USERS MANUAL
BAS RDE-1
(preliminary)

Bioanalytical Systems, Inc®
2701 Kent Ave
West Lafayette
Indiana 47906
Telex: 276141
Tel: (317) 463-4527
Fax: (317) 497-1102

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1.0 Introduction

The BAS RDE-1 Rotator is a miniature rotator system for use in constant-RPM and hydrodynamic modulation rotating disc electrochemistry. The short titanium shaft and 1/4" diameter electrode provide for an extremely low-inertia system capable of highly accurate rotation and modulation, even at radical modulation rates or waveforms.

Electronic control of the rotator is via a proportional- integral closed loop circuit driving a ironless core DC servomotor.

The rotator is automatically raised and lowered onto the cell vial. This provides quick and convenient control of rotator position directly from the front panel.

The rotator assembly is easily inverted for polymer spin coating directly on the unit. The electrodes are small and are rapidly interchangeable. The unit also provides an adjustable valve system for inert gas purging inside the cell vial.

The RDE-1 can be used stand-alone or can be controlled directly by the BAS 100A Electrochemical Analyzer or other voltage source.

1.1 Guarantee and Damages in Transit

The BAS RDE-1 Rotating Disc Electrode System is guaranteed to be of quality material and workmanship. This warranty is valid for 90 days from the date of shipment. Electrochemical cells and working electrodes are warranted for 60 days. This assumes normal usage under commonly accepted operating parameters. The warranty is automatically invalidated by improper use, application, or servicing of the instrument. This warranty and remedy are given expressly and in lieu of all other warranties, expressed or implied, of merchantibility or fitness for particular purpose and constitutes the only warranty made by BAS.

BAS agrees to either repair or replace, at its sole option, any product deemed to be faulty or malfunctioning during the warranty period. BAS will not be obliged to replace or repair any piece of equipment which has been abused, improperly installed, altered, damaged or repaired by others. Defects in equipment do not include decomposition, wear, or damage due to chemical action or corrosion.

All products manufactured by BAS are tested and inspected prior to shipment. BAS shall have no liability whatsoever for special, consequential, or punitive damages of any kind arising from the sale, installation, use, or servicing of its instrumentation.

Limited obligations covered by this warranty include:

- In the case of instruments not of BAS manufacture, the original manufacturer's warranty applies.
- Shipping charges under the warranty are covered in one direction only. The buyer is responsible for shipping charges to the factory, if return of the part is required.
- Consumable items such as carbon paste, reference electrodes, source lights, fuses, etc. are excluded from the warranty.

To validate your warranty and make it easier for BAS to service your needs, please complete and return the BAS User Group Application form inserted on the front page of this manual.

Packaging for BAS products has been designed to assure their safe delivery. After recieving the shipment, the equipment should be immediately unpacked and inspected for any damages in transit. Should damage be present or should the shipment be incomplete in spite of our careful inspection, please use the following procedure:

1. Retain the shipping box and all packing materials.
2. Contact the freight carrier immediately.
3. Contact BAS no later than 3 days after receipt of goods.

BAS products are shipped FOB factory. It will be the customer's resposibility to issue a claim for damages with the freight carrier in a timely manner.

Damages covered by the warranty will be repaired or replaced without charge. Packing and shipping costs for goods returned to BAS will be born by the customer.

1.2 Technical Data

Rotation Range	20 to 10,000 RPM
Accuracy	error <2% at <100 RPM, <1% at >100 RPM
Slew Rate (typical)	400,000 RPM/s (with shaft, brushes, and electrode)
Slew Rate (maximum)	700,000 RPM/s (with shaft, brushes, and electrode)
Band Width	75 Hz at 3500 RPM base and 1000 RPM peak-to-peak modulation.
Rotator Shaft	Titanium
Motor	12V, ironless core, low inertia DC servo
Motor Control	Proportional-integral, closed feedback loop servo drive
Motor Protection	Internal: electronic-limited External: fused
Main Fuse	0.5A Slow-Blow @ 100-120 VAC 0.25A Slow-Blow @ 220-240 VAC
Power	100-120/220-240 VAC, 50/60 Hz, V-A <100 watts
Size	7.25"(18.4 cm) W x 14.6"(37.1 cm) H x 10.8"(27.5 cm) D.
Weight	10 lbs.
Operating Temp.	10 to 50 degrees Celcius
Remote Control	Speed control via external voltage source (1 volt=1000 RPM).

