

Model K0047 Corrosion Cell System

Instruction Manual

Advanced Measurement Technology, Inc.
a/k/a Princeton Applied Research, a subsidiary of AMETEK®, Inc.

WARRANTY

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SHOULD YOUR EQUIPMENT REQUIRE SERVICE

A. Contact the Customer Service Department (865-482-4411) or your local representative to discuss the problem. In many cases it will be possible to expedite servicing by localizing the problem.

B. If it is necessary to send any equipment back for service, we need the following information.

1. Model number and serial number.
2. Your name (instrument user).
3. Your address.
4. Address to which the instrument should be returned.
5. Your telephone number and extension.
6. Symptoms (in detail, including control settings).
7. Your purchase order number for repair charges (does not apply to repairs in warranty).
8. Shipping instructions (if you wish to authorize shipment by any method other than normal surface transportation).

C. U.S. CUSTOMERS – Ship the equipment being returned to:

Advanced Measurement Technology, Inc.
801 S. Illinois Avenue
Oak Ridge, TN 37831
ATTN: Customer Service

PHONE: 865-482-4411
FAX: 865-483-2133

D. CUSTOMERS OUTSIDE OF U.S.A. – To avoid delay in customs clearance of equipment being returned, please contact the factory or the nearest factory distributor for complete shipping information.

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TABLE OF CONTENTS

Safety Instructions and Symbols.....	iv
Cleaning Instructions.....	iv
1. DESCRIPTION.....	1
2. ASSEMBLY AND CONNECTIONS.....	2
2.1 INTRODUCTION.....	2
2.2 ASSEMBLY (Figure 2).....	2
2.3 CONNECTIONS.....	3
3. SPECIMEN HOLDERS.....	4
3.1 STANDARD HOLDER.....	4
3.2 FLAT SPECIMEN HOLDER.....	4
4. VYCOR HANDLING AND REPLACEMENT.....	6

Safety Instructions and Symbols

This manual contains up to three levels of safety instructions that must be observed in order to avoid personal injury and/or damage to equipment or other property. These are:

DANGER Indicates a hazard that could result in death or serious bodily harm if the safety instruction is not observed.

WARNING Indicates a hazard that could result in bodily harm if the safety instruction is not observed.

CAUTION Indicates a hazard that could result in property damage if the safety instruction is not observed.

Please read all safety instructions carefully and make sure you understand them fully before attempting to use this product.

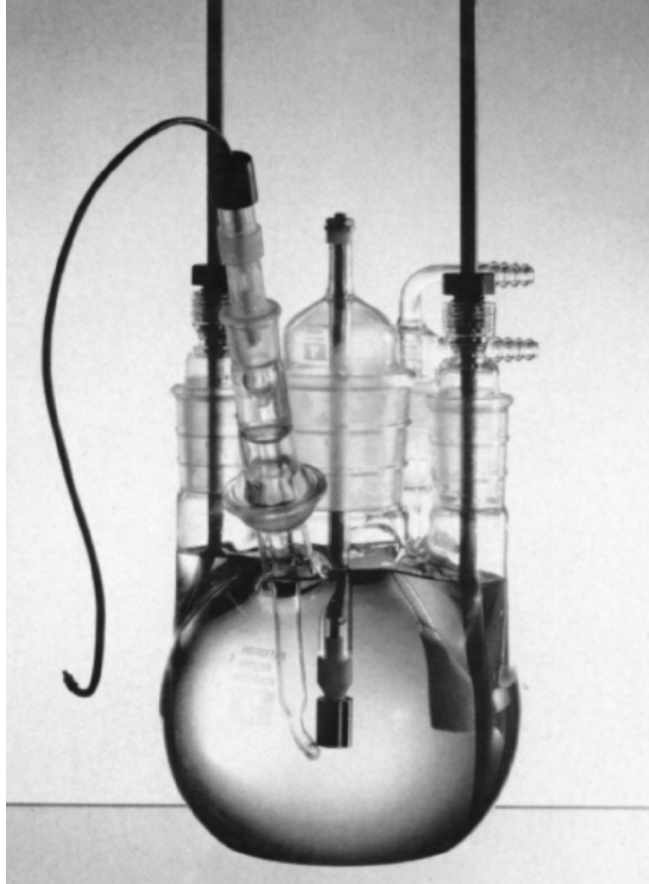
Cleaning Instructions

WARNING Using this instrument in a manner not specified by the manufacturer may impair the protection provided by the instrument.

