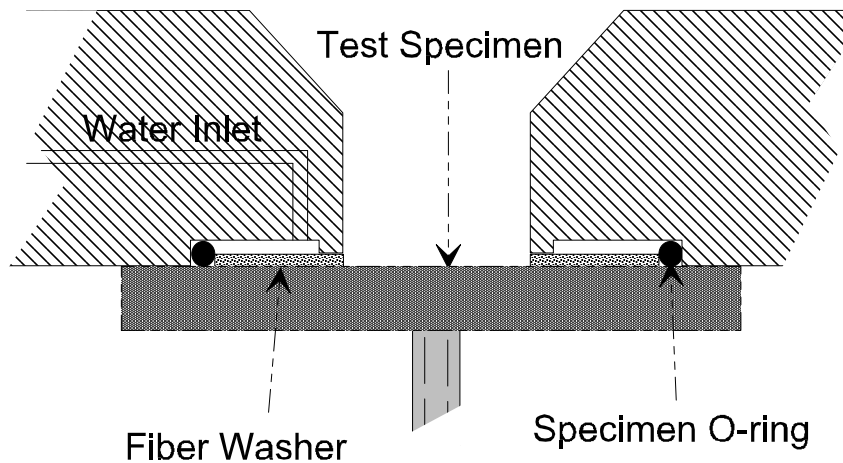




# Flexcell Critical Pitting Test Cell Kit

## Operator's Manual



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## If You Have Problems

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

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

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

Replacement parts for this kit are available from Gamry Instruments, Inc. We do not supply standard metal specimens.

## Disclaimer

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**Flexcell Kit**  
**CPT Version**  
**Operator's Manual**

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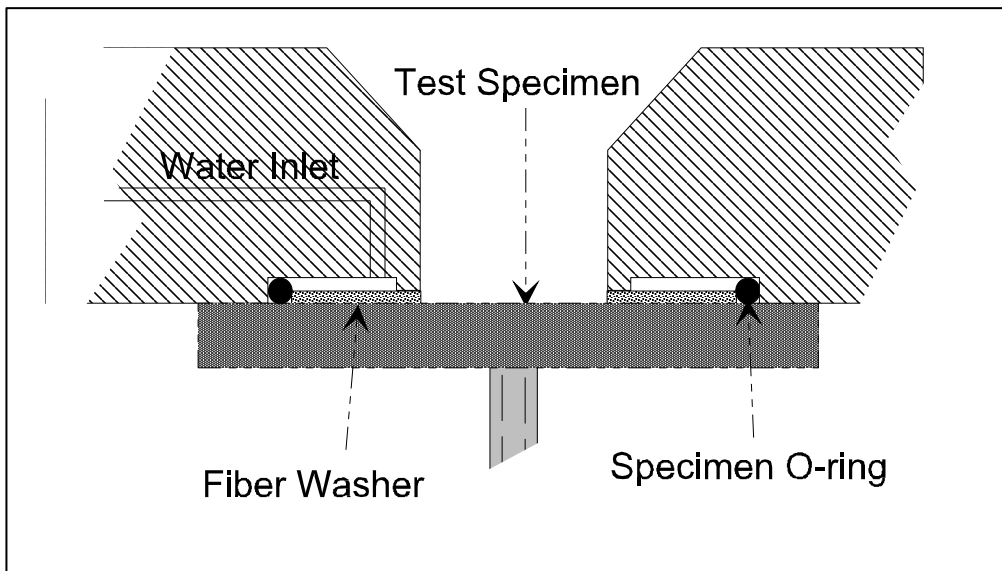
## Chapter 1 -- Introduction

The Gamry Instruments, Inc. Flexcell is a versatile electrochemical test cell intended for use in a number of different configurations. This revision of the Flexcell manual describes only one such configuration, referred to as the CPT version (CPT stands for Critical Pitting Temperature). This is the only Flexcell configuration available at the time this is being written. Consult your local Gamry Instruments representative for information concerning other configurations.

The CPT version of 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 section diagram of the cell in the area of the flooded seal.