This instrument left the factory in technically safe and perfect condition. In order to maintain this condition and ensure danger-free operation, the user must observe the instructions and warnings contained in this manual. Be sure that only fuses of the type and amperage indicated are used as replacements. Use of repaired fuses or short-circuiting of the fuse holder is prohibited.

This instrument is manufactured, either wholly or in part, for research purposes only. Use in medical dignosis is not intended, implied, or recommended by the manufacturer. Use for this purpose and accountability for the same rests entirely with the user.

1.3 Technical Changes

We reserve the right to make technical changes to improve the instrument. Minor changes will be self-evident; improvements affecting use or maintenance will be described in supplementary pages to this manual.

1.4 Accessories

	Quantity	Description	BAS P/N
STANDARD	1	Shaft O-ring	MR-1241
	1	400 ml beaker/cell vial	MF-7011
	1	Power cord	ER-8502
	1	Ag/AgCl reference electrode	MF-2020
	1	Platinum wire auxiliary electrode	MR-1032
	1	RDE-1 Operations Manual	MF-9057
	1	GCDE glassy carbon working electrode	MW-1090
	1	Gas Dispersion Tube	MW-4560
	1	Ref./Aux. Electrode Lead	EW-7530
	1	PK-3 Polishing Kit	MF-2056
	3 ft.	1/8 dia TFE tube	MR-5002
	1	BAS Cell Lead (w/ EF-1090-1)	EW-7524
		or	
	1	Univer. Cell Lead (w/ EF-1090-2)	EW-7525
	1	BAS Remote Cable (w/ EF-1090-1 only)	EW-7526
OPTIONAL		PtDE platinum wrkng. electrode	MW-1092
		AuDE gold wrkng. electrode	MW-1091

Standard electrode materials are glassy carbon, platinum, and gold. Electrode diameter is 3mm. Electrodes of other materials and diameters will be fabricated on a custom basis. Call for a quotation.

BAS will also fabricate special purpose vials on a custom basis. Call for a quotation.

1.5 Replacement Parts

Display Board Assembly	EW-4350
Main Board Assembly	EW-4360
Brush Block Assembly	MW-2110
Bearing Housing Assembly	MW-2120
Shaft Assembly	MW-2130
Shaft O-ring	MR-1241
Auxiliary/Reference Lead	EW-7530
Internal Ribbon Cable	EW-7531

Service other than direct replacement of the above assemblies should be referred to the factory. Please contact:

Service Coordinator, Bioanalytical Systems, Inc.
2701 Kent Ave.
W. Lafayette, IN 47906
PH: (317) 463-4527

1.6 Electrical Connection

The RDE-1 Rotating Disc Electrode System can be used with either 110V or 220V (50-60 Hz) power supply. Make sure that the position of the voltage select card corresponds to the local power supply.

VOLTAGE SELECTION

Should the power option need to be changed, unplug the line cord and open the cover using a small screwdriver or similar tool. Set aside the cover/fuse block assembly and pull the voltage select card straight out of the housing, using the white plastic indicator pin. Orient the selector card so that the desired voltage is readable at bottom of card. Orient indicator pin to point straight up when desired voltage is readable at bottom. Insert the voltage selector card back into the housing, printed side of card facing the On/Off switch. The edge of the card containing the desired voltage should be inserted first. Replace cover and verify that the indicator pin shows the desired voltage.

