

RRDE-3A Operation Manual



CONTENTS

RRDE-3A OPERATION MANUAL.....	1
CONTENTS.....	2
CHAPTER 1. INTRODUCTION.....	4
1-1 INTRODUCTION.....	4
1-2 FEATURES	4
1-3 SPECIFICATIONS	5
CHAPTER 2. GENERAL INFORMATION	6
2-1 USER UPDATES	6
2-2 TECHNICAL CHANGES.....	6
2-3 DAMAGED SHIPMENT	6
2-4 PRODUCT WARRANTY	6
2-5 SERVICE INFORMATION	7
CHAPTER 3 INSTALLATION.....	9
3-1 SHIPMENT INSPECTION.....	9
3-2 OPTIONS	10
3-3 INSTRUMENT SETTING	11
3-4 POWER REQUIREMENTS	11
3-5 BACK PANEL CONTROLS AND CONNECTIONS.....	12
3-6 POWER CORD CONNECTION.....	13
3-7 MAKING CONNECTIONS.....	13
3-7-1 Model 2325.....	13
3-7-2 Gas Inlet Connection	16
3-8 PLACEMENT	17
3-9 PURGE LINES.....	17
3-10 O-RING REPLACEMENT	19
CHAPTER 4. INSTRUMENT DESCRIPTION	20
4-1 RELEASE THE WORKING ELECTRODE FROM MOTOR SHAFT ASSEMBLY	21
CHAPTER 5. OPERATION.....	23
5-1 REMOTE CONTROL.....	23
5-2 GAS PURGE.....	23
5-3 GENERAL INSTRUCTIONS	24
5-4 INITIAL INSTRUMENT OPERATION.....	25

<i>Notice</i>	26
5-5 TYPICAL TEST PARAMETERS	27
5-6 SPIN COATING	28
CHAPTER 6. MAINTENANCE	29
6-1. GENERAL MAINTENANCE	29
6-2. REPLACING WORKING ELECTRODE	29
6-3. RE-POLISHING ELECTRODES	30
6-4. SPARE PARTS FOR RRDE-3A	31
6-5. REPLACEMENT OF SILVER-CARBON BRUSHES	33
6-6. REPLACEMENT OF SHAFT ASSEMBLY	35
6-7. REPLACEMENT OF SHAFT CONTACT PROBE	37
6-8. REPLACEMENT OF HEAT SHRINK TUBING	38
6-9. REPLACEMENT OF BEARING ASSEMBLY	39
CHAPTER 7. TROUBLE SHOOTING	41
7-1 GENERAL	41
7-2 REPORTED PROBLEMS	44
APPENDIX	49
A-1 WORKING ELECTRODES	49
A-1-1 RRDE/RDE	49
A-1-2 DRE – Disk Replaceable Electrode	511
A-2 REFERENCE ELECTRODES	54
A-3 NON AQUEOUS REFERENCE ELECTRODE: RE-7	55
A-4 RHEK – REVERSIBLE HYDROGEN ELECTRODE KIT	56
A-5 COUNTER ELECTRODES	59
A-6 PK-3 ELECTRODE POLISHING KIT	60
How to use:	60
A-7 CONNECTING OTHER POTENTIOSTAT	61
A-7-1 CHInstrument	61
A-7-2 BioLogic Potentiostat	64
A-7-3 Gamry R600	67
A-7-4 Ivium potentiostat	71

Chapter 1. Introduction

1-1 Introduction

RRDE-3A is a miniature rotator system for use in constant rotation and hydrodynamic techniques by rotating ring disk electrode in electrochemistry field. A short stainless steel shaft and 12 mm diameter electrode provide a concentric circle electrode system that is capable of high accuracy.

RRDE-3A is electronically controlled by a proportional-integral closed loop circuit driven by a DC servomotor. The compact electrodes are easily and rapidly interchangeable. The unit also provides an adjustable valve system for inert gas purging inside the cell vial.

RRDE-3A can be operated as a stand-alone unit and directly controlled by Model 2325 or other instruments. During or between electrochemical analyses, a built-in gas control allows to purge the sample.

Besides, easy to remove and replace the cell vial, easy for rinsing, cleaning, and replacing electrodes.

1-2 Features

1. Remote and manual controlled rotation
2. Quasi-oxygen free glass cell design (up to 100 mL)
3. Gas line connector is available
4. Remote and manual controlled purge lines
6. Teflon Cap is adapted for ALS reference and counter electrodes
7. Cell lead connects directly to Model 2325 and CHI potentiostats
8. Open architecture for easy access to cell

1-3 Specifications

Size	190 (w) x 230 (d) x 400 (h) mm
Weight	6 kg
Rotational Range	100 to 8,000 RPM
Accuracy	Error , < 2% at < 1,000 RPM, < 1% at > 2,000 RPM
Rotator Shaft	Stainless steel
Motor	12 V, ironless core, low inertial DC servo
Power	< 100 VA 100-240 VAC, 50/60 Hz
Operating Temperature	10 to 50 Celsius degree
Inlet Gas Pressure	5 psi (34 KPa) maximum
Remote Control	Motor ON/OFF TTL or relay input to back panel connection. Purge TTL or relay input to back panel connection

Chapter 2. General Information

2-1 User updates

In order to receive any updated information about product, and valuable information related to current and other ALS products, please register your e-mail address at our local distributors. We would like to know about your present status and interests regarding electrochemical analysis.

