #7 ACE-Thred™ bushing above the O-ring. Grab one tube and pull it (and the coil) up about 5 cm.
5. Tighten the #7 ACE-Thred™ bushing on the other tube. This holds the coil in place temporarily.
6. Now tighten the #7 ACE-Thred™ bushing of the first tube, locking the coil in place.

Attach the Glass Dome to the Cell.

The next step in the setup is installation of the large glass dome on the bottom cap.

1. Place the cell, legs down, on a flat, stable surface.

2. Install the large O-ring.

The large O-ring is P/N 935-00016. Install it in the bottom cap by pressing it into the groove on the upper surface of the cap. It often stays in place even when the cell is inverted.

3. Install the Quick Clamp.

Locate the metal Quick Clamp (P/N 935-00018). This clamp holds the glass dome to the bottom cap.



Caution: Place the Quick Clamp loosely on the cell **before** the dome is installed. Installing it with the dome already in place can over-stretch the clamp and increase your chance of dropping and smashing the dome.

- a) Hold the clamp with the threaded screw and locking-lever facing you. Notice the cylindrical adjustment nut on the screw. Moving this nut towards the locking lever makes the clamp smaller and the clamping force greater.
 - b) Unlatch the clamp by pulling the locking lever away from the body of the clamp. You should now be able to swing the threaded screw away from the clamp body, completely opening the clamp.
 - c) Turn the adjustment nut until it is roughly $\frac{3}{4}$ of the way toward the unattached end of the screw.
 - d) Expand the Quick Clamp slightly and slip it onto the cell. The bottom of the clamp should rest in a groove cut in the bottom cap while the top of the clamp extends over the top surface of the cap.
4. Install the Glass Dome.
 - a) Expand the clamp slightly and place the glass dome (P/N 930-00016) on the bottom cap. The large O-ring should be in position to seal on the dome's flange. Make sure that the lip on the dome is under the clamp.
 - b) Rotate the threaded screw so that the cylindrical adjustment nut engages the hooks on the other side of the gap in the clamp.
 - c) Gently lock the clamp by pushing the locking lever toward the clamp's body. The lever should move under finger pressure and snap into place. If it doesn't, readjust the position of the adjustment nut.
 - d) Check the clamp's tightness by trying to rotate the glass dome. If it rotates, but only with considerable effort, the clamp tightness is correct. If you cannot rotate it at all or if it rotates easily, readjust the position of the adjustment nut and try again.
 - e) After you have the proper tightness for the clamp, you may want to mark the current adjustment-nut position. Adhesive tape wrapped around the threaded screw is one approach. Alternatively, you can mark the position using a permanent marker.

Complete the Reference Well Assembly.

The reference electrode mounts outside the cell in a self-filling reference well. This keeps the reference from seeing high temperatures when the cell is used in elevated temperature applications.

You need three parts to complete the reference well assembly: the glass reference well (P/N 930-00017), a small plastic clamp (P/N 935-00032), and a silicone-rubber band strap (935-00038). A diagram of the completed assembly is in 3-2.

Procedure:

1. Rotate the glass dome on the cell until one of the four #7 ACE-Thred™ ports on the dome is lined up with the reference Luggin capillary (installed previously). An exact alignment is not required.
2. The band strap contains two holes. Push the smaller diameter end of the reference well through one hole in the band strap. Continue pushing the well through this hole until you almost reach the end of the glass. About 1 cm of glass should remain above the strap.
3. Push the other end of the band strap over the threaded port that is aligned with the reference capillary. When this is done properly, the ball joint on the reference well lies close to the ball-joint socket on the reference capillary.



Caution: Make sure that the fragile reference well does not bang into the cell.

4. Push the two parts of the ball joint together and clamp them with the plastic ball-joint clamp.



In normal use with aqueous electrolytes, this joint does not require stopcock grease or other sealants. A thin film of electrolyte covers the ground glass surfaces and seals the joint.

5. You may want the reference electrode even farther removed from your heated cell:
 - a) Cut the lower part of the reference well into a short piece with the ball joint attached and a longer piece still attached to the well.
 - b) Firepolish the cut ends.
 - c) Attach the cut ends to a length of Tygon® tube.
 - d) Attach the end with the ball joint to the reference capillary tube.
 - e) Place the reference well in a remotely located ringstand or other support.

Test the Partially Assembled Cell for Leaks.

The final step in initial assembly of the cell is a leak test.

