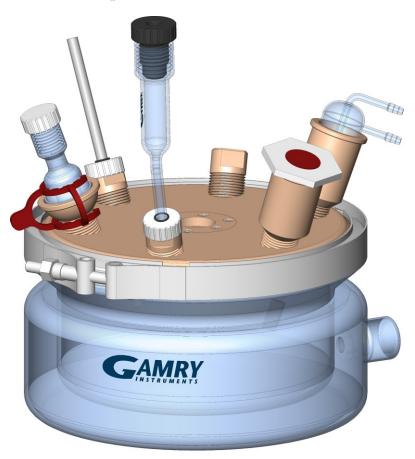


# **RCE Cell**<sup>™</sup> 1L Rotating Cylinder Electrode Cell

# **Operator's Manual**



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## If You Have Problems

Please visit our service and support page at <u>https://www.gamry.com/support-2/</u>. This page contains information on installation, software updates, and training. It also contains links to the latest available documentation. If you are unable to locate the information you need from our website, you can contact us via email using the link provided on our website. Alternatively, you can contact us one of the following ways:

Internet	https://www.gamry.com/support-2/
Telephone	(215) 682-9330 (9:00 AM-5:00 PM, US Eastern Standard Time)
	(877) 367-4267 (toll-free US & Canada only)

We will be happy to provide a reasonable level of free support for registered users. Reasonable support includes telephone assistance covering the normal installation and use of the RCE Cell Kit.

A service contract that extends both the hardware warranty and software-update period is available at an additional charge. Software updates **do not** include software enhancements offered to our customers at additional cost.

Enhancements to Gamry's standard applications software that require significant engineering time on our part may be performed on a contract basis. Contact us with your requirements.

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## **Chapter 1: Introduction**

The Gamry Instruments RCE Cell<sup>™</sup> Kit was designed for simple, reliable operation. The cell is normally used to run electrochemical tests on rotating cylinder electrodes (RCEs) or other rotating electrodes such as rotating disk (RDE) and rotating ring-disk (RRDE) electrodes. You can also customize the cell for use with other electrode types.



A reference electrode is not included in the cell kit. Requirements for this electrode vary too much from user to user to make its inclusion in the standard kit practical. Gamry Instruments sells three types of reference electrodes (SCE, Ag|AgCl, and Hg|Hg<sub>2</sub>SO<sub>4</sub>) that are suitable for use with your cell kit. Please order your optional reference electrode <u>here</u>.

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

Too often, temperature control is neglected in designing electrochemical experiments. Temperature is an important variable in the rate of both heterogeneous and homogeneous chemical reactions. Comparing test results recorded at different temperatures can be vital in gaining a full understanding of a chemical system.

For these reasons, Gamry provides the RCE Cell Kit with a jacketed glass body. When the cell is connected to a circulating water bath, accurate temperature control of your experiments becomes possible.

## **Chemical Compatibility**

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

- Rotating electrode
- Borosilicate glass (Pyrex<sup>®</sup> or equivalent)
- Unfired glass frit (ACG40)
- PTFE
- PEEK
- Polyethylene (PE)
- FEP-silicone
- Ace Glass' FETFE O-rings

Chemical resistance tables for most of these materials are available online. One exception is FETFE, which is an elastomer proprietary to Ace Glass, which consists of PTFE particles in a fluorinated rubber base similar to Viton<sup>®</sup>. According to Ace Glass, it offers slightly better chemical resistance than Viton<sup>®</sup>.

The black Ace-Thred<sup>™</sup> fittings supplied with the cell do not normally come in contact with the cell electrolyte. These are nylon fittings, so you can use nylon's properties as an indication of these fitting's suitability for use in any specific chemical environment.



The nylon bushings in the Ace-Thred<sup>™</sup> fittings and the FETFE O-rings may not be suitable for use in some electrolytes (particularly non-aqueous media). If you need better chemical resistance than that offered using the standard Ace-Thred components, Ace Glass (<u>www.aceglass.com</u>) can provide replacement fittings made from PTFE and Kalrez<sup>®</sup>, which are extremely resistant to chemical attack. Contact Gamry Instruments if you need help selecting the proper replacement fittings.