2-2 Technical changes

We reserve the right to make technical changes to improve the instrument without notice.

2-3 Damaged shipment

Breakage of any part of this instrument during shipping should be reported immediately to the freight handler and ALS Customer service.

It is necessary to keep the original packing box and contents for inspection by the freight forwarder. ALS will replace any new instrument damaged in shipping with an identical product as expediently as possible after the claim filing date. Claims not filed within 7 days after shipping may be invalid.

Do not return damaged goods to ALS directly. Please contact with your local distributor informing them of its damaged status.

They will contact with our service department.

2-4 Product warranty

ALS Co., Ltd warrants equipment manufactured by the company to be free from defects in material and workmanship for a period of 90 days from the date of shipment. This assumes normal usage under commonly accepted operating parameters. ALS agrees to either repair or replace, at its sole option and free of part charges to the buyer, any parts of such instrumentation which, under proper and normal conditions of use, prove to be defective within 90 days from date of shipment. Electrochemical cells and working electrodes are warranted for 30 days.

ALS neither assumes nor authorizes any person to assume for it any other liability in connection with the sale, installation, service, or use of its instrumentation.

All products manufactured by ALS are tested and inspected prior to shipment. Upon prompt notification by the buyer, ALS will correct any defects in warranted equipment by its manufacture either by return of the item to our factory, or shipment of a repaired or replacement part. ALS will not be obliged, however, 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 by chemical action or corrosion.

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

Limited obligations covered by this warranty include:

In the case of instruments not of ALS manufacture, the original manufacturer's warranty applies.

Shipping charges under warranty are covered only in one direction. The buyer is responsible for shipping charges to the factory, if return of the part is required.

Expendable items including disposable items such as working electrode, reference electrodes, source lights, panel lights, fuses, etc. are excluded from the warranty

2-5 Service information

ALS provides a skilled service staff to solve your equipment oriented problems. For further details, please contact us by e-mail (service@bas.co.jp).

Following discussion of your specific difficulties, an appropriate course of action will be described and the problem resolved accordingly.

Please contact with local distributor and describe to them the problem you are having in full detail.

They will obtain a RETURN MERCHANDISE AUTHORIZATION NUMBER (RMA#). The RMA# identifies you as the sender. All correspondence and shipments should be sent to ALS.

Chapter 3 Installation

3-1 SHIPMENT INSPECTION

After unpacking the instrument carefully, check the package contents and inspect for breakage. **Table 3-1** lists the parts of the **RRDE-3A**. This list is subject to change. Please refer to the packing slip with your instrument. Assembly of these various parts will be outlined in the following chapters.

Please keep the shipping box and packing material until you have fully tested the unit to be certain that no damage was incurred during shipping.

If a shortage exists, please contact with local distributor or ALS Customer Service and describe the shortage. A replacement part will be sent immediately subject to stock availability.

If you do not find out one of accessories, please contact with distributor or local dealer **within one month** from RRDE-3A arrival date. Otherwise you won't get full support.