**Figure 1**  
**Cross Section View 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.

Deionized 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 TFE (Teflon<sup>®</sup>). The cell is therefore usable with a wide variety of test solutions, including nonaqueous media.



## 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" by 2" by 1/8" (approximately 5 cm x 5 cm x 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 CPT 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](mailto: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**

To prevent shipping damage, your Flexcell Kit has been shipped disassembled. Most of the cell components are individually wrapped and packaged in small boxes within the larger shipping carton.

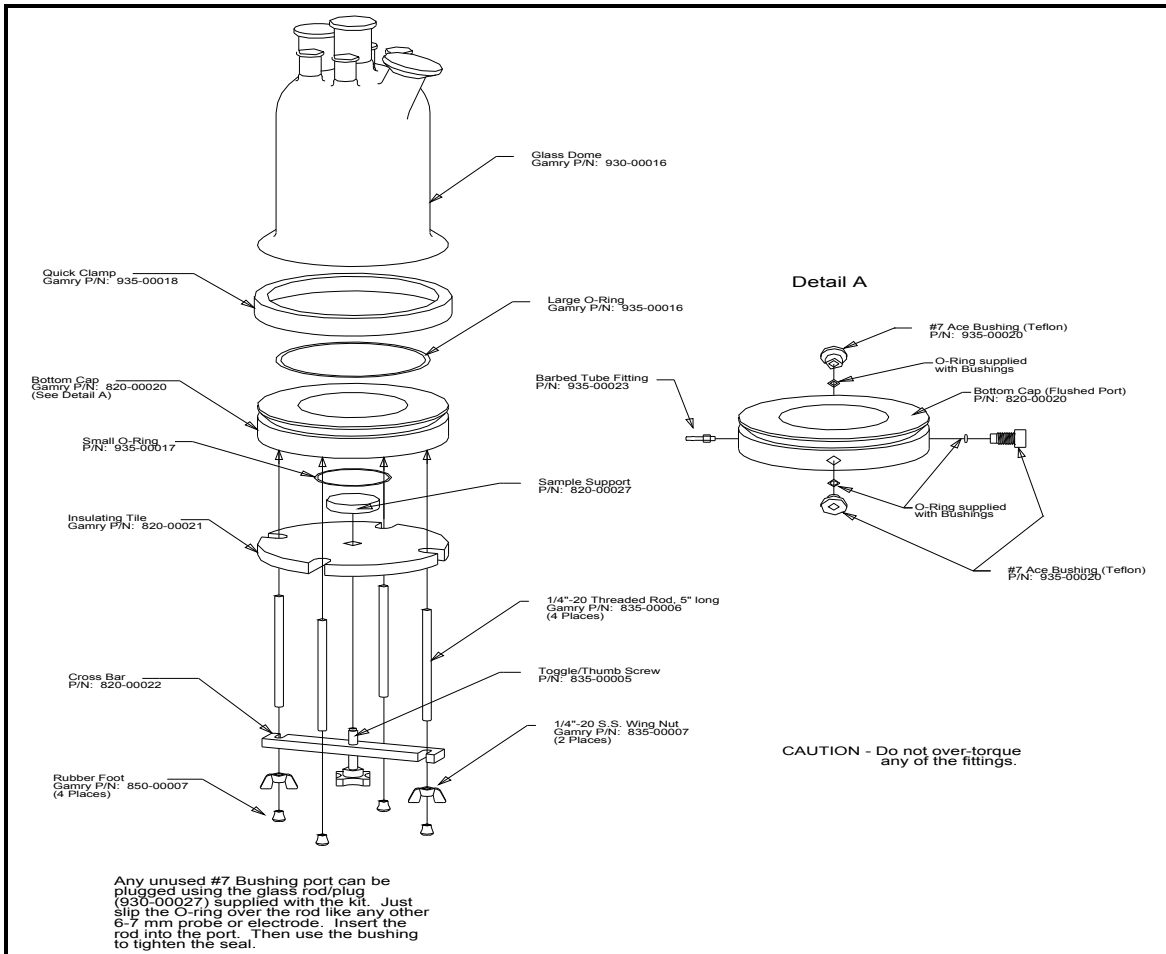
All of the pieces have been carefully packaged in anticipation of rough handling in shipment. Unfortunately, no matter how carefully glass pieces are packaged, damage will sometimes occur.