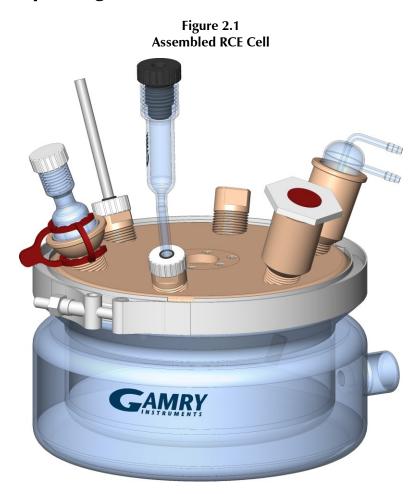
Gamry's RCE Cell is not designed for use with electrolytes that dissolve glass (extremely basic solutions or HF-containing solutions).



The glass components in the cell and the glass frits used in the reference bridge tube are not suitable for use with extremely basic solutions or solutions containing hydrofluoric acid. We do not know of any substitute cell components that will completely overcome this limitation. If you need to work with solutions that will damage the cell, we recommend that you design and build an all-plastic cell.

The simple construction of the polyethylene stoppers used to seal the unused port makes them easily adaptable as a vent for purge gas: you simply poke a hole in the flat surface of the stopper. While they provide chemical resistance to most aqueous electrolytes, they may not be a good choice for use with some aggressive solvents. If polyethylene is not suitable for your application, glass and PTFE stoppers are available from most laboratory supply houses. If you use these "more solid" stoppers, do not forget the need to vent purge gas from the cell.

## **Chapter 2: Unpacking and Installation**



## Unpacking

When you receive your RCE Cell Kit, please verify that you have received all items. Please refer to the parts list in Table 1. When shipped, all of the RCE Cell Kit components should be labeled with their Gamry Instruments, Inc. part number:

Quantity	Part number	Description
1	988-00098	Operator's Manual
1	990-00544	1L RCE Cell Kit - Top Assembly
1	820-00156	RCE Cell - Lid, PEEK
1	820-00157	SJ 28/15 joint adapter, PEEK
1	820-00158	Threaded stopper, PEEK
2	820-00159	ST/NS 24/40 joint adapter, PEEK
2	820-00200	#7 Ace-Thred™ adapter, PEEK
1	930-00045	Reference Electrode Bridge Tube
1	930-00050	SJ 28 to #7 Ace-Thred™ adapter
1	930-00063	Gas dispersion tube, short, w/ ST/NS 24/40 joint

Table 1 – 1L RCE Cell Kit, jacketed (992-00180)

1	931-00006	RCE Cell glass body, jacketed
1	935-00014	Graphite rod, .242" x 6"
2	935-00052	#7 Ace-Thred™ Bushing, Nylon, w/ 2 FETFE O-rings
1	935-00053	#11 Ace-Thred™ Bushing, Nylon, w/ FETFE O-ring
1	935-00073	Clamp for SJ 28 ball joint
3	935-00074	#7 Ace-Thred™ Plug, Nylon, w/ 2 FETFE O-rings
2	935-00076	Stopper w/ ST/NS 24/40 joint, Polyethylene
1	935-00131	O-Ring, FEP-silicone, fits flange ID 150mm
1	935-00132	Flange clamp, 150 mm
1	955-00003	AGC40 Porous glass frits and sleeves (Pkg/5)
1	966-00054	Rotary shaft seal - Sub-Assembly

Contact Gamry's support or your local Gamry representative as soon as possible if any of the parts are missing.

Please refer to the table above for replacement parts and Chapter 4 : Customizing Your Cell for a list of additional accessories for your RCE Cell.

## Initial visual inspection

After you remove your cell components from its shipping carton, check all parts for any signs of shipping damage. If any damage is noted, please notify Gamry Instruments, Inc., and the shipping carrier immediately. Save the shipping container for possible inspection by the carrier.

Your RCE Cell Kit is shipped disassembled to prevent shipping damage. All of the pieces have been carefully packaged in anticipation of rough handling in shipment. Unfortunately, no matter how carefully glass pieces are packaged, damage sometimes occurs.