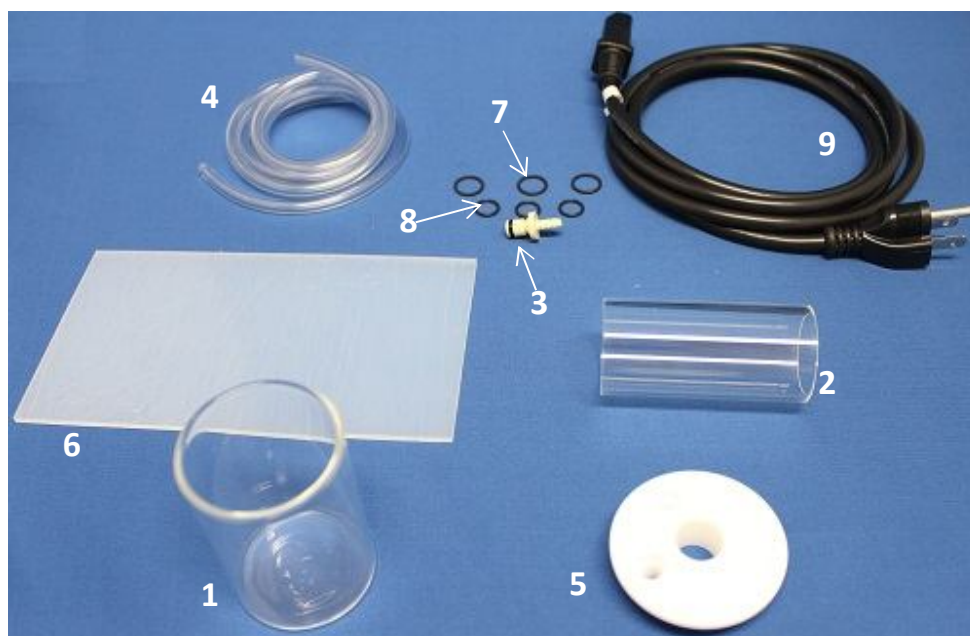


Figure 3-1-1 RRDE-3A Accessories

	Cat. #	Description	Qty	Inspect
	012623	RRDE-3A Rotating Ring Disk Electrode Apparatus Ver.1.2	1	
	RRDE-3A Accessory kit consists of			
1	012632	Sample vial (100mL)	1	
2	012064	Spin coating adapter	1	
3	012065	Male connector for gas purge (PP)	1	
4	013392	TYGON tubing , OD1/4 x ID1/8	1	
5	013271	RRDE-3A Teflon cap V.2	1	
6	012642	RRDE-3A Silicon sheet 100x180mm	1	
7		O-ring for RRDE-3A bearing assembly	3	
8		O-ring for RRDE-3A shaft assembly	3	
9		Power cable	1	
		Quick manual	1	

Table 3-1-1

3-2 Options

Descriptions of working electrodes are available in Appendix **A-1**.
You will find details on RRDE, RDE, DRE.

3-3 Instrument setting

1. Provide a surge-free power source. Other laboratory equipment such as ovens, vortex mixers, centrifuges, and large motors may cause spikes in the power supply.
2. Make sure that all components of the system share the same ground circuit. It can be done by plugging all devices into the same multi-outlet power strip. Plugging devices into independent outlets can produce ground loops, which can produce baseline noise.
3. Place the **RRDE-3A** on a stable bench free of vibrations.
4. Select a room where temperature remains stable throughout the day. Avoid installing the **RRDE-3A** near windows air ducts, ovens, and refrigerators. A water-jacketed cell may be required for very precise work.
5. Place the **RRDE-3A** away from busy, congested areas. Remote, isolated areas are best for high-sensitivity work.
6. Avoid very dry areas and carpeted areas. Static electricity can affect instrument performance. Antistatic floor mats and bench mats are useful if spiking caused by static charge is a problem.
7. Avoid areas where radio-frequency interference is possible. Beeper-type pager can be a problem.

3-4 Power requirements

The **RRDE-3A** can be used with either 100V or 220V (50-60 Hz) power supply. Power switching regulator is used, therefore it can be used anywhere without any electronic modification.

3-5 Back Panel Controls and Connections

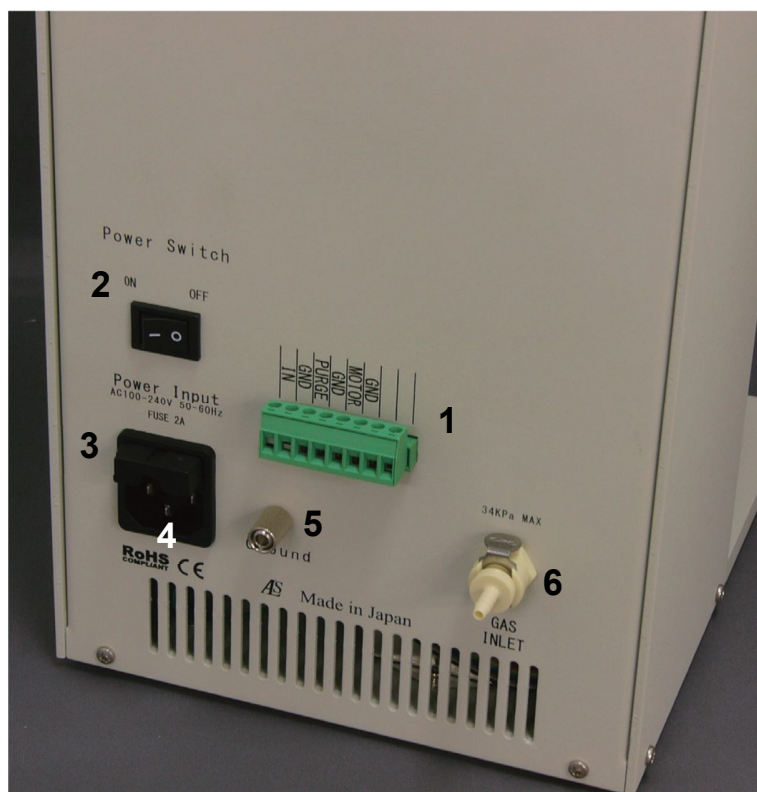


Figure 3-5-1. Back Panel

No	Function
1	Remote Terminal
2	Power On/Off Switch
3	Fuse Holder
4	Power Cord. Be sure that the power cord is connected to a grounded circuit
5	Ground terminal
6	Gas Inlet Connector for Cell Purging. Attached tubing providing connection to external gas source NOTE: 5 psi maximum

3-6 Power Cord Connection

Put the power cord into the port located in the back-panel (**Figure 3-6-1**). Then, power switch ON (position “I”) or power switch OFF (position “O”).