When you first receive your Flexcell Kit, please unwrap it completely and check it for any signs of shipping damage. Be especially careful if the shipping container shows signs of rough handling.

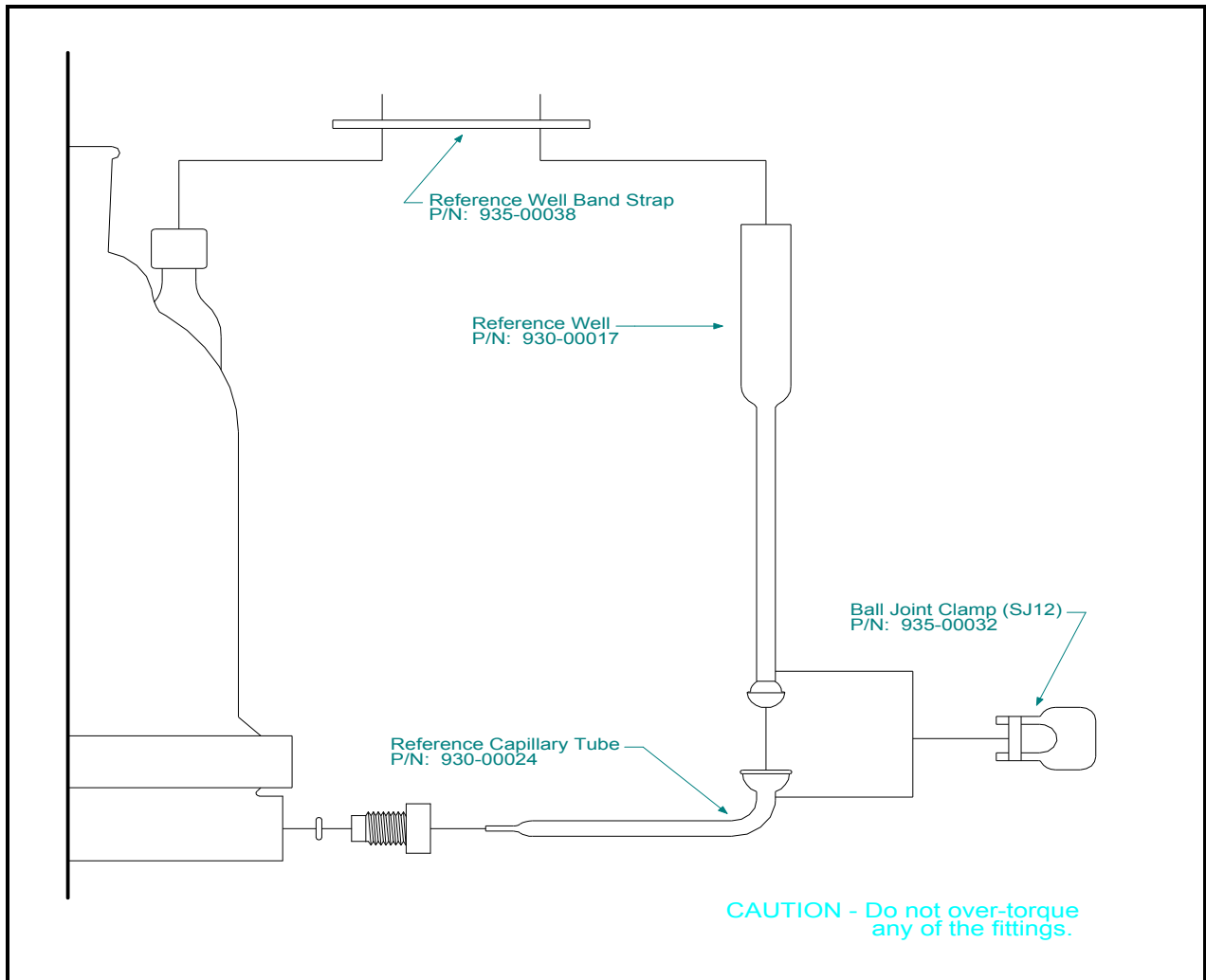
Obviously, the glass pieces are the most susceptible to damage. Check the glass pieces for chipping and small cracks as well as for major damage.