When you first receive your RCE Cell Kit, please check it for any signs of shipping damage. Be especially careful if the shipping container shows signs of rough handling. Obviously, the glass pieces are 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 the shipping carrier as well as Gamry's <u>support</u> team or your <u>local Gamry representative</u> as soon as possible. In most cases, Gamry should have replacement parts in stock. Please retain the shipment's packaging material for a possible inspection and claims against the shipping company.



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.

## Assembly

This section of the manual tells you how to assemble the kit's components into a complete RCE Cell. The descriptions are based on a "standard" cell configuration consisting of a graphite-rod counter electrode, a single-junction reference electrode in a reference bridge tube, and a gas dispersion tube. A diagram of an assembled cell is shown in Figure 2.1.

Feel free to customize your cell configuration. You are only limited by your imagination and the number and size of the ports available on the top of the cell. Chapter 4: Customizing Your Cell describes various options to expand the functionality of your RCE Cell and includes a list of all available joint adapters which are not included in this cell.

There is only a T10 Torx screwdriver required to assemble or disassemble the cell. If you are assembling your RCE Cell for the first time, you may want to assembly the entire cell dry first. After you are comfortable with the cell's assembly, you can fill the cell with a test solution and run real tests.



In vacuum work in a chemical laboratory, ground-glass joints are often greased. This is unnecessary with the RCE Cell and may even cause problems if the grease gets into the test solution or on an electrode. Never grease any of the ground-glass joints on your cell.

Use a flat surface (e.g., lab bench, etc.) to assemble your RCE Cell. Even though the base of the RCE Cell is flat, smaller glass parts can roll off your work area and break. The cell can stand up on a lab bench as long as it is not subject to accidental jostling. If you are concerned that it might be knocked over, clamp the cell to a ring stand, available from laboratory supply houses.



The cell can be damaged and valuable data can be lost if the cell topples over. Ensure that the cell is stable on your work surface.



Pay careful attention to cell cleanliness. In many corrosion-testing situations, contaminants in the cell and test solution are not a problem if you take minimal precautions. In other cases, trace contaminants can lead to poorly reproducible results. If you touch the cell components with your fingers, you can inadvertently add salts and organic compounds to your cell solution. We recommend that you carefully clean the cell components using good laboratory practice. After the components are clean, avoid touching their wetted surfaces.

The RCE Cell includes a number of Ace-Thred<sup>M</sup> connectors used for a wide variety of functions. #7 Ace-Thred<sup>M</sup> connectors are particularly common. Ace-Thred<sup>M</sup> fittings are designed to seal cylindrical objects into the cell. These objects can include glass tubes, glass plugs, thermometers, and plastic electrode-bodies. Ace-Thred<sup>M</sup> fittings are designed to be tightened with finger pressure only.

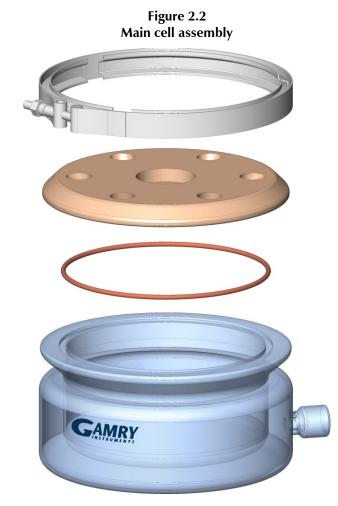


Ace-Thred<sup>™</sup> fittings should always be tightened "finger-tight". Never use tools such as a wrench or pliers to tighten an Ace-Thred<sup>™</sup> fitting. Over-tightening a fitting can break the cell. In extreme cases this can cause personal danger because broken glass can have very sharp edges.

A given Ace-Thred<sup>™</sup> size can only accommodate specific diameter objects. A #7 Ace-Thred<sup>™</sup> is specified to work with object with a diameter between 6.5 mm and 7.5 mm. If you need to add non-standard options to your RCE Cell kit, make sure you are aware of this restriction, for this joint type is two of the three "spare" ports.

#### 1. Main cell assembly

Assembly of the cell starts with placing the **Cell Top** (P/N 820-00156) onto the **Cell Bottom** (P/N 931-00006), with the **O-ring** (P/N 935-00131) in the O-ring grove on the Cell Bottom flange.