Install the Drip Tube.

You can only perform this test after all the ports in the bottom cap have a plug or a fitting installed. If you have followed the installation procedures above, the de-ionized water inlet is the only port that needs attention. Rather than seal the inlet with a clamp, simply attach the tubing to be used on this inlet, and allow it to fill with water.

1. Locate the drip tube (P/N 935-00041) that is supplied with the cell. This tube, familiar to many hospital patients, includes a drop counter and regulator clamp that are nearly perfect for controlling and monitoring de-ionized water flow into the flushed port.
2. If the drip tube is still in its original packaging, it has a medical fitting on the tube end **without** the drip counter. You must remove this fitting. The easiest way is to cut the tube just above the fitting. Use scissors or a sharp knife to cut the tube about 1 cm above the fitting.
3. Open the regulator clamp by turning the adjustment wheel to the larger end of its housing.

4. Slip the cut end of the drip tube over the barbed-hose fitting on the de-ionized water inlet. The end of the tube must extend beyond the barb on the fitting. If this step is difficult, try putting a drop of water on the fitting before slipping the tube in place.

Test for Leaks.

Check the tightness of the fittings on the bottom cap. Make sure the Quick Clamp is in the locked position.

You do not require a sample for this test. The sample support seals on the small O-ring that normally seats on the metal sample. You also do not need a filter-paper washer.

1. Loop the drip tube over the top of the cell. This tube fills with water to the same level as the water in the cell.
2. Fill the cell with water, pouring the water into one of the ports on top of the glass dome. It is best if you use de-ionized water for this test, although tap water may be used if your supply of deionized water is limited. Fill the cell until at least $\frac{3}{4}$ of the glass dome is full.
3. Check all the seals for leaks, both immediately after filling, and 15 minutes after filling. You should not see any water on any fitting. Contact Gamry Instruments' technical support department if you have a leak, and you are uncertain how to remedy the problem.
4. Empty the cell by carefully inverting it over a sink and allowing the water to exit the cell from the open fittings in the glass dome.

Chapter 4: Routine Use of the Flexcell

Introduction

This chapter discusses use of the Flexcell in controlled-temperature experiments.

The previous chapter discussed initial assembly of the cell. You perform most of the assembly steps in that chapter only once, as the cell is first brought into service. This chapter also discusses assembly steps: steps that you repeat during use of the cell.

Issues such as de-ionized water flow rates, filling and emptying the cell, heating and stirring, and electrolyte deaeration are also discussed in this chapter.

Fittings in the Glass dome

The Flexcell was designed for versatility. One source of this versatility is the large number of ports on the cell's glass dome. These ports will accept a variety of accessories.

The standard cell configuration includes a counter electrode, a bubbler (sparger), and a cooling coil. You can also use these ports for a stirrer, a thermometer, an additional electrode, reagent addition, etc.

Counter Electrode

One graphite counter electrode (P/N 935-00003, 30 cm long \times 6.2 mm dia.) is provided with the Flexcell. Mount it directly in one of the four #7 ACE-Thred™ ports in the dome using a threaded bushing (P/N 935-00020) and its associated O-ring.

A second graphite counter electrode may be purchased separately and added to a second port if you require more uniform current distribution.

You can also use a noble-metal wire counter electrode. Bend one end of the wire into a hook and hang it over the lip of one of the ports. If you desire a diffusion barrier to slow counter-electrode reaction products reaching the working electrode, suspend the wire counter electrode in a fritted glass tube. The tube can enter the cell through one of the #7 ACE-Thred™ ports or a 24/40 ground-glass joint.

Gas Bubbler

A bubbler/vent tube is provided with the Flexcell. This tube fits in a 24/40 ground-glass joint. If you use both a stirrer and this bubbler tube, the stirrer enters the cell through the central 24/40 port and the bubbler is placed in the other 24/40 port.

Use of this bubbler/vent tube is described in the section of this chapter that discusses deaeration of your electrolyte.

Top-mounted reference ball joint

The glass dome is equipped with a 35/25 ground-glass ball-joint socket. Do not use this port in the "standard" version of the Flexcell, so the 35/25 port is available for expansion purposes. Your kit includes two parts to seal this port: a glass stopper (P/N 935-00028) and a plastic ball-joint clamp (P/N 935-00026). Instructions in other parts of this manual assume that you have used these pieces to seal the port.