If any parts have been broken in shipment, please contact us as soon as possible for replacement. Our phone number and address are located just inside the Title page of this manual. Please retain the shipment's box and packing material for a possible claim against the shipping company.

**Figure 3-1**  
**Flexcell -CPT Assembly**



**Figure 3-2**  
**Flexcell -CPT Reference Well Assembly**



## Checking versus the Cell's 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, 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, it can often be identified by referring to Figure 3-1 and Figure 3-2. These figures are also useful if you must check the completeness of an older kit.

## Assembly Process

Assembly of the cell is quite simple and 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.

### Bottom Cap - Leg installation

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

#### Leg installation

Lay the bottom cap (P/N 820-00020) on a table with the face containing the conical depression facing the table top.

Locate 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.

#### **Warning**

**Do not use tools to screw the rods in place. Over tightening the rods can damage the bottom cap.**

#### Wingnut Installation

Locate the two wingnuts (P/N 835-00007). Screw one wingnut, with the flat surface facing down, about 1/2 way down one of the cell's threaded legs. The exact position on the leg is not important. Install the second wingnut the same distance down the diagonally opposite leg.

#### Rubber Feet

Press one rubber foot (P/N 850-00007) on the exposed end of each cell leg.

Invert the cell and place it on a flat surface, with the four rubber feet touching the surface. 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 isn't stable, the length of one or more legs can be adjusted by unscrewing them slightly.

## Sample Holder Assembly

The sample holder mounts underneath the cell. It is held in place by the wingnuts mounted on the cell's legs. Again, place the bottom cap on a table top with the conical depression facing the table.

### Thumbscrew Installation

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.

### O-Ring Installation

Install the Teflon coated silicone rubber o-ring (P/N 935-00017) on the cell's bottom cap by pressing it into the groove on the visible side of the bottom cap.

### Crossbar and Sample Support Installation

Locate the sample support (P/N 820-00027). It looks like a small hockey puck. Note the depression in one face of the support.

Place the sample support, depression up, on the o-ring installed in the previous step.

Hold the crossbar with the pointed end of the thumbscrew pointing downwards. Slide the crossbar between the legs on the cell, below the wingnuts. Rotate the crossbar so that each slot in the crossbar engages one of the legs that has a wingnut on it. A glance at Figure 3-1 may be helpful.

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 will be pressed against the wingnuts on the legs and the thumbscrew will press the sample support against the cell.

### **Warning**

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

## Bottom Cap Fittings

The rounded outer surface of the bottom cap contains four threaded ports. Three ports are designed to accept #7 thread Ace bushings. During normal cell operation these ports are used for the reference electrode Lugin 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 deionized water into the flooded gasket seal.

### **Note**

**On early versions of the cell, one of the #7 Ace thread 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 Teflon thread sealant tape to the threads on the plug and replace the plug in the bottom cap.**

### Temperature Probe Installation

The CPT version of the Flexcell is often used in conjunction with a Gamry Instruments TDC4 in controlled temperature studies. In this system, you 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 ports in the bottom cap holds this probe.

If you will not be using the temperature probe port, you must plug it. Use a solid glass rod (P/N 930-00027) and a #7 Ace bushing (P/N 935-00020) with its associated o-ring.

If you are using the RTD probe, you install it using a #7 Ace 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). You do not use the o-ring provided with the Ace bushing.

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. Now slide the fitting onto the RTD probe, followed by the special o-ring. Screw the fitting into any of the three ports on the bottom cap. 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. Tighten the Ace 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.

#### Reference Electrode Lugin Capillary Installation

The CPT version of the Flexcell uses an external reference electrode coupled to the cell via a Lugin capillary. You install the Lugin capillary into one of the #7 Ace ports in the bottom cap. Figure 3-2 may be helpful.

Locate 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 bushings (P/N 935-00020) and its associated o-ring.

Slide the fitting on the capillary, followed by the o-ring. Insert the capillary into a #7 Ace port on the bottom cap. 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). Tighten the fitting finger tight.