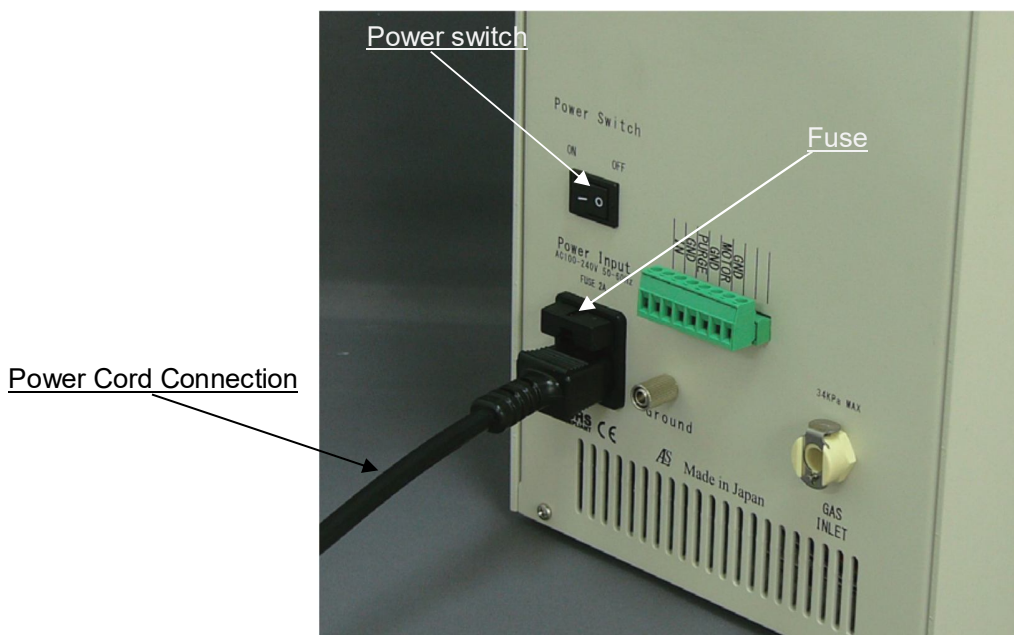


Figure 3-6-1. Power Cord Connection

The fuse is located over the power cord connector (**Figure 3-6-1**). Pull fuse holder, and check whether it works or not. If it does not work, it should be replaced with new one.

3-7 Making Connections

3-7-1 Model 2325

Here we are connecting model 2325 potentiostat . For detail about other potentiostat connections, see appendix **A-7**

Remote connection

Electrode speed rotation and **purge** can be remotely controlled from the potentiostat. Four wires are required to use potentiostat as remote controller. The remote control cable is an optional cable,

connected to the remote terminal on the back of the RRDE-3A (**Figure 3-7-1-1**).

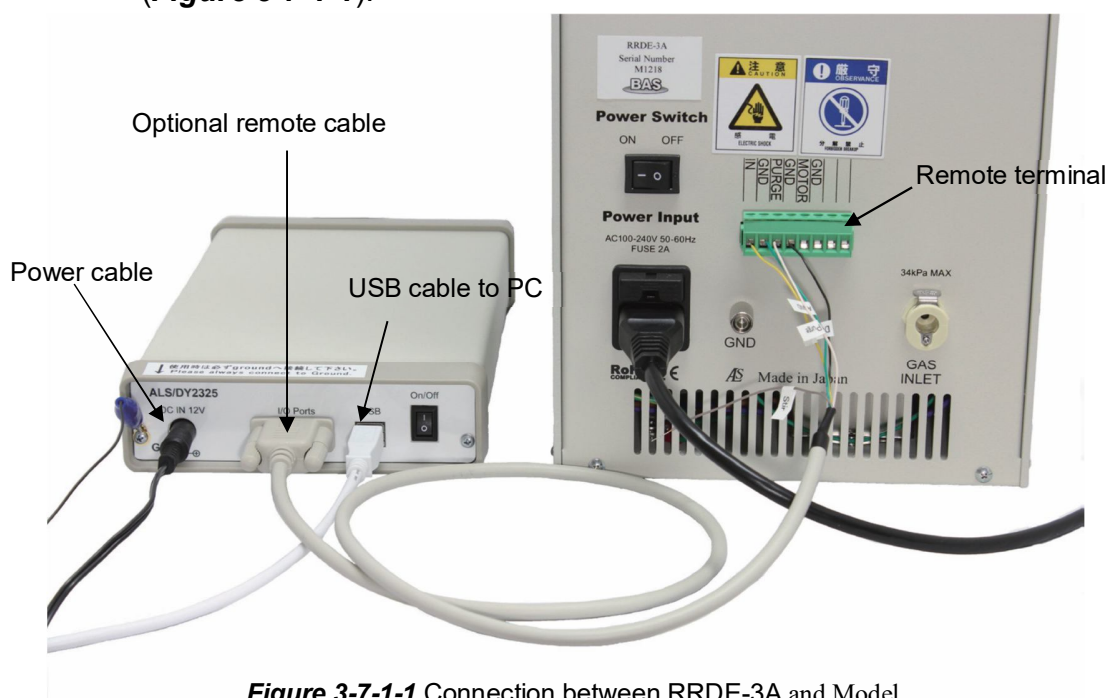


Figure 3-7-1-1 Connection between RRDE-3A and Model

I/O port for Model 2325 is connected with IN/GND/PURGE/GND of RRDE-3A via optional cable of Model 2325.