Thermometer

You can immerse a standard glass thermometer in the cell through one of the #7 ACE-Thred™ ports. Use a threaded bushing (P/N 935-00020) and its associated O-ring.

Reference Electrode

A reference electrode is not included with the Flexcell. This omission allows you to choose the type of reference electrode appropriate for your chemistry.

Gamry Instruments currently sells two suitable reference electrodes. They are an SCE (P/N 930-00003) and a Ag|AgCl electrode (P/N 930-00015). Each is equipped with a male-pin plug connector.

You may also use other reference electrodes. Avoid commercial reference electrodes with a 15 mm outside diameter (the reference well inside diameter is 14 mm). Electrodes with a 12 mm outside diameter are less common but are available.

Regardless of the reference electrode that you are using, it is mounted in the external reference well.

Install the Metal Specimen.

The method you use for metal specimen installation depends on the thickness of the specimen. Thin specimens require a sample support that prevents bending of the specimen. Thick specimens do not require this support. In aluminum, the crossover between thick and thin specimens occurs at a thickness of about 3.2 mm ($\frac{1}{8}$ "). In stiffer metals, the crossover occurs at lower thickness.

On thin specimens, we recommend that you bend one corner of the sample at a right angle to the rest of the piece. This bent corner serves to make electrical contact with the specimen.

Installation procedure:

1. If the cell has previously been used, empty it and remove the glass dome. Rinse the top side of the bottom cap with deionized water to remove any residual electrolyte.
2. Invert the bottom cap assembly, exposing the sample holder.
3. Remove the previous sample (if any) by loosening the thumbscrew and rotating the sample-holder crossbar away from its mounting position, then lifting the sample away from the bottom cap.
4. Check the condition of the filter-paper gasket. If it is undamaged, it may be reused. If a new gasket is required, place it in the seal area. The inner edge of the gasket lies on top of a ridge in the PTFE bottom cap. The outer edge must lie entirely within the O-ring. (The part number for a package of 50 gaskets is 935-00019.)
5. Place the new sample on the seal area. Try not to slide the sample across the seal. If you are using a thin specimen with a bent corner, the bend should point away from the bottom cap.
6. If you have a thin specimen, place the sample support on the specimen. The central depression in the support should be facing away from the sample.
7. Loosen the thumbscrew on the sample-holder crossbar.
8. Place the foam insulating tile (P/N 820-00021) over the sample (and sample support if present).



If you are using the sample support, you may want to enlarge the hole in the tile to match the diameter of the support or the size of the sample. For best thermal insulation, add a second unmodified tile on top of the first. Extra tiles are available from Gamry Instruments.

9. Replace the crossbar on the legs equipped with wingnuts. The knurled portion of the thumbscrew must be pointing away from the sample.
10. Hold the crossbar in place with one hand. Tighten the thumbscrew with the other hand. The pointed end of the thumbscrew must enter the depression in the sample support (if the support is used). Do not overtighten the thumbscrew: moderate finger-pressure is all that is required.
11. Turn the cell over and replace the glass dome on the cell.

Install the De-ionized Water System.

You normally use the Flexcell with a slow flow of de-ionized water through the water inlet. This water is essential to the crevice-free, flooded seal described above. The optimal water flow rate depends on the details of your experiment. Experimentation may be needed to find the lowest flow rate at which crevice corrosion is not seen.

We recommend that you start with a flow rate of 6 mL/hour and adjust the flow rate as you gain experience with your system.

The cell kit includes a simple mechanism for control of the water flow rate. You use an “intravenous set” to gravity-feed water to the cell.

The bag from the intravenous set is normally shipped filled with sterile water. This water is not guaranteed to be de-ionized, so you must discard it. This water does however perform a function: If we shipped the bag dry, the bag’s walls could stick together.

Follow these instructions to install a de-ionized water system based on the intravenous set:

- 1) Empty the bag, rinse it, and then fill it with at least 300 mL of deionized water.
- 2) Insert the end of the tube with the drop counter into the compatible opening in the bag.
- 3) Close the control valve on the tubing by rolling the control away from the drop counter.
- 4) Raise the intravenous bag about one meter above the cell. The higher the bag, the less variation in flow rate as the experiment proceeds.
- 5) Open the control valve fully. After all the air has been cleared from the tubing (except in the drop counter itself) close the valve again.
- 6) Attach the open end of the tube to the deionized water inlet on the cell.