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

#### Drain Cap

The Flexcell CPT 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 ports can be used for the drain connection.

The CPT cell kit includes a spare Ace 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.

#### Flushed Gasket Seal Water Entry Fitting

You should always use the CPT version of the Flexcell with deionized water flowing through the flooded gasket seal. Paradoxically, in the absence of this flow, the CPT version of 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 different version of the Flexcell, currently under development.

Locate the 1/4" x 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 do see a leak, drain the cell and remove the barbed fitting. Apply a tiny piece of Teflon thread sealant tape to the threads on the fitting and replace the fitting into the bottom cap. You can obtain Teflon thread sealant tape at hardware stores and plumbing supply houses.

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

### **Cooling Coil**

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

Note: You Flex Cell kit may have either a glass cooling coil (P/N 930-00023) or a Teflon coated copper cooling coil (P/N 935-00045). The glass version of the cooling coil makes the cell more fragile, so you should not install it if cooling is not required.

You 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 bushings (P/N 935-00020) and their associated o-rings.

#### Cooling Coil Installation

Take the glass dome in one hand and the cooling coil in the other. The coil has two straight tubes extending from its top.

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 threaded ports on the top of the dome. Use either pair of ports. Once 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.

Carefully slide an o-ring several centimeters down each tube. Place an Ace bushing above the o-ring. Grab one tube and pull it (and the coil) up about 5 centimeters. Tighten the Ace bushing on the other tube. This will hold the coil in place temporarily. Now tighten the Ace bushing of the first tube, locking the coil in place.

### **Attaching the Glass Dome to the Cell**

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

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

#### Large O-ring Installation

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 will often stay in place even when the cell is inverted.

#### Quick Clamp Installation

Locate the metal quick clamp (P/N 935-00018). You use this clamp to hold the glass dome to the bottom cap. Place it loosely on the cell before the dome is installed. Installing it with the dome already in place can a) over-stretch the clamp and b) increase your chance of dropping and smashing the dome.

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.

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. Turn the adjustment nut until it is roughly 3/4 of the way toward the unattached end of the screw.

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.

#### Glass Dome Installation

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.

Rotate the threaded screw so that the cylindrical adjustment nut engages the hooks on the other side of the gap in the clamp. 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.

Check the clamp's tightness by trying to rotate the glass dome. If it rotates, but only with considerable effort, the clamp tightness is OK. If you cannot rotate it at all or if it rotates easily, readjust the position of the adjustment nut and try again.

Once you have the proper clamp tightness, 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.

### **Completing 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. They are the glass reference well (P/N 930-00017), a small plastic clamp (P/N 935-00032) and a silicone rubber band strap (935-00038). You can see a diagram of the completed assembly in Figure 3-2.

#### Reference Well Assembly Procedure

Rotate the glass dome on the cell until one of the four #7 Ace ports on the dome is lined up with the reference Lugin capillary (installed previously). An exact alignment is not required.

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.

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 will lie close to the ball joint socket on the reference capillary.

#### **Warning**

**Make sure that the fragile reference well doesn't bang into the cell.**

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 will not require stopcock grease or other sealants. A thin film of electrolyte will cover the ground glass surfaces and seal the joint.

**Hint**

**You may want the reference electrode even farther removed from your heated cell. You can do so by cutting 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.**

**Firepolish the cut ends. Attach the cut ends to a length of tygon tube. Attach the end with the ball joint to the reference capillary tube. Place the reference well in a remotely located ringstand or other support.**

## **Leak Testing the Partially Assembled Cell**

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

### **Drip Tube Installation**

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 deionized water inlet is the only port that needs attention. Rather than seal the inlet with a clamp, simply attach the tubing that will be used on this inlet and allow it to fill with water.

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 deionized water flow into the flushed port.

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.

Open the regulator clamp by turning the adjustment wheel to the larger end of its housing.

Slip the cut end of the drip tube over the barbed hose fitting on the deionized 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.

### **Leak Test**

Check the tightness of the fittings on the bottom cap. Make sure the quick clamp is in the locked position.