To clean the instrument exterior:

- Unplug the instrument from all voltage sources.
- Remove loose dust on the outside of the instrument with a lint-free cloth.
- Remove remaining dirt with a lint-free cloth dampened in a general-purpose detergent and water solution. Do not use abrasive cleaners.
- Allow the instrument to dry before reconnecting the power cord.

CAUTION To prevent moisture inside of the instrument during external cleaning, use only enough liquid to dampen the cloth or applicator.



Model K0047 Corrosion Cell System

1. DESCRIPTION

Electrochemical measurements of corrosion phenomena require a cell system that is versatile, convenient to use, and that can provide reproducible conditions from one experiment to another so that a rational comparison between specimens and/or environments can be drawn. The K0047 Corrosion Cell System fulfills these requirements by incorporating the necessary cell, glassware, and hardware for performing rapid, accurate, and reproducible corrosion measurements. Unique features of this system are:

1. A 1 L flask with a flat bottom to prevent tipping.
2. A leak proof assembly for mounting specimens to be tested.
3. Twin high-density, non-permeable, graphite counter electrodes.
4. A reference electrode bridge tube incorporating an ultra low-leakage Vycor^{®1} frit.

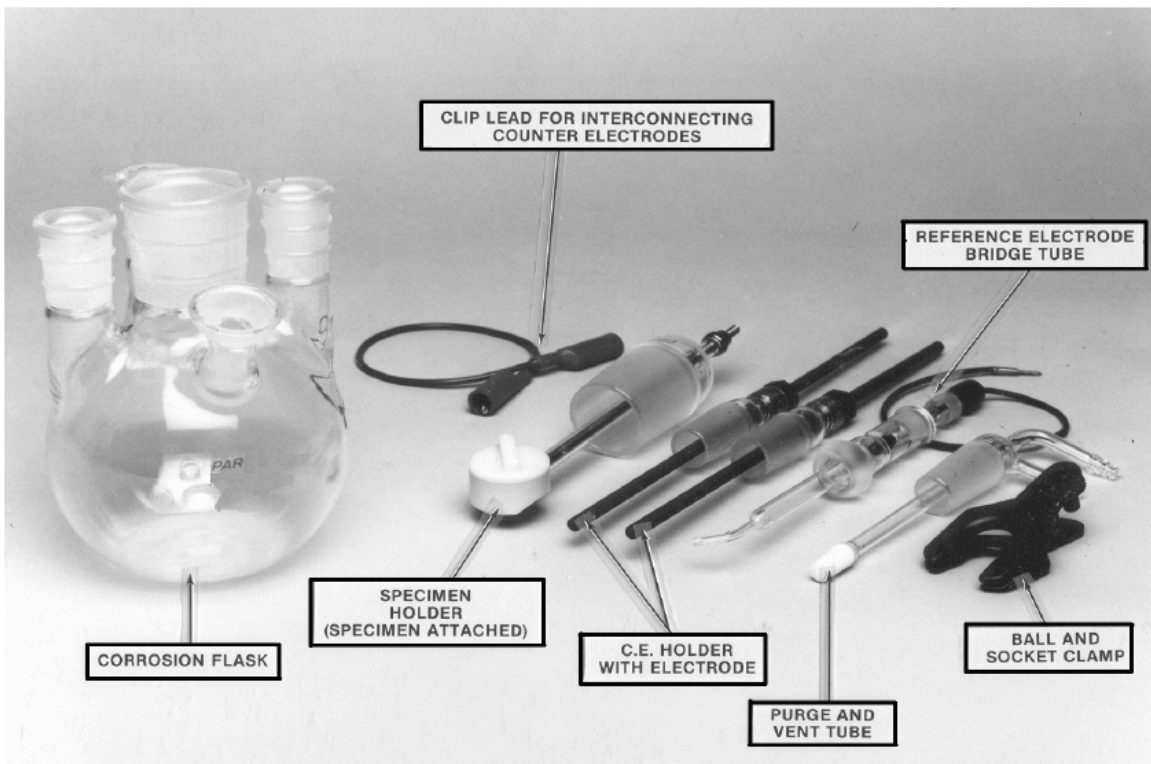


Figure 1. Model K0047 Corrosion Cell System Components.

¹Vycor is a registered trademark of Corning Glass Corporation.

2. ASSEMBLY AND CONNECTIONS

2.1. INTRODUCTION

Before proceeding to the assembly procedure, carefully unpack all items supplied and check them against the packing list. Also, wash and dry all glassware *EXCEPT FOR THE REFERENCE BRIDGE TUBE*.