Figure 1-1 *Removal of Voltage Selector Card*

FUSING ARRANGEMENT

To change from North American to European fusing arrangement: open cover, using a small screwdriver or similar tool. Loosen Phillips screw 2 turns, then remove fuse block from cover by lifting up and away from Phillips screw. Change fuses (note that 2 European fuses are required), invert fuse block and slide back onto Phillips screw and cover so that the 2 European fuses are up. Tighten Phillips screw and replace cover. Note that fuse(s) that go into the housing first are the active set.

Figure 1-2 *Voltage Selector Card Orientation*

FUSE RATING

Be sure that correctly-rated fuse(s) are used:

100-110 V.....0.5 Amp
220-240 V.....0.25 Amp

Connect the instrument only to sockets with a ground contact by using the three-pronged power cord included with delivery. For problem-free operation and for safety considerations, be sure that the instrument is firmly connected to a positive ground via the power cord.

2.0 Instrument Description

2.1 Controller Module

Figure 2-1. *Instrument Front Panel*

1. *RPM Display*
2. *Up/Off/Down Switch*
3. *Remote/Set/Local Switch*
4. *RPM Adjust*
5. *Auxiliary Electrode Jack*
6. *Reference Electrode Jack*
7. *Gas Adjust (Rate and on/off)*
8. *Gas Out*
9. *Vial Plate*
10. *Working Electrode*
11. *Rotator Shaft*
12. *Shaft Skirt*
13. *Cell Top*
14. *Rotator Motor Housing*

1. RPM Display: Displays actual rotator RPM when switch is on either “Remote” or “Local”. Displays preset RPM value when switch is in “SetRPM” position.

2. Down/Off/Up Switch: Controls position of rotator chassis. Electronics will sense and automatically halt the movement of the chassis at the top and bottom of travel. An additional sensor built into the cell top stops the downward movement of the rotator housing when it comes in contact with the cell vial.

3. Local/SetRPM/Remote Switch: Source of signal for rotator speed control. “SetRPM” allows user to preset rotator RPM for use in “Local” position. “Remote” allows external control of rotator with a BAS 100A, overriding local control. Note that an applied voltage at “RPM In” on the rear panel will always be summed with the instrument RPM voltage, regardless of whether the instrument RPM voltage source is local or remote.

4. RPM Adjust: Determines actual rotator RPM in “Local” mode and preset RPM value in “SetRPM” mode. Overrode by external control in “Remote” mode.

5. Auxiliary Electrode Jack: Female jack (red) connected to auxiliary electrode with Ref./Aux. Lead.

6. Reference Electrode Jack: Female jack (white) connected to reference electrode with Ref./Aux. Lead.

7. Gas Adjust: Needle valve controlling rate of gas flow to Gas Dispersion Tube.

8. Gas Out: Bulkhead fitting connected to the Gas Dispersion Tube.

9. Vial Plate: Provides for positive positioning of the cell vial. Will accomodate 250, 400, and 600 ml beakers.

10. Working Electrode: Small plastic body containing the actual electrode surface. Threaded design allows for quick exchange of electrodes. Surface polished extremely flat and smooth for consistant flow dynamics.

11. Rotator Shaft: Titanium for chemical inertness. Electrode contact made with a multipoint spring plunger compressed against the electrode material. Contact to the shaft made by low-noise, silver graphite leaf-spring brushes riding directly on the shaft.

12. Shaft Skirt: Protects the rotator shaft bearing from fluid when the rotator chassis is inverted for polymer spin coating.

13. Cell Top: Contains locations for reference electrodes, auxiliary electrodes, and gas purging tube around its perimeter. Spring-loaded to allow the cap to automatically stop on the cell vial, providing for rapid experiment setup. Provides a closed cell vial for more efficient gas purging.

14. Rotator Motor Housing: Unit supports the rotator motor, brush assembly, cell vial sensor, shaft bearing, contact brushes, and cell top. It is easily inverted for spin coating the working electrode.