You don't require a sample for this test. The sample support will seal on the small o-ring that normally seats on the metal sample. You also don't need a filter paper washer.

Loop the drip tube over the top of the cell. This tube will fill with water to the same level as the water in the cell.

Fill the cell with water, pouring the water into one of the ports on top of the glass dome. It's best if you use deionized water for this test, although tap water can be used if your supply of deionized water is limited. The cell should be filled until at least 3/4 of the glass dome is full.

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.

You can 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 CPT Version of the Flexcell

### Introduction

This chapter discusses use of the CPT version 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 deionized 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) is provided with the CPT version of the Flexcell. Its dimensions are 30 cm long and 6.2 mm in diameter. You mount it directly in one of the four #7 Ace 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 more uniform current distribution is required.

You can also use a noble metal wire counter electrode. Simply bend one end of the wire into a hook and hung 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 ports or a 24/40 ground glass joint.

#### Gas Bubbler

A bubbler/vent tube is provided with the CPT version of the Flexcell. This tube fits in a 24/40 ground glass joint. If both a stirrer and this bubbler tube are used, 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 mount reference ball joint

The glass dome is equipped with a 35/25 ground glass ball joint socket. You don't use this port in the "standard" CPT version of the Flexcell, so it is available for expansion purposes. It will be used in other versions of the Flexcell.

Your kit includes two parts to seal this port. They are 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 ports. Use a threaded bushing (P/N 935-00020) and its associated o-ring.

## Reference Electrode

A reference electrode is not included with the CPT version of 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 silver/silver chloride electrode (P/N 930-00015). Each is equipped with a male pin plug connector.

You can also use other reference electrodes. Avoid commercial reference electrodes with a 15 mm OD (the reference well ID is 14 mm). Electrodes with a 12 mm OD are less common but are available.

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

## Metal Specimen Installation

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 (1/8"). In stiffer metals, the crossover will occur 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 will be used to make electrical contact with the specimen.

The sample installation procedure is:

- 1) If the cell has previously been in use, 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 Teflon 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).

### Hint

**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 thumb screw 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 over tighten the thumbscrew. Moderate finger pressure is all that is required.
- 11) Turn the cell over and replace the glass dome on the cell.

## Deionized Water System Installation

You normally use the CPT version of the Flexcell with a slow flow of deionized water through the water inlet. This water is essential to the crevice free, flooded seal described above. The optimal water flow rate will depend on the details of your experiment. Experimentation may be needed to find the lowest flow rate at which crevice corrosion isn't 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 deionized, 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 deionized 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. Once 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.

## Filling the Cell with Electrolyte

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

You should 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 has been 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 in the capillary.

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

Avoid overfilling the cell. Remember that the deionized 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 deionized water flow of 6 ml/hour, 300 ml will be 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. 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. Once the dome has been removed, you can 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. Remove any fittings in the standard taper ports on the glass dome. Every other port will retain its fitting when the cell is tipped over so their fittings can be left in place. Tip the cell over, so that the electrolyte can pour from one or both standard taper port(s).

Once you have removed the dome from the bottom cap, you can rinse the dome, its contents, and the bottom cap with deionized water. A squeeze bottle filled with deionized water is very useful in this regard. Once you have rinsed the dome, you can 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, you should 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 simply 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 will 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.

### Hint

**You may want the reference electrode even farther removed from your heated cell. You can do so by cutting 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.**

**Firepolish the cut ends. Attach the cut ends to a length of tygon tube. Attach the end with the ball joint to the reference capillary tube. 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.

### Warning

**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/or 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 is given later in this chapter.

## Stirrer Installation

Stirring in the CPT version of 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 will be referred to as 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.

### **Warning**

**The bushing can overheat if the rotation speed exceeds 200 rpm. Always adjust the stirring rate lower than this 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/or 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.

The Flexcell should be clamped 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's 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.

Be careful, 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, you must make the following connections to the electrodes.

Plug the reference electrode lead into the white pin jack on the cell cable.

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.

Clip the red lead on the cell cable to the counter electrode.

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, will reduce noise in your experimental results.