2.2. ASSEMBLY (Figure 2)

1. Clamp the cell bottom to a stand. If stirring will be required, the cell should be mounted on the stirrer and an appropriate stir bar placed in the cell. A two-inch Teflon[®] coated bar will give good results in many applications.
2. Insert the high-density graphite rods into the glass adapters, making sure that the O-ring in the adapter is fitted over the graphite rod. Insert the adapters containing the graphite rods into the 24/40 joints on either side so that they just clear the bottom of the flask. Connect the two rods with the clip lead provided for this purpose.
3. Assemble the specimen under test (working electrode) to its holder and insert the assembly into the 45150 joint. Detailed information on the holders and specimen preparation is provided in Section 2.
4. Rinse the reference electrode bridge tube thoroughly with distilled or deionized water. Then fill the bridge tube with the electrolyte to be used in the flask or with any other suitable electrolyte.
5. Insert the reference electrode into the bridge tube, making sure that the bottom of the electrode contacts the solution in the bridge tube.
6. Expose the filling hole of the reference electrode. Then insert the reference electrode assembly into the ball and socket joint and clamp it loosely. Adjust the bridge tube so that the Vycor[®] tip is positioned to within about 1 mm of the specimen surface. **NOTE:**

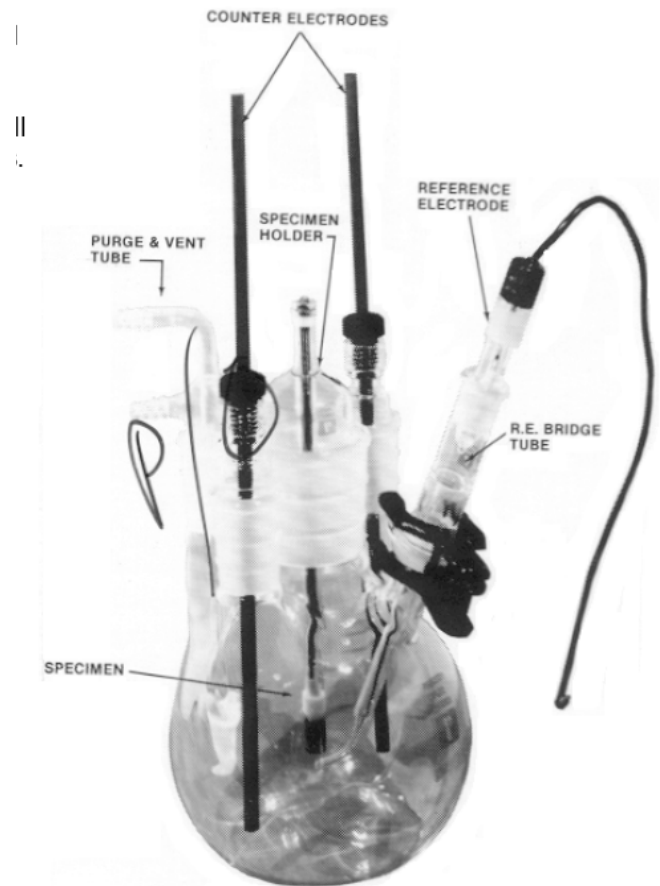


Figure 2. Assembled Corrosion Cell.

Vycor[®] must be properly handled to prevent damage. See Section IV.

7. Pour enough electrolyte into the cell to cover the entire surface of the specimen under test.
8. Insert the purge/vent tube into the cell. If using purge gas, connect the purge gas supply to the upper tube.

2.3. CONNECTIONS

Make the connections between the potentiostat and cell as follows.

WARNING: SOME OF THE POTENTIOSTAT/GALVANOSTATS WITH WHICH THE CORROSION CELL IS USED HAVE OUTPUTS AS HIGH AS 100 V AT CURRENTS AS HIGH AS 1 A. THUS IT IS POSSIBLE TO RECEIVE DANGEROUS, EVEN LETHAL, ELECTRICAL SHOCKS IF THE CELL CONNECTIONS ARE TOUCHED WHEN THE OUTPUT IS LIVE. FOR SAFETY, THE CELL SWITCH OF THE INDIVIDUAL POTENTIOSTAT/GALVANOSTAT SHOULD BE SET TO THE OFF POSITION WHEN MAKING OR BREAKING CELL CONNECTIONS.