Figure 2-2. *Instrument Rear Panel*

15. Cell Lead Connector
16. Gas In Connector
17. Rotator Fuse
18. Voltage Select Indicator
19. Power Cord Outlet
20. Power On/Off
21. BAS 100A Accessories
22. RPM In Jack
23. Common Jacks (2)
24. RPM Out Jack

NOTE: *Do not cover the heat exchange vents at the top of the rear panel. Improper ventilation may cause overheating of the electronics.*

15. Cell Connector: Socket for cell lead containing reference, auxiliary, working and ground conductors. The connection to BAS 100A or other controller.

16. Gas In Connector: Bulkhead fitting providing connection to external gas source. **NOTE:** 10 psi is the maximum pressure.

17. Rotator Fuse: Contains O.5 Amp fuse to prevent rotator motor damage during high current conditions. Use only a O.5 Amp North American Slow-Blow fuse.

18. Voltage Select Indicator: Shows the selected line voltage. Be sure that the selected line voltage corresponds to the local power supply. If not, see Section 1.6; Electrical Connection.

19. Power Cord: Socket for power cord. Be sure that the 3-prong power cord included with the instrument is connected to a grounded outlet.

20. Power On/Off: Display LED's on front panel will be lit when unit is on.

21. BAS 100A Accessories: 37-pin port for remote control via BAS 100A.

22. RPM In: An external voltage applied at this input jack will create a corresponding RPM (1 volt=1000 RPM) at the rotator regardless of the position of the Local/SetRPM/Remote switch. This voltage will always be summed with the instrument RPM voltage, allowing various local/remote/external control combinations. Further explanation is in Section 3.3; Remote Control.

23. Common: Common jacks.

24. RPM Out: This output jack carries real rotator RPM voltage generated directly by the output tachometer mounted on the rotator motor.

2.2 Rotator Chassis

Figure 2-3. *Rotator Chassis*
25. Rotator Motor
26. Pivot Bolt
27. Sensor
28. Brush Connector
29. Brush Block Assembly
30. Bearing Housing Assembly
31. Shaft Coupler

25. Rotator Motor:	12V ironless core servomotor with integral digital tachometer. Wires from the motor and elsewhere go through a strain-relief ziptie and out the wire guide bushing (a).
26. Pivot Bolt:	Shoulder bolt compresses a spring washer system so that the chassis plate (b) and the carriage plate (c) are preloaded. This allows for smooth inversion of the chassis while preventing movement due to vibration.
27. Sensor:	This sensor is triggered by a pin located on the beaker cap. The lift system will halt with the chassis in position on the cell vial.
28. Brush Connector:	Disconnects for easy replacement of the brush assembly. Also transfers the working signal to shielded cable.
29. Brush Block Assembly:	4 silver-graphite leaf-spring brushes mounted directly on a small insulator block. All 4 brushes connected in parallel before entering the brush connector.
30. Bearing Housing Assembly:	Precision ball bearing in an insulator housing.
31. Shaft Coupler:	Insulating sleeve clamped by two dynamically balanced hub clamps.

2.3 Lift System

Figure 2-4: *Lift System*
32. Bushings
33. Leadscrew
34. Leadnut
35. Leadscrew Coupler
36. Lift Motor
37. Carriage Plate

32. Bushings:	Support and align the leadscrew. Bottom bushing utilizes a thrust washer due to heavier loading.
33. Leadscrew:	Translates rotational movement into translation to lift the rotator chassis.
34. Leadnut:	Fixed to the chassis, the nut will move vertically as the leadscrew rotates.
35. Leadscrew Coupler:	Connects the leadscrew and the lift motor together. Flex-type for misalignment.
36. Lift Motor:	24V high-torque DC servomotor to drive the leadscrew.
37. Carriage Plate:	Chassis bolts to this plate for inversion feature. Wire guide bushing allows the wiring to exit the chassis.

3.0 Operational Set-up and Instructions

3.1 Installation and Initial Instrument Operation

Placement

Locate the RDE module in as clean and as spacious laboratory bench area as possible. Normal operation requires space in front of the instrument to replace cell vials/beakers and to clean or remove electrodes and gas lines. Space is also needed to manipulate the switches that move the rotator up and down, to adjust the rotation rate and gas purge rate and for general housekeeping.