The **Cell Clamp** (P/N 935-00073) holds the top and bottom together. This clamp is a draw type. With the draw portion released, fit the clamp around the flange, then fit and secure the clamp on the hooks and push the bar back to bring the ends together. If necessary, the clamp can be loosened or tightened either by fingers or using a flat-head screwdriver.



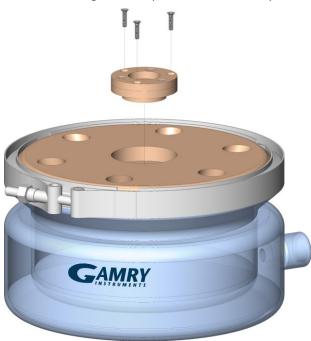
The cell requires very modest clamping force. If you have difficulty closing the clamp, loosen the bar and try again. **Do not** force the clamp or tighten it beyond what is necessary to keep the top from sliding or rotating against the bottom (and even that degree is unnecessary in many cases). Overtightening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

#### 2. Mount rotary shaft seal

The **Rotary Shaft Seal Assembly** (P/N 966-00054) is used to provide an air-tight fit for the rotating electrode. It consists of a seal body made out of filled PTFE and a 302 SS spring which provides a snug fit to the electrode. The maximum allowed rotation speed for the rotary seal is 10500 rpm. The seal is embedded in two precision-machined PEEK housings.

To install the rotary seal, insert the assembly at the center hole of the lid. Align the outer screw clearances and use three provided M3 x 12 mm Torx screws to mount it to the cell top.

#### Figure 2.3 Mounting the rotary shaft seal assembly



#### Disassembling and cleaning of the rotary shaft seal assembly

After a measurement is done, you might want to clean the individual components of the rotary shaft seal assembly. Generally, the rotary shaft seal assembly does not have to be disassembled for cleaning. In most cases it is sufficient to wipe off any contamination of the PTFE seal with a damp cloth.

Please follow the steps below if you must disassemble the rotary shaft seal:

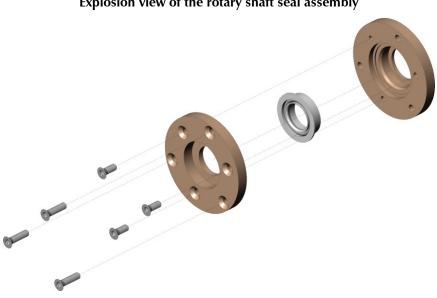


Figure 2.4 Explosion view of the rotary shaft seal assembly

- 1. Remove the assembly from the cell's lid by opening the outer M3 x 12 mm Torx screws.
- 2. Open the remaining M3 x 8 mm Torx screws which hold upper and lower seal housings together.
- 3. Carefully remove the rotary seal.



Do not use any harsh chemicals or cleaning detergents for cleaning. It is recommended to use only a damp cloth to wipe off any contamination of the PTFE seal. Avoid making the inner stainless steel spring wet.

#### 3. Attach joint adapters and stoppers

The RCE Cell can hold six individual joint adapters which can be screwed into the cells lid. See Chapter 4: Customizing Your Cell for a list of all available joint adapters. This section only discusses adapters which are included in the RCE Cell Kit.



In vacuum work in a chemical laboratory, ground-glass joints are often greased. This is unnecessary with the RCE Cell and may even cause problems if the grease gets into the test solution or on an electrode. Never grease any of the ground-glass joints on your cell.

#### SJ 28/15 ball-joint adapter

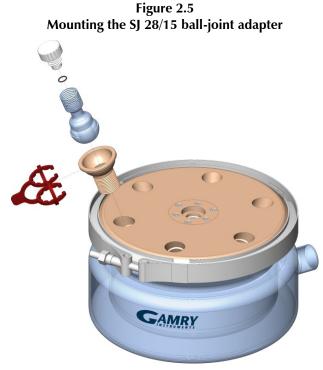
The **SJ 28/15 adapter** (P/N 820-00175) ) can be used in combination with the **#7-to-15/28 adapter** (P/N 930-00050) included in the cell kit. The ball-joint allows some flexibility because not only a part's depth but also its general position within the cell can be adjusted.