1. REFERENCE ELECTRODE. The lead coming off the reference electrode installed in the K0047 plugs into the reference lead jack (white) at the end of most PAR potentiostat cell cables.
2. WORKING ELECTRODE LEAD. The working cable lead (green) attaches via alligator clip to the contact at the top of the specimen-holder assembly.
3. COUNTER ELECTRODE LEAD. The counter lead (red) attaches via alligator clip to either of the K0047's carbon electrodes. In addition, there must be a clip lead connected between the two carbon electrodes so that the Counter Electrode lead drives both of them.
4. GROUND LEAD. This lead (black) is ordinarily not used. It is provided for those applications where a convenient source of ground may be required, such as grounding a metal shield (Faraday shield). Take care that the ground lead does not short against any of the other electrodes or leads.
5. SENSE LEAD (if available). The sense lead (gray) is connected to the working electrode along with the working lead. A four-inch patch cord is provided for interconnecting the Working Electrode contact and the Sense jack. As a general rule, the sense lead is required when measuring currents in excess of 200mA, but must always be connected if present on the cell cable.

3. SPECIMEN HOLDERS

3.1. STANDARD HOLDER

This Specimen Holder (Figure 3.1) is designed to accept cylindrical specimens 1/2" (1.27 cm) long, 3/8" (0.95 cm) in diameter, drilled to a depth of 1/4" and tapped to accept a 3-48 thread. (The cell will accommodate other working-electrode geometries as long as the specimen can clear the 45 mm (1-3/4") diameter opening provided for the electrode holder.) The Teflon[®] compression gasket between the specimen and the glass electrode holder ensures a leak-proof assembly. The specimen should be threaded onto the assembly to finger tightness. Too much pressure will break the holder; too little will cause leakage. The Teflon compression gasket will "cold flow" under continuous stress and should be replaced when leakage is detected or the distortion of the gasket becomes excessive.

Specimens should be prepared by wet grinding with 240-grit SiC paper, followed by wet polishing with 600-grit SiC paper until scratches are removed. Prior to immersion in the cell, the specimen should be rinsed, dried, and then thoroughly degreased in boiling benzene or trichlorethylene, followed by a final rinse in deionized water.

WARNING: EXERCISE EXTREME CAUTION IN CARRYING OUT THESE OPERATIONS. BOTH BENZENE AND TRICHLOROETHYLENE ARE BOTH TOXIC AND VERY FLAMMABLE.

3.2. FLAT SPECIMEN HOLDER

This accessory extends the versatility of the basic corrosion measurement system by allowing the corrosion characteristics of flat specimens to be readily determined. The K0105 Flat Specimen Holder is manufactured from Tefzel[®] Dupont fluorocarbon with mechanical properties superior to Teflon. The holder is designed to accept specimens 0.625 ± 0.010 inches in diameter and up to 1/8 inch thick. The sealing washer is made of Kalrez[®], a new fluorocarbon elastomer with a chemical resistance approaching that of Teflon. The Kalrez washer exposes 1 cm² of the specimen to the test solution. The flat specimen holder attaches conveniently to the Electrode Mounting Rod and the Electrode Holder so that it can be used with the K0047 Corrosion Flask. Also available is an O-ring replacement kit for the K0105 Holder. The K0106 O-ring kit contains three pairs of replacement Kalrez O-rings and washers. The Kalrez washers are elastic and can be used many times before needing replacement. Note that a substitute Teflon washer (MP1239) is also available. This washer is disposable, that is, it must be replaced after each use. These washers are sold in packages of twenty five. The superior seal provided by the Teflon washers eliminate crevice corrosion of the sample. Figure 3.2 is an exploded view drawing of the K0105 Flat Specimen Holder. Note that the K0105 Flat Specimen Holder is not furnished with the K0047 Corrosion Cell System but must be purchased separately.