Whenever analytical instruments are being used to measure small electrical signals as is often the case when using the RDE, it is highly advisable to power the instrument from an isolated main power line or from a line with few other electrical devices on it. This will reduce electrical noise in the output response. Appliances or instruments that require large amounts of intermittent power should not be on the same power line as this instrument.

Power Requirements

The RDE can be powered from either 100V, 110V, 220V or 240V at 50 or 60Hz. The main power source in your location must be the same as the voltage selected in the power cord connector on the rear panel of the unit. All RDEs are configured for the correct voltage and frequency for the “shipped to” destination. See section 1.6 for details on how to change input power. If the RDE is purchased with a BAS100A Electrochemical Analyzer, then the voltage input is always assummed to be 110V because all peripheral devices of the BAS100A can be powered from the auxiliary power source of the Electrochemical Analyzer.

Assembly

The RDE is completely assembled and throughly tested before leaving the BAS factory. The only requirement for operation is to connect the electrical cables and purge gas lines.

Power Cord Connection

Push the socketed(female) end of the power cord into the port on the Voltage Selector Card located in the lower center of the backpanel(see figure 2-2, item 19). Before making this connection, make certain the power on/off switch is in the off position. This switch is labelled with a 0 and 1. When 0 is pushed, the power is off and when 1 is pushed, the power is on.

Rotation Rate Control Cable Connection

Rotation rate can be manually controlled by the front panel RPM ADJUST knob or remotely through either the BAS100A Accessories Port or the RPM IN jack and an external voltage source. Manual or LOCAL control of rotation rate is explained in the next section. Connecting the BAS100A or an external voltage source is described below.

BAS100A Accessories Port Connection (Remote Control)

To control the rotation rate as well as the gas purging on and off from the BAS100A Electrochemical Analyzer, insert the 37 pin ribbon cable into the appropriately labelled ports on the back panels of both the RDE and the BAS100A. Figure 2-2 illustrates the position of this port on the RDE back panel. To operate in this mode, the front panel “RPM Adjust” selector switch must be in the REMOTE position. The BAS100A must be in the OPERATION MODE= RDE.

External Voltage Source Connection (Remote Control)

To control the rotation rate from an external voltage source, e.g., a waveform generator, plug the high output from the voltage source into the jack labelled “RPM IN” and the low or common line into one of the jacks labelled “COMMON”. One thousand RPM will be generated for every 1 volt of input up to a limit of 10 volts or 10,000 RPM. A voltage corresponding to the actual rotation rate is available at the “RPM OUT” jack. Again, the conversion factor is 1V/1000 RPM.

Cell Lead Connection

The cell lead contains the electrode lead wires; working, auxiliary, and reference plus a chassis ground-shield wire. There are two possible configurations and corresponding cables for this connection. One is for the BAS electrochemical equipment; BAS100A(or BAS100) Electrochemical Analyzer or CV-27 Voltammograph, and the other is for any potentiostat/current transducer.

To make the connection to BAS equipment, the correct cable has a silver LEMO connector on both ends. One end plugs into the “CELL” socket on the back panel of the RDE module and the other plugs into the “CELL LEAD” socket on the back of the BAS instrument; BAS100, BAS100A, or CV-27.

To make the connection to other equipment, the correct cable has a LEMO connector on one end and three open wires on the other. The color code of the wires is: black-working or the rotating electrode, red-auxiliary electrode and white- reference electrode. Connect the corresponding leads from the other equipment to these wires.

Gas Inlet Connection

The RDE Module package contains 0.25" O.D. tygon tubing and a gas line fitting. One end of the gas line fitting is barbed to fit inside the tygon tubing. Push the barbed end into the tubing. The other end of the fitting connects to the “GAS INLET” port on the rear panel of the RDE chassis. To attach the line, simply push the connector into the port. A retaining clip will snap into place. The open end of the tygon tube is connected to a regulated gas supply. The inlet gas pressure must not exceed 10 psi.