In case of coupling **RRDE-3A** with Model **2325** Bi-Potentiostat:

Model 2325 optional cable (008133)	RRDE-3A input
IN (yellow)	IN
A GND (green)	GND (next to IN)
PURGE (purple)	PURGE
D GND (black)	GND

One volt corresponds to 1,000 rpm. It is possible to set from 90 to 8,000 rpm from 0 to 8 DC Volt.

The purge and pinch valve in the RRDE-3A is controlled by TTL level active low.

In case of coupling RRDE-3A with Non-ALS Instruments, the RRDE-3A should work, at least in manual mode, with virtually any instrument. Electrode cables should be attached to the appropriate connector according to device's specifications.

Cell Connections

The **reference electrode** and **counter electrode** lead wires extend from the potentiostats. The connectors are press-on type. Simply push the connector over the corresponding pin in the electrode to make the connection (**Figure 3-7-2-1**). Alligator clips are color coded to the electrode it attaches.

The **ring electrode** and the **disk electrode** connector are located under the motor shaft assembly.

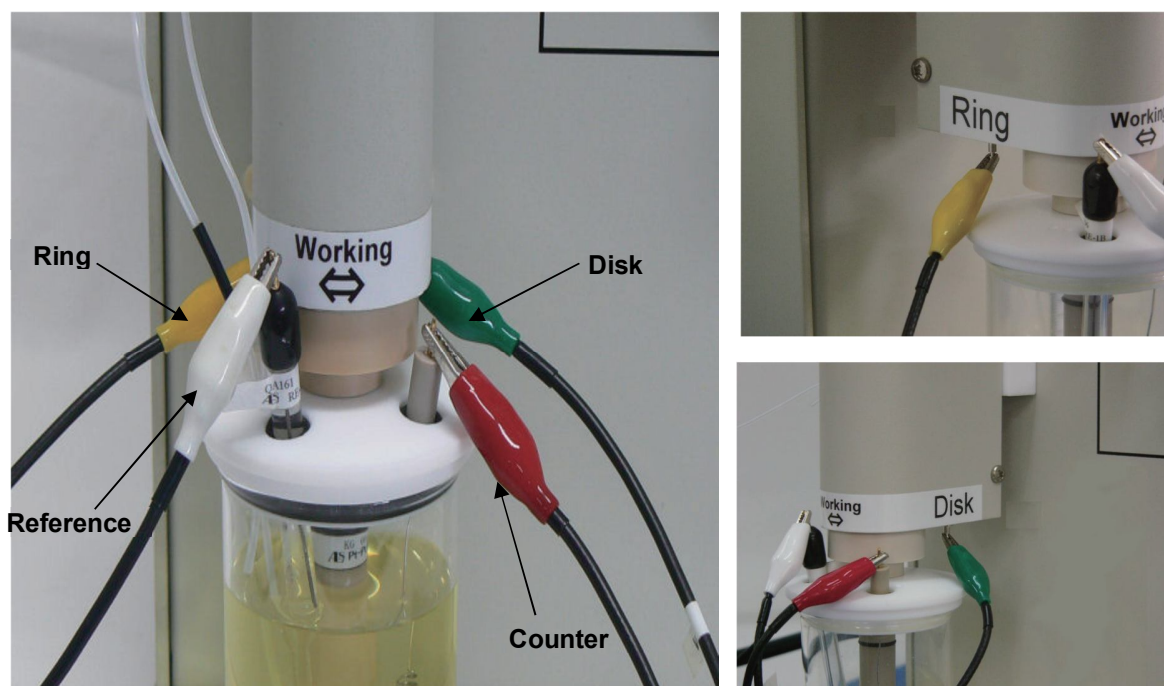


Figure 3-7-1-2. Electrode lead connections

Wire color	Electrode
Green	Disk
White	Reference
Red	Counter
Yellow	Ring

3-7-2 Gas Inlet Connection

The RRDE-3A includes 0.25" O.D. Tygon tubing and a gas line fitting. One end of the fitting is connected to the 'GAS INLET' port on the rear panel of the **RRDE-3A**. To attach the line, simply push the connector (**Figure 3-7-2-1**). The open end of the Tygon tube is connected to a regulated gas supply. The inlet gas pressure must not exceed 5 psi.