For more precise flow control, you can pump the water into the cell using a peristaltic pump with fine-bore tubing. Suitable pumps are available from most laboratory supply vendors. You may need to provide some form of pulse dampening if you take this approach.

Fill the Cell with Electrolyte.

Leak test the cell (with water) before filling it with electrolyte for the first time. See Chapter 3.

Check all the items on this list every time that you prepare to fill the cell:

- All the fittings on the bottom cap are tight.
- The tube for deionized water flow is attached to its fitting.
- A metal sample is properly installed on the bottom of the cell.
- The Quick Clamp is in its locked position.

You can fill the cell through any port on the glass dome. The large ball joint on the dome is generally convenient, because it is not used in the standard cell configuration.

The reference capillary and reference well should spontaneously fill with electrolyte as the cell is filled. If they do not, apply gentle suction on the top of the reference well to remove any air trapped inside the capillary.

Fill the cell until solution reaches the wide part of the reference well. This requires approximately one liter of electrolyte. Any less solution, and the reference electrode cannot contact the solution.

Avoid overfilling the cell. Remember that the de-ionized water will slowly fill the cell during the experiment. Assume that 300 mL will fill the cell from the fill point suggested above to overflowing. With a de-ionized water flow of 6 mL/hour, 300 mL is added to the cell in 50 hours.

Emptying the Cell

If you have a drain installed in the drain port in the bottom cap, emptying the cell is very simple.

1. Open the valve in the drain line and wait for the cell to empty. A few mL of electrolyte will remain in the depression in the bottom cap. This remaining solution does not prevent removal of the glass dome.
2. After you remove the dome, pour the remaining electrolyte into a waste container.

If you do not have a drain installed, you must empty the cell using one of the ports in the glass dome.

1. Remove any fittings in the standard taper ports on the glass dome. Every other port retains its fitting when the cell is tipped over so all fittings can be left in place.
2. Tip the cell over, so that the electrolyte can pour from one or both standard taper port(s).

After you have removed the dome from the bottom cap, rinse the dome, its contents, and the bottom cap with de-ionized water. A squeeze bottle filled with de-ionized water is very useful for rinsing. After you have rinsed the dome, place it on its rim on a clean surface until the next time it is needed.

Heating Jacket Installation

If the system includes an electrical heating jacket, install it on the cell **after** the cell is filled with electrolyte. The following description applies for both the 120 VAC and 240 VAC versions of the jacket.

The heating jacket wraps around the cell. It is held in place using Velcro® fasteners. The jacket must be tightly pressed against the cell. The sides of the jacket nearly touch when the jacket is installed.

Pass the jacket under the reference well (the well is still fully visible when the jacket is in place). Be careful to avoid unfastening the reference-well ball joint while you are placing the jacket around the cell.

The reference well can be unintentionally heated by the jacket. Try to keep the well from directly touching the jacket. If the well gets hot, you may need to add thermal insulation between the well and the jacket.

You may want the reference electrode even farther removed from your heated cell:

1. Cut the lower part of the reference well into a short piece with the ball joint attached and a longer piece still attached to the well.
2. Firepolish the cut ends.
3. Attach the cut ends to a length of Tygon® tube.
4. Attach the end with the ball joint to the reference capillary tube.
5. Place the reference well in a remotely located ringstand or other support.

Slide the jacket up slightly from the Quick Clamp prior to applying power to the jacket. Otherwise, the metal clamp could transfer too much heat to the bottom cap.



Caution: Never apply power to the heating jacket if the cell is not filled with water or electrolyte. The liquid level in the cell must be higher than the top of the jacket. The jacket can overheat in the absence of liquid. This can damage the cell, the heating jacket, and the temperature controller.

Electrical connections to the heating jacket are discussed in Chapter 5. The power applied to the heating jacket is normally controlled using a Gamry Instruments TDC4 Temperature Controller. Consult its Operator's Manual for detailed information concerning its use.

Additional heating instructions and safety information are given later in this chapter.

Stirrer Installation

Stirring in the Flexcell is normally done using a stirrer inserted through the central port on the top of the cell.

Gamry Instruments offers several optional accessories useful in adding stirring capability to your cell. P/N 990-00117 is a stirring-rod kit. It includes:

- A precision glass stirring rod
- A stirring paddle that attaches to the rod
- A bushing that guides the stirring rod through a 24/40 ground-glass port

If you are using the stirring-rod kit with your cell, install it after electrolyte has been added to the cell.