Reference and Auxiliary Electrode Leads (front panel)

The reference and auxiliary electrode lead wires connect from the front panel jacks to the respective electrodes in the cell top assembly. To connect, simply push the red(auxiliary) and white(reference) lead wire's pins into the front panel jacks labelled REF. and AUX. The socketed end of the lead wires connect to the electrode pins.

Gas Outlet (front panel)

The RDE package contains a length of 0.125" O.D. plastic tubing, a plastic fitting with a barbed end, and a gas dispersion tube(a glass tube with a frit on the end). Push one end of the plastic tubing over the barbed end of the plastic fitting and the other end over the open end of the gas dispersion tube. Push the plastic fitting into the "GAS" port on the front panel, the retaining clip will snap into place. Guide the dispersion tube into the small hole in the cell top.

If all of the above connections have been made, then the RDE is ready to have the power applied and use.

Initial Instrument Operation

With all front and back panel connections made, follow the procedure below for the initial use.

1. Place the Down/Off/Up, 3-position switch (item 2, figure 2-1) in the Off, middle position.
2. Place the Local/SetRPM/Remote, 3-position switch (item 3, figure 3-1) in the SetRPM, middle position.
3. Push the rear panel power switch to the on, "1", position. When power is applied, LEDs light status indicators on the front panel display (item 1,figure 2-1).
4. Push the Down/Off/Up switch to the Up position. The Rotator Motor Housing (item 14, figure 2-1) will move up in its track to its highest position and stop automatically.
5. Remove the electrode by holding the Shaft Skirt (item 12, figure 2-1) and turning the electrode body (item 10, figure 3-1) counterclockwise. See section 6.1 for more information. Polish the electrode by following the instructions in the Polishing Kit.
6. Replace the electrode. (See section 6-1) Note that a slight upward pressure must be applied to the electrode body to start the threads.
7. Place a test solution in the cell vial/beaker. A common test solution is 2mM ferricyanide in 1 M KCl. This is prepared by adding 330mG Potassium Ferricyanide and 50.6G Potassium Chlorlide to 500mL deionized, distilled water. Caution: do not fill the 400mL beaker beyond the 275mL mark. The bottom tray is designed to hold 3 different sized beakers. Make sure the correct cut-out is below the rotator

assembly. Put the cell vial/beaker in the bottom tray cut-out.

8. With the cell vial/beaker in place lower the rotator assembly onto the cell vial. This is done by pushing the Down/Off/Up switch to the Down position. The rotator will automatically stop when it touches the top of the beaker.

9. Place the reference and auxiliary electrodes in two of the holes in the cell top and attach the cell leads. Start purging the solution with inert gas. For this initial experiment, purging 5-10 minutes will be adequate.

10. The Local/SetRPM/Remote switch should be in the SetRPM position. If not, push the switch to this position and use the “RPM ADJUST” knob to adjust the rotation rate to 400 RPM. If the RDE is being used with the BAS100A then push the Local/SetRPM/Remote switch to the Remote position and fix the rotation rate to 400 RPM using the OPERATION MODE= RDE and answering the ROTATION RATE prompt with 400.

11. The instrument is ready to use. The electrode will begin to spin when the Local/SetRPM/Remote switch is put in the Local position or when the RUN command is initiated on the BAS100A.

Typical test parameters are:

Working Electrode: GCDE

Reference Electrode: Ag/AgCl

Auxiliary Electrode: Platinum Wire

Test Solution: 2mM Ferricyanide/1M KCl

Initial E: 600mV

Final E: 100mV

Scan Rate(V): 20mV/S

Rotation Rate: 400RPM

Sensitivity: 10E-5 (10s of microamperes)

12. After the experimental run, the Rotator Housing can be raised and the electrodes can be rinced or polished as needed. The system is then ready to do the next experiment.

The power switch on the back panel should be in the OFF position. Make sure that the Voltage Select Indicator on the rear panel corresponds to the local power supply. If not, see Section 1.6. The Rotator is then connected to the mains with the power cable included with delivery. The mains connection must have proper grounding.