If you are measuring very small currents, a metal enclosure completely surrounding your cell may further reduce noise. In this case, connect 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 will occasionally leave one of the cell cable leads lying on the lab bench.

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

## Controlling the Deionized 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 due to 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. However, in most cases the variation should be small enough to be of no consequence.

## Heating, Cooling and Stirring

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

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 cell's contents can pressurize the cell with potentially explosive results. The vent on the gas bubbler is a suitable escape path if blanketing gas will not be used. If blanketing gas is used, an alternate vent must be provided.

**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.**

**Warning**

**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 and/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' CPT110 Critical Pitting Test software allows computer control of several Neslab 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.

Heating via the external heating jacket lessens these problems. The Gamry Instruments TDC4 Temperature Controller works with the CPT110 CPT software and a heating jacket for optimal controlled temperature pitting testing.

**Warning**

**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/or 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**

**Do not 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.**

The CPT version of 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.**

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.

**Hint**

**If your tap water isn't 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.

Stirring greatly reduces the size 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.

**Hint**

**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 will allow you to develop a calibration curve for temperature correction in your experimental system. This calibration curve will change 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, your tests should probably be conducted without deaerating your electrolyte.

On the other hand, if the system you are studying is oxygen free, your tests should be done after deaeration of the test electrolyte. The CPT version of 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 deaeration gas has a path to escape the cell. If no gas exit path exists, the entire glass dome can be pressured causing a dangerous condition.**

A small piece of string or a rolled up piece of Teflon 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 cm<sup>2</sup> (0.775 in<sup>2</sup>).

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



## Chapter 5 -- Accessory Equipment

### CPT110 CPT Systems

The CPT version of the Flexcell is most often used with Gamry Instruments' CPT110 Critical Pitting Test System. This application that runs under the Gamry Framework is used to determine the temperature at which pitting occurs in a passivated metal/solution system.

The following pieces are required for a CPT110 system.

- An IBM compatible computer
- A Gamry Instruments potentiostat
- A temperature controller
- A crevice free cell
- A means for heating/cooling the cells contents
- Gamry Framework software
- CPT110 CPT software

In general, any IBM compatible PC can be used if a) it is capable of holding the potentiostat and b) it can run Microsoft Windows. Consult the Gamry Framework, CPT110, and potentiostat Operator's Manuals for detailed computer specifications.

All Gamry potentiostats available at the present time (PC4, Series G, and Reference Family) are useable with the CPT110.

The CPT110 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 Neslab recirculating baths (RTE series with RS232 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 CPT110 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 CPT version of the Flexcell or with any Gamry Instruments temperature controller. However, Gamry does offer Flexcell compatible heating jackets as extra cost options.

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 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.

### **Heater Cord Modification For Use With the TDC2**

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:

#### **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.**

#### **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 will 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 in the hole marked H and the white wire in 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 outlet. It's 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.

### **Warning**

**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**

**Do not 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 has been discussed in Chapter 4.

Many labs already own a stirring motor and controller that can be used in conjunction 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.

### **Warning**

**The bushing in the stirring rod kit can overheat if the rotation speed exceeds 200 rpm. Always adjust the stirring rate lower than this 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 extra cost option. 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 220 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.

NOTE: This troubleshooting guide only applies if you are running a potentiostatic experiment on the cell. Galvanostatic experiments will show different symptoms.

### **Very small current or no current when you run an experiment**

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

### **Full scale current when you run an experiment**

- The reference electrode (white) lead in the cell cable is not connected to the cell properly.
- The working sense (blue) lead in the cell cable is not connected to the cell properly.
- You have incorrect experimental settings (e.g. wrong potential).
- There is a gas bubble in the reference electrode or the Lugin 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 surface.
- There is a gas bubble in the reference electrode or the Lugin 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 <sup>2</sup> (0.775 in <sup>2</sup> ) nominal
Minimum Size	4.0 cm x 4.0 cm
Maximum Size	7.5 cm x any length or 11.0 cm x 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, Teflon, Filter Paper
Wetted Materials (deionized water only)	Polypropylene, Tygon
Non-Wetted Materials	List above, plus stainless steel, Viton, silicone rubber

--Stirrer

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