- 1) Note that the bottom end of the stirring rod is bent and has a glass disk on it. The other end of the rod is the "top end."
- 2) Slide the stirring paddle from the top end of the glass stirring rod all the way to the bottom end.
- 3) Rotate the paddle so its long axis is parallel with the rod.
- 4) Poke the bottom end of the rod and the paddle through the central 24/40 standard taper port in the glass dome.
- 5) Carefully push the paddle against the bottom of the cell to rotate the paddle to a position perpendicular to the stirring rod.
- 6) Loosen the nut on the bushing (if necessary) and slide the bushing over the top of the stirring rod.
- 7) Push the bushing down the rod until the bushing seats in the port on top of the glass dome.



Caution: The bushing can overheat if the rotation speed exceeds 200 rpm.
Always keep the stirring rate lower than this maximum value.

You can use the stirring rod kit with a variety of stirring motors. The details of connection to a stirring motor can vary. Consult the manufacturer's documentation for specific instructions for your motor and controller. Gamry Instruments sells an optional stirrer-motor kit (P/M 990-00118) that allows precise control of the stirring apparatus in the stirring-rod kit. More information can be found in Chapter 5.

Clamp the Flexcell to a sturdy ringstand whenever it is used with a stirrer.

You can remove the stirring rod assembly from the cell by reversing the steps above. Removal of the stirring rod from the top of the cell with the paddle attached can be quite tricky. The paddle rotates quite easily, so it is difficult to keep it aligned with the rod as you pull it from the cell. Be patient; we have found that with practice this can be done.

If you find removal of the stirring rod too difficult, disassemble the cell with the stirring rod still in place, and remove the stirring rod from the bottom of the cell.



Caution: The stirring rod is easily broken. Take care if you must remove the dome with the rod in place.

Electrode Connections

If you are using your Flexcell with a Gamry Instruments, Inc. potentiostat, make the following connections to the electrodes:

1. Plug the reference-electrode lead into the white pin jack on the cell cable.
2. Attach both the green and blue leads from the cell cable to the working electrode (the metal specimen).
 - If you are using a thick specimen without the sample support, clip the alligator clip on each of these leads to the threaded portion of the thumbscrew.
 - If you are using a thin specimen with the sample support, clip these leads directly to the metal. If you have bent one corner of the sample, clip to the bent area.

Some potentiostats may not have a blue cell lead. In this case, simply connect the green lead to the working electrode.

3. Clip the red lead on the cell cable to the counter electrode.
4. Make sure that the floating ground (the longer 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, reduces noise in your experimental results.
5. If you are measuring very small currents, a metal enclosure completely surrounding your cell may further reduce noise. In this case, connect this shield, known as a Faraday shield, to both ground leads (short and long black leads) in the cell cable.



Always double-check your cell connections. Even an experienced experimenter occasionally leaves one of the cell-cable leads lying on the lab bench.

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

Controlling the De-ionized Water-flow Rate

For gravity feed using the intravenous set, you control the flow rate using the control valve that came with the set. You can measure the flow with the drop counter. Each drop in the drop counter corresponds to 0.1 mL.

Set the flow rate with the cell filled with electrolyte and with a metal sample in place. Both the pressure from the height of fluid and the tightness of the sample mounting affect the flow rate.

We recommend that you start with a flow rate of 6 mL/hour or one drop per minute. On one system that we studied, varying the flow rate by a factor of two from this setting had no serious consequences. However, test systems vary. The optimal flow rate for your particular chemistry could be quite a bit different.

There are some limits on the flow rate. Fast flow rates can unnecessarily dilute the electrolyte in the cell, affecting the electrochemistry. Very slow flow may not produce a good flooded seal, with the possibility of crevice corrosion.

Your flow rate will normally vary somewhat with time and with the levels in both the bag and the cell. If you wish, you can monitor and periodically readjust the flow rate. In most cases, however, the variation should be small enough to be of no consequence.

Heating, Cooling, and Stirring

The Flexcell offers two means for altering the temperature of your tests.

Temperature-control via the Cooling Coil

The first means is the cooling coil that is included with the cell. Although we refer to this as a cooling coil, you can also use this coil to heat the cell's contents. The second means of temperature alteration is an external electric heating jacket. Both 110 VAC and 220 VAC versions of the heating jacket are available as options from Gamry Instruments. See Chapter 5 for details.