Always use the included **ball-joint clamp** (P/N 935-00073), for the joint is not stable without it.

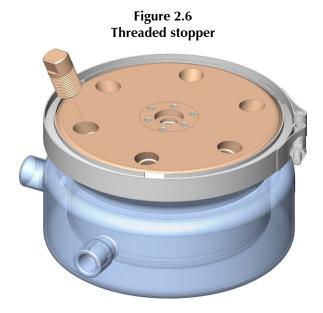
To attach the SJ 28/15 adapter, simply screw it into any of the six threads of the cell lid. The threads are slightly angled for better accessibility to the electrode.

If you are not using the ball joint, place the #7-to-15/28 adapter on top and secure it with a ball-joint clamp. Use a **#7 Ace-Thred plug** (P/N 935-00074) and one of the provided O-rings to seal the opening. Do not forget to secure the joint with a ball-joint clamp.



#### Threaded stopper

The RCE Cell Kit includes a single **threaded stopper** (P/N 820-00158) to seal off any unused port. Simply screw it into any of the six threads of the cell lid.



#### ST/NS 24/40 tapered-joint adapter

The 24/40 tapered joint is a universal adapter used in laboratories. It allows usage of many different adapters such as a gas dispersion tube (see also page 18 which discusses the installation of such parts).

The RCE Cell Kit provides two **ST/NS 24/40 tapered-joint adapters** (P/N 820-00159). Simply screw them into any of the six threads of the cell lid. The threads are slightly angled for better accessibility to the electrode.

If you are not using a 24/40 joint adapter, close them off using a one of the provided polyethylene stoppers (P/N 935-00076).

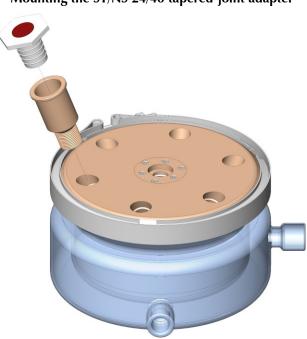


Figure 2.7 Mounting the ST/NS 24/40 tapered-joint adapter

#### **#7** Ace-Thred<sup>™</sup> adapter

Similar to 24/40 tapered joints, #7 Ace-Threds are indispensable connections in laboratories. They allow usage of many different adapters and tools required in experiments such bridge tubes or counter electrodes (see also section on page 16 and page 17).

The RCE Cell Kit provides two #7 Ace-Thred adapters (P/N 820-00200). Simply screw them into any of the six threads of the cell lid. The threads are slightly angled for better accessibility to the electrode.

If you are not using any of the #7 Ace-Thred adapters, use a **#7 Ace-Thred plug** (P/N 935-00074) and one of the provided O-rings to seal the opening.



Figure 2.8

#### 4. Bridge Tube and Reference Electrode

The bridge tube allows the reference electrode to be placed outside the test solution, isolating it from thermal-gradient experiments and more caustic test solutions. Ensure that the bridge tube is filled to the tip with a conductive solution (the test solution when possible) for ideal performance of the reference electrode.

The general position of the bridge tube is with the tip near the sample but not touching. For rotating electrodes, the distance between tip and electrode should be increased to avoid disturbance of the forced flow towards the electrode.

The provided Bridge Tube (P/N 930-00043) fits any #7 port mounted to the cell lid. Because of the shorter length of the bridge tube, it is recommended to use one of the provided #7 Ace-Thred adapters. The bridge tube already comes with a porous glass frit installed. Replacement frits and sleeves are included in the cell kit (P/N 955-00053).

Screw a **#7 Ace-Thred bushing** (P/N 935-00052) into one of the **#7 Ace-Thred adapters** (P/N 820-00200). Carefully guide the bridge tube through the opening. You can adjust the insertion depth with an O-ring provided with the bushing.

At the upper end of the bridge tube, mount the #11 Ace-Thred bushing (P/N 935-00053) and the provided O-ring. Fill up the bridge tube with a conductive solution and insert the reference electrode into the #11 bushing. The reference electrode must contact the test solution inside the tube. Various reference electrodes that work with this system are available; contact us for details.