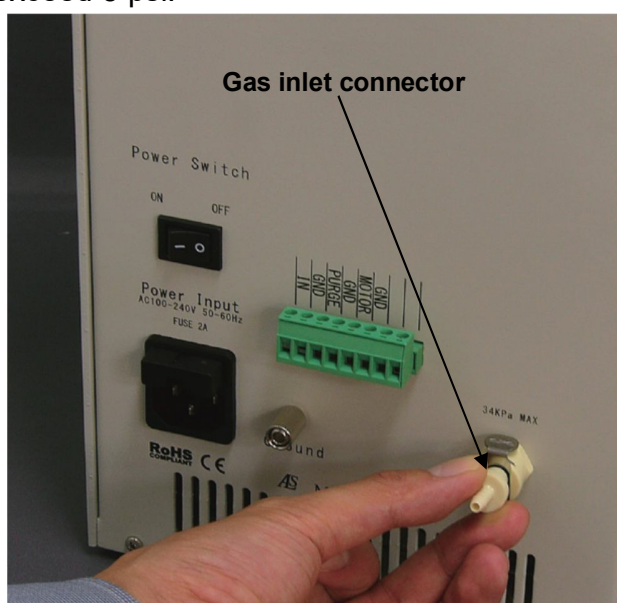


Figure 3-7-2-1. Gas Line Connection to RRDE-3A

To remove the tube from **RRDE-3A**, squeeze the retaining ring tab against the connector and then pull the tube and connector away from **RRDE-3A**.

3-8 Placement

The **RRDE-3A** is designed for easy cell replacement and to accommodate regular cells. Follow these instructions for initial cell placement:



Fig 3-8-1. Cell vial placed on the silicon plate.

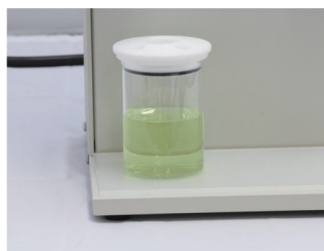


Fig 3-8-2. Teflon cap putted on the cell vial

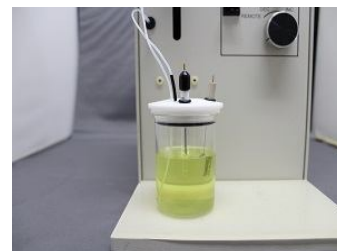


Fig 3-8-3. Reference, counter electrode and purge tubes are fixed at the Teflon cap.



Fig 3-8-4. Six holes are located on the Teflon cap. These holes are for the reference, counter, working electrode and purge tubes. Place the electrodes in the most convenient way. The last hole is for the gas outlet.

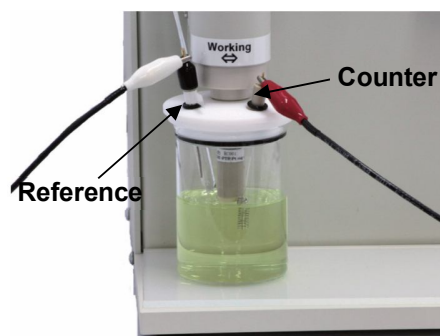


Fig 3-8-5. Adjust the desired position of the electrodes by moving O-ring.

3-9 Purge Lines

The **RRDE-3A** has the ability to purge the sample solution with an inert gas. The purge removes oxygen by bubbling with an inert gas, typically nitrogen or argon through the solution.

The purge consists of two Teflon tubes going into the cell through the Teflon cap (**Figure 3-9-1**).

The tube with the black cover should be above the sample surface and the other one should be immersed into the sample solution.

You can adjust the tube length by moving the black part of the tube if needed.

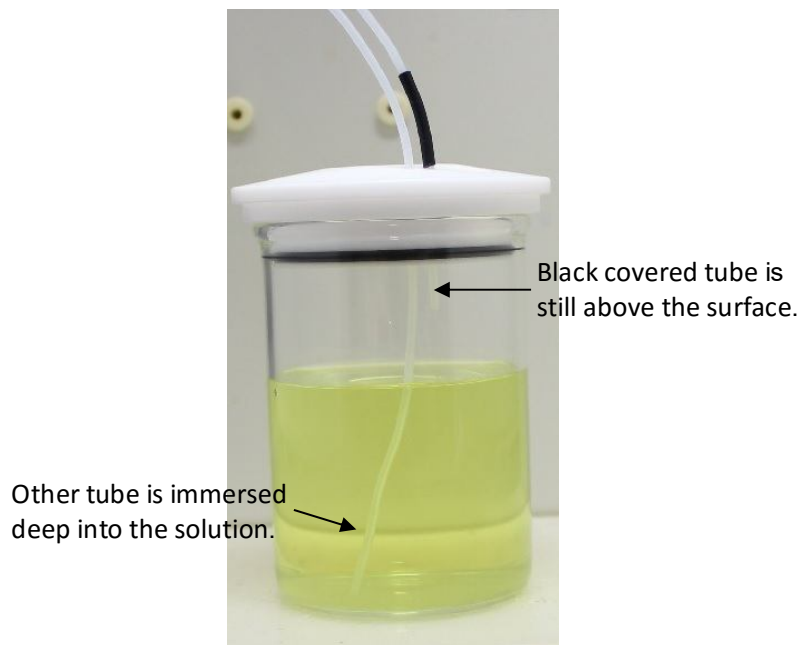


Fig 3-9-1. tubing.