You must provide an air vent whenever the cell is heated, otherwise expansion of the cells contents can pressurize the cell with potentially explosive results. The vent on the gas bubbler is a suitable escape path if blanketing gas is not used. If blanketing gas is used, you **must** provide an alternate vent.



Warning: Make sure that there is a path for gas to escape a heated cell. Failure to vent the cell can cause dangerous conditions, including danger of an explosion. The possible consequences include **severe injury and even death.**



Caution: We do not recommend unattended operation of a heated cell without one or more fail-safe systems to prevent overheating the system. A variety of thermal cutoff switches are available from laboratory supply houses.

The cooling coil allows heating or cooling via heat exchange with a liquid flowing through the coil. If the cell is to be both heated and cooled via this coil, a recirculating temperature bath is a suitable source of controlled-temperature fluid. Gamry Instruments' Critical Pitting Test software allows computer control of several Thermo Scientific baths.

Heat-transfer through the cooling coil is quite slow. This can limit both the rate at which temperature can be changed and the precision of the temperature control.

Temperature-control via an External Heating Jacket

Heating via the external heating jacket lessens these problems. The Gamry Instruments TDC4 Temperature Controller works with the Critical Pitting Temperature software and a heating jacket for optimal controlled-temperature-pitting testing.



Caution: Never apply power to the heating jacket if the cell is not filled with water or electrolyte above the top of the jacket. The jacket can overheat in the absence of water. This can damage the cell, the heating jacket, and the temperature controller.

The heating jacket is not designed for operation when wet. Make sure that the jacket does not come in contact with water or electrolyte.



Warning: Never operate the heating jacket when it is wet. A wet heating jacket creates a significant danger of electrical shock. Allow the jacket to dry thoroughly if it does become wet.

Usage at High Temperatures

The Flexcell has been designed for high-temperature operation. Many electrolytes will not boil until the temperature is well above 100°C. When the cell is operated at high temperature, many exposed surfaces of the cell become dangerous. Avoid contact with any portion of the cell when it is operating at an elevated temperature.



Warning: Avoid touching any portion of the cell when it is operated at elevated temperature. Painful burns can occur.

Usage at Low Temperatures

If you need operation below ambient temperature, a flow of cold water through the cooling coil can be used in conjunction with the heater. If you turn the water flow on and off using a solenoid valve under TDC4 control, you can greatly improve the thermal response time of the system.



If your tap water is not cold enough, you can place the water hose in an ice bath.

Temperature gradients in the cell can be a problem. A significant temperature difference can arise between the tip of the RTD, where temperature is measured and controlled, and the surface of the metal sample.

Temperature Gradients within the Cell

Stirring greatly reduces the magnitude of temperature gradients. We recommend stirring whenever temperature control to better than 5°C accuracy is desired and whenever the temperature is stepped or ramped.

Thermal insulation can also help reduce temperature gradients. One or more foam insulating tiles (P/N 820-00021) can greatly slow heat-loss from the sample, lowering the temperature gradient in the critical area near the sample.

If your system contains a TDC4 Temperature Controller, you can attach a thermocouple to the dry side of the metal specimen and use the TDC4 to read its temperature directly. A comparison between the set-point

temperature and the sample temperature allows you to develop a calibration curve for temperature-correction in your experimental system. This calibration curve changes as the experimental conditions change.

Electrolyte Deaeration

Many corrosion mechanisms involve the reduction of oxygen. If the “real-world” system you are studying is saturated with air, conduct your tests without deaerating your electrolyte.

On the other hand, if the system you are studying is oxygen-free, do your tests after deaeration of the test electrolyte. The Flexcell includes a means for deaerating the electrolyte by passing an electrochemically inert gas (such as nitrogen) through the solution. The oxygen in the solution is swept away in the stream of inert gas.

P/N 930-00018 is a glass bubbler/vent. You install it in one of the 24/40 ports on the glass dome. If you use a stirrer, it occupies the central port, so you use the other 24/40 port for the bubbler.

In normal use, you attach an inert gas hose to the central tube on the bubbler. Use a precision gas-flow valve to control the gas-flow rate. You can vent the gas, after it bubbles through the solution, through the second port on the bubbler. The deaeration time depends on a number of factors including gas-flow rate, temperature, stirring rate, and solution composition. Allow at least 15 minutes for complete deaeration, even under optimal conditions.