WARNING: Any disconnection of the ground wire either inside or outside of the instrument may be dangerous. DO NOT DISCONNECT!

Turn on power and raise the chassis to the top of the unit using the Down/Off/Up switch on the front panel. Set the Local/SetRPM/Remote switch to “Local” and turn the RPM Adjust knob clockwise until the display reads approx. 500 RPM. Visually inspect the shaft and electrode body for any obvious wobble during rotation. Check that the top of the electrode body rests lightly against the white O-ring on the shaft. Stop rotation by switching to “SetRPM”.

Carefully place a 400 ml beaker in position in the vial plate.

IMPORTANT: Be sure that the beaker is positioned squarely upright in the vial plate. An unstable beaker could cause spilled stationary phase or damage to the rotator.

Lower the rotator chassis onto the beaker using the Down/Off/Up switch. The lift system will halt the chassis on the beaker.

IMPORTANT: The Rotator System is designed for use with a specific level of fluid in the cell vial. The ends of the working, reference, and auxiliary electrodes are approximately 1.950 inches from the beaker cap during operation. Therefore, the cell vials must be filled to within 1.500 inches of the top of the vial. Fill levels for the three standard beakers:

250 ml beaker	approx. 160 ml
400 ml beaker	approx. 275 ml
600 ml beaker	approx. 460 ml

This allows for quick and efficient setup of experiments as well as repeatable fluid dynamic characteristics in the vials.

The solution should be thoroughly degassed before use with the gas purging function on the front panel of the unit. The most effective degassing is done by using a low pressure helium source connected to the gas fitting on the rear panel. A porous frit produces good helium solubility in the sample by means of a finely distributed bubble stream. The “Gas Adjust” valve on the front panel controls the rate of purging. Dissolved air in the solution will cause unwanted bubbling or cavitation when subjected to the stirring action of the rotator.

4.0 Use and Typical Applications

5.0 Adjustments

6.0 Maintenance/Service

6.1 Changing The Working Electrode

Attach the electrode body to the protruding shaft by threading it on lightly, while holding the shaft steady with the knurled shaft skirt. DO NOT BEND! Be careful and turn slowly-you should feel the contact plunger compress against the electrode material. Turn the electrode until it rests lightly against the teflon (TFE) O-ring. DO NOT OVERTIGHTEN! If the electrode is over-tightened, it will align with the very end of the threaded portion of the shaft and will be eccentric. By threading the electrode against the O-ring, the ground diameter of the shaft will align the electrode and allow for a more consistant position.

To remove, reverse above operations.

Figure 6-1 *Working Electrode Assembly*

6.2 Repolishing Electrodes

1. Carefully remove the electrode as outlined above in Section 6.1.

2. Polish the electrode according to instructions enclosed in the PK-3 Polishing Kit that comes with the RDE accessories:

The objective of polishing the electrode is to remove the products of the redox reaction or physical adsorption which accumulate during some experiments. The rate of electrode coating (and corresponding decrease in responsiveness) will depend upon the following factors:

a. Analyte molecule
b. Concentration of the analyte molecule
c. Composition of stationary phase
d. Applied potential
e. Frequency of use

Electrodes used in electrochemistry could need repolishing after each experiment, depending on the application. Rotating disc and hydrodynamic modulation applications have the advantage of a moving solution which helps to remove redox products and often deals with lower concentrations of analyte than are typically seen with voltammetry methods in nonstirred solutions. The need for pol-

ishing varies greatly with the application. The general rule is that polishing is justified when a gradual decrease in electrode response is observed. Electrodes also can accumulate contaminants by adsorption from the environment. Cigarette smoke, aerosols, and other airborn materials can be adsorbed quite easily.

The polishing process should remove a neglible amount of electrode material. A series of one to three different abrasive systems are used, progressing from coarse particle sizes to very fine polishing powders. Most electrode surfaces need only a single polishing step to physically remove the contaminants. Precious metal (gold, silver, and platinum) electrodes may do best with either an alumina or a diamond polish. It is best to start with the diamond polish and then determine if the desired results are obtained with that step alone. Chemically modified (gold/mercury amalgam) and glassy carbon electrodes tend to require a two or three step series of abrasives to restore the original mirror-like finish.