3-10 O-ring replacement

Two kinds of O-ring are used at rotator shaft (**Figure 3-10-1**):

- One for RRDE-3A **bearing assembly (A)** is used to improve airtightness between Teflon cap and shaft assembly, so oxygen penetration from atmosphere is prevented.
- Other one for RRDE-3A **shaft assembly (B)** is used to protect from corrosion of electrical contact for RRDE electrode.

If **RRDE-3A** is used frequently, these O-rings should be replaced with new ones every 3 month, and trouble with corrosion would be prohibited.

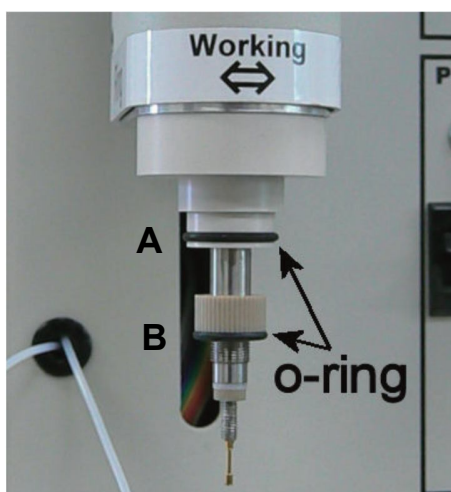


Figure 3-10-1. O-ring

Chapter 4. Instrument Description

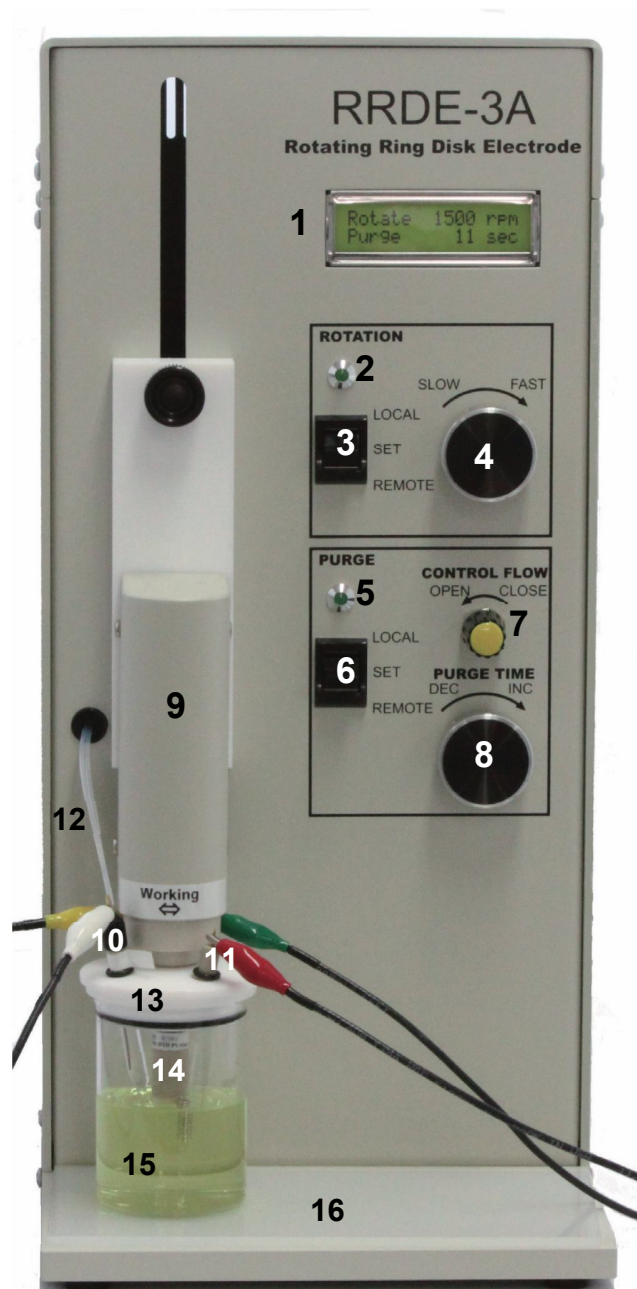


Figure 4-1. Panel of RRDE-3A

Front panel components in RRDE-3A

No	Description	No	Description
1	Digital rpm and purge time display	9	Motor shaft assembly
2	rpm LED : The LED is ON when rotation is turned ON; either remotely or manually	10	Reference electrode
3	Rotation control switch (Set, Local, Remote)	11	Counter electrode
4	Rotation speed knob (X10)	12	Teflon tube for gas purge
5	Purge LED : The LED is ON when purge is turned ON, either remotely or manually	13	Teflon cap
6	Gas purge switch (Set, Local, Remote)	14	Working electrode
7	Gas purge flow control knob	15	Glass cell vial
8	Purge time knob	16	Silicon sheet

Table 4-1

4-1 Release the working electrode from Motor shaft assembly

Power OFF **RRDE-3A** device. Then after removing all cables from electrode (reference, counter, ring and disk electrode), turn counter clockwise the motor shaft assembly.