A reference electrode is not included in the cell kit. Requirements for this electrode vary too much from user to user to make its inclusion in the standard kit practical. Gamry Instruments sells three types of reference electrodes (SCE, Ag|AgCl, and Hg|Hg<sub>2</sub>SO<sub>4</sub>) that are suitable for use with your cell kit. Please order your optional reference electrode here.

#### 5. Counter Electrode

The Counter Electrode is a very-high-density Graphite Rod (P/N 935-00014).

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

Figure 2.10 Mounting the graphite counter electrode using a #7 Ace-Thred adapter.



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

#### 6. Gas Dispersion Tube

In the RCE Cell, gas-flow control is via a **Gas Dispersion Tube** (P/N 930-00063) that fits a 24/40 standard taper. At Gamry, we often refer to the gas bubbler assembly as a three-way adapter, because it can be used to implement three functions: purge, blanket and vent. Please see Appendix B: Gas-flow Overview for additional information.

Figure 2.11 Mounting a gas dispersion tube using a ST/NS 24/40 tapered-joint adapter.



The vent function is critical. Regardless of whether gas is flowing through or over the test solution, you must provide a way for it to leave the cell. If you do not, the gas may not flow, or worse, the cell may burst apart unexpectedly. Not providing a vent for the escape of purge gas is a very common and often dangerous mistake made when setting up an electrochemical cell.



If you use purge of blanket gas, you **must** provide a vent for the gas to escape the cell. The RCE Cell was not designed to withstand gas pressure! Failure to vent the cell can cause damage to the cell, uncontrolled loss of electrolyte from the cell, and risk of personal injury to the cell's operator.

#### Attaching Gas Tubing to the Cell

The hose barb on the side of the Gas Flow Adapter can be used as a vent or as an inlet for blanket gas.



Your gas flow system should include a needle valve to control the gas flow rate. Make all gas tubing connections to the cell with this valve **turned all the way off**. Making connection with a cell filled with electrolyte or adding electrolyte to a system when the gas flow is on can lead to severe accidents. Excessive gas flow can damage the cell and result in a loss of electrolyte. In extreme cases, this can represent a significant safety hazard.

Connect the gas-flow system and add the cell electrolyte before the needle valve is turned on. Open the valve slowly, while you watch the bubbles in the cell. Bubbling should not be vigorous enough to splash large quantities of electrolyte on the cell walls.

In addition to the needle valve, a three-way valve is very useful in purge and blanket gas control. Three-way valves are available in both electrically switched and manual versions. A three-way valve switches one gas stream so it flows from a single inlet to one of two outlets.

If your system includes a three-way valve for switched purge **and** blanket gas control, we recommend that you:

- Connect the purge gas to the top adapter of the Gas Dispersion Tube.
- Connect the blanket gas to the lower adapter of the Gas Dispersion Tube.
- Use the spare port to provide a vent. A small hole poked into one of the polyethylene stoppers in the cell kit is generally a sufficient vent.

If you do not have a three-way gas control valve, you can switch from purge mode to blanket mode manually by switching the tubing from the top to bottom hose barb.

#### 7. Inserting a rotating electrode

After the cell is fully assembled, you are ready to insert the rotating electrode through the rotary shaft seal. We assume that the RxE 10k rotator is fully assembled and both shaft and electrode are mounted.



Please refer to the <u>RxE 10k Operator's manual</u> for a detailed description on electrode mounting and the correct cable connections which depend on the electrode type.

Place the cell on the base plate of the rotator. Slowly move the rotator downwards while aligning the electrode and the shaft seal. It is recommended to have a stopper installed on the rotator side to fully insert the electrode, see Figure 2.12.



Figure 2.12 RxE 10k rotator in use with a 1L RCE Cell.

Carefully guide the electrode though the rotary shaft seal. Do not use too much force, otherwise you might damage the PTFE seal. Make sure to keep some distance between the cell and the slightly wider shaft sleeve.