All polishing steps require extensive rinsing and flushing of the electrode before moving on to the next stage. Without thorough washing, minute particulate from the previous polishing step will hinder the progression towards a finely polished surface. A major concern of polishing is that you do not erode the softer electrode material below the the level of the supporting plastic or glass. This can be avoided by always polishing the electrode on a very flat surface. Always follow the general precautions listed below when handling your working electrode:

a. It is important to use only BAS-manufactured polishing materials and follow the recommended procedure. The PK-3 kit provides the materials needed for all polishing procedures.

b. Polish the electrode on pads attached to the heavy glass provided in the polishing kit. You must keep the electrode as parallel to the surface of the glass as possible when polishing. This is especially important with the RDE Working Electrodes and will ensure that the surrounding plastic is not worn unevenly. You must be sure to polish very slowly to maintain the surface as flat as possible. Any deviation will be obvious when the electrode rotates.

c. Never attempt to remove the actual electrode material from the supporting plastic. This will destroy the electrode.

d. Do not heat the electrodes when drying; allow them to dry at room temperature. This will cause problems due to the difference in the coefficients of expansion of the electrode material and the supporting plastic or glass.

e. You must be very careful that no debris enters the open back of the electrode body. The opening must be kept clean in order for the spring-loaded plunger to effectively contact the electrode material.

3. Carefully replace the electrode on the rotator per Section 6.1.

6.3 Replacing Rotator Shaft

1. Carefully insert the shaft assembly through the beaker cap and into the ball bearing. The shaft should ride smoothly in the bearing with a good sliding fit. Using a small probe, move the brushes out of the way one at a time and slide the shaft into the coupler. Be careful that the two align and fit smoothly. Spin the motor and observe the end of the shaft. It should turn in a tight, concentric circle around it's axis.

2. Tighten the coupler clamps(motor side clamp first)until the shaft and motor are firmly gripped by the coupler. You should alternately tighten each screw a small amount so that the gaps on each side of the clamp remain even during the entire process.

3. Replace the chassis cover.

4. To remove, reverse above operations.

6.4 Replacing Rotator Bearing

1. Gently slide the bearing housing into the opening in the bottom of the rotator chassis. Bolt the housing into place with 3 #0-80 x 1/4 socket head cap screws. Be sure to alternately tighten each screw a small amount at a time so that the housing is seated evenly.

2. Return the beaker cap to its position under the rotator chassis. The small stainless steel pin in the beaker cap should be oriented so that it protrudes into the small hole on the bottom of the chassis. Fix the cap to the chassis with 3 shoulder bolts, making sure that the springs are on the shoulder bolts between the cap and the chassis. The shoulder on the bolts must bottom cleanly against the chassis in order for the microswitch to work correctly. Manually compress the springs several times-they should compress freely with no binding. Check that the microswitch is actuated when the cap is approximately 1/16" from the chassis.

3. Replace the shaft assembly as outlined in Section 6.3.

4. To remove the bearing housing, reverse above operations.

NOTE: The ball bearing inside the housing should be occasionally oiled with standard all-purpose oil. This will increase the life and performance of the rotator unit.

6.5 Replacing Working Brushes

1. Connect the brush block assembly to the 1/8" diameter shielded cable by snapping together the small 2-pin connector.

2. Flex the cable and place the brush block inside the chassis so that the dowel pin in the block is in the small hole in the chassis. Secure with a #4-40 x 1/4 binding head machine screw and a #4 flat washer. Make sure that the brushes are centered over the bore in the bearing housing.

3. Secure the cable to a chassis post with a small tiedown.

4. Replace the shaft assembly per Section 6.3. Be careful as you move the brushes to insert the shaft assembly.

5. Make sure the brushes are evenly spaced and perpendicular to the rotator shaft.

7.0 Troubleshooting