Some workers do not like gas bubbling through the solution during data acquisition. In order to prevent reabsorption of oxygen in their electrolyte, they blanket the electrolyte with inert gas after the initial deaeration. You can adapt the bubbler/vent tube for this purpose by flowing gas through the central bubbler port during initial deaeration and through the second bubbler port during blanketing. If you do this, you **must** provide a means for the gas to escape the cell.



Warning: Make sure that there is a path for deaeration gas to escape the cell. If no gas exit path exists, the entire glass dome can be pressured causing a dangerous condition. Failure to vent the cell can cause dangerous conditions, including danger of an explosion. The possible consequences include **severe injury and even death.**

A small piece of string or a rolled-up piece of PTFE pipe-sealant tape in the ball joint on the cell provides an escape path for the deaeration gas.

Electrode Area

It is very difficult to assign an electrode area to the cell when it is operating with a flushed seal. The nominal inside diameter of the filter paper gasket is 2.52 cm (0.993 in). The nominal area is therefore 5.00 cm² (0.775 in²).

In practice, the active area is bordered by a concentration gradient, so it does not have an exact area.

Chapter 5: Accessory Equipment

Critical Pitting Test Systems

The Flexcell is most often used with Gamry Instruments' Critical Pitting Test System. This application that runs under the Gamry Framework™ software is used to determine the temperature at which pitting occurs in a passivated metal/solution system.

The following pieces are required for a CPT system:

- An IBM-compatible computer
- A Gamry Instruments potentiostat
- A temperature controller
- A crevice-free cell
- A means for heating and cooling the cell's contents
- Gamry Framework software
- CPT software

In general, any IBM-compatible PC can be used if it is capable of holding the potentiostat and it can run Microsoft Windows®. Consult the Gamry Framework, CPT, and potentiostat Operator's Manuals for detailed computer specifications.

All Gamry potentiostats available currently (PC4, PC5, Series G, and Reference Family) are usable with the CPT.

The CPT can be used with one of three temperature controllers:

- The Gamry Instruments TDC4
- The Gamry Instruments TDC2 (no longer being sold)
- The controller integrated into Thermo Scientific™ recirculating baths (RTE series with RS-232 I/O and remote temperature sensing).

A cell can be cooled with tap-water flow or a recirculating bath. Three forms of cooling control can be used:

- No cooling control; the heater is used to overpower the cooling
- Solenoid valve on/off control; typically used with cold water flow
- True PID control of cooling in a recirculating bath

Consult the documentation for the CPT and TDC4 or TDC2 for more details on system configurations.

Heaters

Introduction

In the interests of flexibility, a heater is not included with either the Flexcell or with any Gamry Instruments temperature controller. However, Gamry does offer Flexcell-compatible heating jackets as options at extra cost.

Two different heating jackets are available: one rated for 120 VAC operation (P/N 990-00115) and the other rated for 240 VAC (P/N 990-00119).

Both heating jackets are designed for duty-cycle modulation as the means of temperature control. In plain English, controlled temperature is achieved by controlling the fraction of time that AC power is applied to the heater. If the cell requires very little heat-input, the AC power to the heater is off most of the time. When more

heat is required, the controller turns on the AC power more of the time. Using this control method, the voltage applied to the heating jacket doesn't vary. It remains fixed at the AC power-line voltage.

The heating jackets can be used with any duty-cycle-modulating controller. The Gamry Instruments TDC4 and TDC2 are two such controllers. You may already have a suitable controller in your laboratory.

The heating jackets are equipped with a short, permanently attached electrical connection. A removable cord is also provided. One end of this removable cord mates with the connector on the heating jacket. The other end terminates in a US 120 VAC or 240 VAC connector.

Unfortunately, this "US" end is often incompatible with the controller that will be used with the system. Any connector that we choose for this end will be wrong for many, if not most, of our customers. In many cases, you will have to modify the cord, changing this connector to the type that is required by your controller.

Modification of the Heater Cord for Use with the TDC2



Warning: This procedure should only be performed by a qualified electrician or electronics technician. Improper addition of the new connector to the cord could create a hazardous condition with a severe risk of injury or even death.

The heating jacket is shipped with an add-on connector that can be used to modify this cord for use with the TDC2. This connector is added to the cord as follows:



Caution: These assembly instructions apply to the Shurter Model 8143 connector. They may not apply to other connector models.