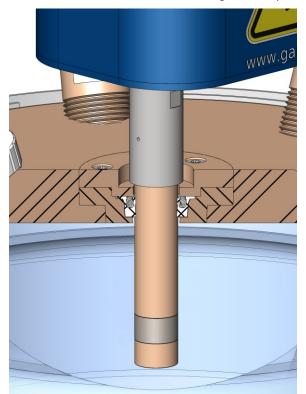


Figure 2.13 Detailed view of an electrode inserted through a rotary shaft seal.

When rotating the shaft manually, you can feel some pressure on the electrode. This is ok due to the tight seal. You are now ready to start your first experiment. In order to maintain the seals functionality, it is not recommended to run experiments with the rotary seal for an extended amount of time, especially at higher rotation rates.



Use extra caution when running experiments with glass cells.



Always install the Safety Shield<sup>™</sup> and wear safety goggles while operating the RxE system. While extremely rare, the rotating electrode or allied parts can break and fly off, injuring the operator's eyes.



Never block or restrict the motion of the rotator shaft and electrode. Always operate the RxE system in compliance with maximum speed specifications of your electrode.

## **Chapter 3: Electrode Connections**

If you are using your RCE Cell with a Gamry Instruments potentiostat, make the following connections to the electrodes.

- The Reference Electrode (RE) lead plugs into the **white** pin jack on the cell cable.
- The **green** Working Electrode (WE) and **blue** Working Sense (WS) leads from the cell cable are attached to the RxE 10k rotator system where the rotating electrode is mounted. Please refer to the <u>RxE 10k Operator's manual</u> for a detailed description on the cable connections for your working electrode.



Some potentiostats may not include a blue clip lead. In this case, connect only the green lead.

- The **red** lead on the cell cable clips to the counter electrode (CE).
- Make sure that the long **black** lead on the cell cable cannot touch any other cell connection. You may find that connection of this lead to a source of earth ground, such as a water pipe, will reduce noise in your experimental results.

If you are measuring very small currents, you may find that a metal enclosure, also called Faraday cage, completely surrounding your cell will further reduce noise. For smaller setups, use Gamry's <u>Faraday Shield</u><sup>™</sup> and connect the black lead from the cell cable.



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

## **Chapter 4: Customizing Your Cell**

The RCE Cell was designed to have options available even while fully accommodating the ASTM G5 standard. There are seven total ports and only three or four necessary for most experiments (five or six for full ASTM G5 compliance with opposed counter electrodes and a thermometer or temperature probe).

With anywhere from one to seven access ports from the cell top, customization options are myriad. Besides the various adapters shipped with your RCE Cell Kit (see Table 1), additional options are available to fully customize your cell. Table 2 lists all the different joint adapters available for the RCE Cell.

Part number	Description
820-00157	SJ 28/15 joint adapter, PEEK
820-00158	Threaded stopper, PEEK
820-00159	ST/NS 24/40 joint adapter, PEEK
820-00160	ST/NS 24/25 joint adapter, PEEK
820-00200	#7 Ace-Thred™ adapter, PEEK

Table 2 - List of joint adapters for the RCE Cell

Some common modifications to experiments are mentioned below.

#### **Addition of Corrosive Agents**

In many experiments, you record a baseline curve before you add a vital reactant to the cell. You then add that reactant, stir the cell, then record another curve. Many of Gamry's analysis packages allow you to subtract the baseline curve from the data curve. The resulting curve shows only electrochemistry related to this reactant. Another common use for added reactant is current-versus-concentration studies.

In either case, the additional ports allow you to make changes to the solution easily.

## **Temperature Sensing and Control**

The rate of almost all chemical reactions is strongly temperature-dependent. For this reason, you might want to either measure or control the temperature of your cell.

Many thermometers will fit the standard bushing for a #7 Ace-Thred<sup>™</sup>. This is a convenient way to add temperature measurement to your system.

In many cases, temperature must be controlled, not measured. The RCE Cell Kit already comes by default with a jacketed cell body. Connect the cell to a recirculating constant-temperature bath to thermostat the glass cell at a set temperature. If the bath offers remote temperature sensing, place a sensor in the spare port of the cell. This may require purchase of another option (for example an RTD-to-14/20-port adapter).

In some cases, Gamry's software supports setting automatically the temperature. Add the controlled temperature to the experiment's Setup window, and the software controls the temperature bath via an RS-232 port. In other cases, a modified script is required.