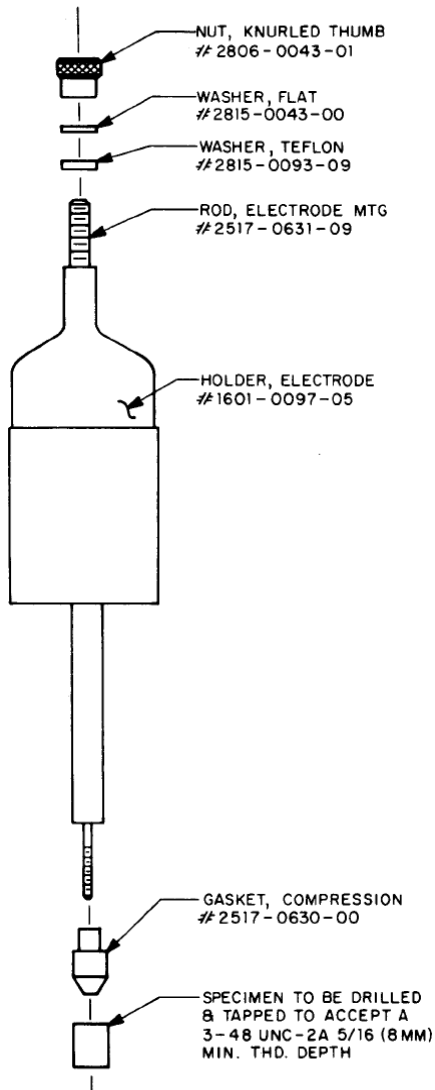


Figure 3.1. Standard Specimen Holder Assembly.

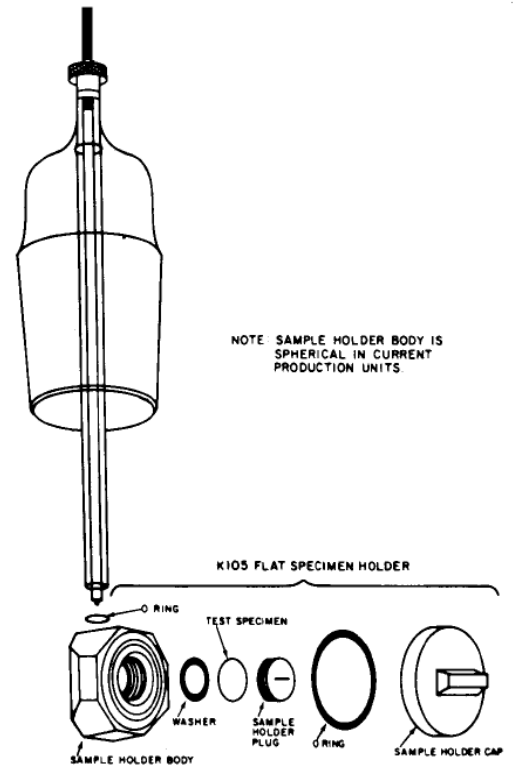


Figure 3.2. Exploded View of K0105 Flat Specimen Holder.

4. VYCOR HANDLING AND REPLACEMENT

The bridge tube furnished with the K0047 Corrosion Cell System incorporates an unfired Vycor tip designed specifically to provide ultra low leakage rates with minimum IR drop through the tip. This arrangement eliminates complications arising from poisoning of test solutions by reference electrode filling solutions or by unwanted species produced at the counter electrode, and allows high sensitivity operation of potentiostats and polarographic analyzers. The Vycor tip is deliberately seated to the tube in a dry state. **ONCE THE TIP IS WETTED, IT MUST BE MAINTAINED IN A WET STATE.** What this means practically is that, when the tube is not in use, it must be stored in a solution - ideally a solution that closely approximates the solutions employed in the electrochemical experiment. If the tip is allowed to remain dry *for more than a few minutes* once it has been wetted, it will crack. A cracked tip must be replaced before the tube can be used again. Also, the tip may crack if it is exposed to drastically different environments, e.g.:

1. The bridge tube is immersed in an aqueous environment but filled with an organic solvent (or vice versa).
2. The bridge tube is removed from an aqueous environment and placed directly in an organic solvent (or vice versa).

If it should be necessary to use the bridge tube in different environments, i.e., organic solvents and aqueous solutions, it may be possible to prevent the tip from cracking by equilibrating the tip (inside and out) with a mixture (or mixtures) of the two environments. Alternatively, two bridge tubes should be used, one for each environment.

The following procedure is recommended for replacing a Vycor tip.

1. Slice off the old Teflon sleeve with a razor blade. Then discard the sleeve and the old tip.
2. Wash and dry the bridge tube.
3. Slide a new length of heat-shrink Teflon tubing onto the bridge tube.
4. Stand the bridge tube on the new Vycor tip. **DO NOT WET THE TIP AND AVOID FINGER CONTACT WITH IT.**
5. Heat the Teflon tubing on all sides with a hot-air stream from a heat gun. (A direct flame should not be used because it will char the Teflon.) Allow the tubing to shrink over the disk. **BE SURE TO ALLOW THE TUBING TIME TO COOL TO ROOM TEMPERATURE BEFORE LIFTING THE ASSEMBLY.**

Extra tips and Teflon sleeves are provided with the bridge tube. Replacements can be purchased from Princeton Applied Research.