- 1) Identify the end of the cord that plugs into the heating jacket. Cut off the other end of the cord.
- 2) Disassemble the IEC male connector (P/N 720-00040) that was provided with the heating jacket. You need to remove three screws.
- 3) Slide the rubber hood onto the cut end of the cord, with the tapered end pointing toward the remaining connector. Stop sliding when approximately 2.5 cm of cable extends from the hood.
- 4) Strip approximately 1.5 cm of outer insulation from the cable. Expose the pair of wires.
- 5) Strip approximately 0.5 cm of insulation from each of the two wires.
- 6) Loosen the screws on both the H and L terminals in the connector until the screws almost fall out.
- 7) Poke the stripped end of the wires into the circular holes under the screws. The black wire goes into the hole marked H and the white wire into the hole marked L.
- 8) Tighten the screws on the terminals. Make sure that both wires are secure. Check that there are no wire whiskers that can short the wire to another terminal.
- 9) Slide the hood (from step 2) into place on the connector. The ridge on the hood should slip into a groove on the connector body.
- 10) Install the small strain-relief bracket in the connector. This bracket is held in place by the two shorter screws.

11) Reassemble the connector, using the longer screw to hold the two sides of the connector together.

Using the Heating Jackets

Always use the heating jacket with a closed-loop temperature-control system. Never plug it directly into an AC wall output: It is too easy to become distracted and walk away from your cell, which can quickly overheat.

Installation of the heating jacket on the cell is described in Chapter 4.

Always turn off the temperature controller attached to the heating jacket before the jacket is removed from the cell and/or the cell is emptied.



Caution: The heating jacket must only be powered when it is attached to a cell filled with a liquid above the level of the jacket. Operation without a filled cell can damage the heating jacket and/or temperature controller.

The heating jacket is not designed for operation when wet. Make sure that the jacket does not come in contact with water or electrolyte.



Warning: Never operate the heating jacket when it is wet. Allow the jacket to dry thoroughly if it does become wet.

Be careful to avoid contact with the heating jacket when it is powered. The exterior surface of the jacket can become very hot.



Warning: Painful burns can result from touching the jacket when it is under power.

Stirrer

P/N 990-00117 is a stirring-rod kit. It can be used with a variety of stirring motors. The stirring rod kit includes:

- A precision 10 mm glass stirring rod (P/N 930-00020)
- A stirring paddle for attachment to the rod (P/N 935-00024)
- A bushing that guides the stirring rod through a 24/40 ground-glass port (P/N 930-00019)

Installation of this stirring-rod kit into the cell was discussed in Chapter 4.

Many labs already own a stirring motor and controller that can be used with the stirring rod kit. A flexible connection between the rod and the motor is recommended. Make sure that the stirring rate does not exceed 200 rpm.



Caution: The bushing can overheat if the rotation speed exceeds 200 rpm. Always keep the stirring rate lower than this maximum value.

The stirring motor must be mounted on a ring stand or other strong support. You may want to clamp the cell itself to the same stand.

Gamry Instruments sells a stirring motor kit (P/N 990-00118) as an option at extra cost. This kit includes a stirring motor and controller, a flexible coupling to connect to the stirring rod, and a heavy-duty ring stand. The motor and controller in this kit operate from 120 VAC only. Consult your local Gamry Instruments representative if you need a 240 VAC stirring motor kit.

Chapter 6: Troubleshooting

This section of the manual is a list of problems that you may encounter. Following each problem are possible causes for that problem. Neither the list of problems nor the lists of their causes is comprehensive.



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 reference electrode or the Luggin capillary.
- Two or more of the cell leads are shorted together.

Noisy cell current or open-circuit potential

- 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's surface.
- There is a gas bubble in the reference electrode or the Luggin capillary.

Excess back pressure required to bubble deoxygenation gas

- No path is available for the gas to escape.

Poor experimental reproducibility

- 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.

Appendix A: Specifications

Volume

Volume	1000 mL minimum
	1300 mL maximum

Metal Specimen

Active Area	5.00 cm ² (0.775 in ²) nominal
Minimum Size	4.0 cm × 4.0 cm
Maximum Size	7.5 cm × any length or 11.0 cm × 11.0 cm (rectangular) 11.0 cm diameter (circular)

Temperature Range

Operating	−25°C to +140°C
Storage	−40°C to +180°C (not assembled)

Chemical Compatibility

Wetted Materials (electrolyte)	Pyrex, PTFE, Filter Paper
Wetted Materials (deionized water only)	Polypropylene, Tygon [®]
Non-Wetted Materials	List above, plus stainless steel, Viton [®] , silicone rubber

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