### Addition of a pH-electrode

Another possible use for the space port is addition of a pH-electrode. Depending on the adapter that is required.

### Getting a Stir Bar into the Cell

If you want to magnetically stir, and you forget to add a stir bar to your cell, you can add it using the spare port.

## Chapter 5: Troubleshooting

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

One quite common and often embarrassing error is forgetting to connect one of the cell leads! Always doublecheck your cell connections.

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

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

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

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



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

# Very small current or no current when you run an experiment but no overload indication

- The working electrode (green) lead in the cell cable is not connected to the cell properly.
- There is a gas bubble completely blocking the face of the working electrode.

Stop the experiment, fix the error, and restart. The working electrode is not damaged.

# Very small current or no current when you run an experiment, with a control amp overload

- The counter electrode (red) lead in the cell cable is not connected to the cell properly.
- The counter electrode is partially pulled out of the cell.
- If your cell includes a counter-electrode isolation tube, it is not filled with electrolyte.

Stop the experiment, fix the error, and restart. The working electrode is not damaged.

# Full-scale current and voltage when you run an experiment, with many overloads

- The reference electrode (white) lead in the cell cable is not connected to the cell properly.
- The working sense (blue) lead in the cell cable is not connected to the cell properly.
- You have incorrect experimental settings (e.g., wrong potential).
- Two of your electrodes are shorted together.
- There is a gas bubble in the Reference Bridge Tube.

Large currents have passed through the working electrode. It may need to be resurfaced or replaced.

#### Noisy Cell Current: overloads may be present

- Your de-oxygenation gas is still bubbling through the solution.
- You have a high impedance in the reference electrode path.
- There is a gas bubble in the Reference Bridge Tube.
- You are picking up noise: try a Faraday cage.

#### Excess back pressure required to bubble deoxygenation gas

• No path is available for the gas to escape.

#### **Poor Experimental Reproducibility**

- Your cell, solution, or working electrode surface has a contamination problem. Carefully clean the cell and components. Avoid touching any wetted surface 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**

Description	Dimensions
Volume	1000 mL (standard operating volume)
Number of available ports	7
Port types (included)	SJ 28/15 ball joint (x1)
	Threaded stopper (x1)
	ST/NS 24/40 (x2)
	#7 Ace-Thred™ adapter (x2)
	Rotary shaft seal assembly for 0.5" OD electrode
Cell type	jacketed
Rotation speed of rotary seal	10000 rpm max.

All specifications are subject to change without notice.

## **Appendix B: Gas-flow Overview**

### Terminology

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

Oxygen is an electrochemically active gas. Its reduction can act as the cathodic half-reaction in a corrosion reaction. You probably want to remove oxygen from the solution whenever the real-world corrosion system that you are modeling is oxygen-free.

Remove oxygen from the test solution by bubbling nitrogen, or another electrochemically inert gas, through the solution. This process is often (imprecisely) called deaeration. It is more correctly called deoxygenation. At least half an hour of vigorous bubbling with nitrogen is required to remove most of the oxygen from a test solution.

Bubbling gas through your test solution can cause noise while you are running your experiment. To avoid this noise, you can stop gas purging during the data-acquisition phase of your experiment. Instead, flow the inert gas over the top of the test solution, often referred to as "blanketing" the cell. In general, blanketing is used after solution purging, where blanketing prevents acquiring new oxygen from the gas above the solution.

Many modern electrochemical test systems include automatic control of gas flow in their experimental sequencing. This is true of Gamry Instruments' PV and PHE software. These sets of software generate a digital signal that is intended to control a solenoid valve, which in turn routes gas flow to the cell.

## Pre-saturation of the Purge Gas

Bubbling dry purge gas through your cell electrolyte can cause significant evaporation of the electrolyte's solvent during the purge process. This can be a significant source of error in some experiments. This problem can often be avoided by pre-saturation of the purge gas with the electrolyte prior to it entering the cell. This is commonly done using a "gas washing bottle" which can be obtained at most laboratory supply companies.

The RCE Cell does not include a gas washing bottle, because they are already available in many laboratories.

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