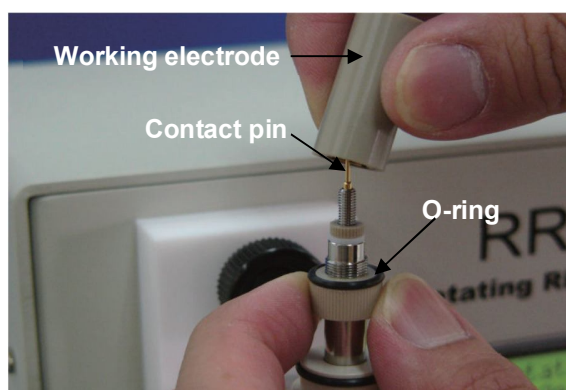


Figure 4-1-1. Motor shaft is turned counterclockwise slowly, and working electrode is unscrewed carefully.

The working electrode is just screwed on the shaft. The contact is made with the contact pin attached in the shaft. As the contact pin is very sharp and fragile, handle it with care. To remove the working electrode, just unscrew it carefully (**Figure 4-1-1**).

An O-ring is placed between the working electrode and the shaft to prevent from any corrosion due to the acidity or basicity of electrolyte.

Chapter 5. Operation

Notice: Please use Protective eyeglasses to protect yourself from chemicals under experiment.



5-1 Remote Control

The remote connectors of the **RRDE-3A** allow controlling rotation and gas purge functions by an external unit. The **RRDE-3A** is specifically designed to be controlled by the appropriate commands from potentiostats as Model **2325** or **CHI Electrochemical Analyzer**.

When it is not in use, please make sure power is turned out.

These functions can be activated by either a TTL signal or from any controller providing a contact closure to the ground. All lines are active low (negative edge triggered).

Follow operational instructions given under “**5-4 Initial Instrument Operation**” for remote rotation.

5-2 Gas Purge

The LED is ON when the purge function is ON, either manually or remotely.

To control purge remotely, the purge and rotate remote cable should be connected on the back panel of the RRDE-3A. Time of purging can be controlled manually or via the “immediate purge/rotate” option under the control menu of the CHI operating software.

5-3 General Instructions

Switch power OFF on the back panel. Connect the power cable. The main power connection must have proper grounding.

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

Check that the top of the electrode body is connected on the shaft firmly. Then switch power ON.

Set the rotation speed at 1,000 RPM and start rotation. Please do not touch the RRDE shaft when it is rotating. Visually inspect the shaft and electrode body during rotation. It should be straight.

Stop rotation by switching the rotation button.

The solution should be thoroughly purged with the gas purging function on the front panel of the unit, before use. The most effective purging is done by using a low pressure inert gas source connected to the gas tubing on the rear panel for 15min. The “gas adjust valve” controls the rate of purging.



Figure 5-3-1.

5-4 Initial Instrument Operation

Completing all front and back panel connections, the following procedures are needed for initial use to connect potentiostats.

1. Push rear panel power switch to OFF ("O").
2. Remove the electrode by unscrewing it.
3. Polish the electrode by following the instructions in the Polishing Kit. See Page 32 for more information.
4. Replace the electrode. Note that a slight upward pressure may be applied to the electrode body to start the threads. When the electrode is replaced with another working electrode, the power switch should be "OFF".
5. Place a test solution in the cell vial. A common test solution is 1 mM potassium ferricyanide in 1 M KCl.
6. Push rear panel power switch to ON ("I").
7. Place the reference and counter electrodes inside two holes of the Teflon cap and attach the cell leads. Start purging the solution with inert gas as described previously. For this initial experiment, purging for over 15 minutes will be adequate. Please regulate gas pressure carefully.
8. If the RRDE is used with the Model 2325 or CHI Model as potentiostats, the control of the rotation speed can be achieved remotely via **IMMEDIATE PURGE/STIR** option under the **CONTROL** menu or manually from the front panel
9. The instrument is ready to use. Now the electrode will begin to spin when the **RUN** command is initiated from the software.
10. After the experimental run, the motor shaft assembly can be raised and electrodes can be rinsed or polished as needed. The system is then ready for the next experiment.

Notice

1. The range of rotation speed of RRDE-3A is from 100 to 8,000 rpm.
2. Please run the electrochemical measurement when indication of RPM on LCD display becomes constant.
3. Depending on room temperature, it takes several minutes until a low-speed rotation becomes stable. Please let sufficiently RRDE-3A apparatus warm up.
4. For the REMOTE speed control, please use a Model 2325 electrochemical analyzer.
5. If it is used at high-speed rotation, a whirlpool may occur in the cell around the rotation axis of the electrode, the oxygen in the air will be dissolved. In addition, noise may be caused.
6. Please set rotating speed to less than 3,000 rpm, in order to make the life-time of motor and silver-carbon brush longer.
7. Please use an inert gas and avoid the usage of explosive gas, like hydrogen gas. It could cause a risk of gas leakage.
8. During rotation sometimes the motor may be noisy due to the rotation speed, but there is no influence to the measurement.
9. RRDE-3A is non explosion-proof device.

5-5 Typical test parameters

Working Electrode:	RRDE (Gold disk and platinum ring)
Reference Electrode:	Ag/AgCl
Counter Electrode:	Platinum Wire
Test Solution:	0.5 mM Ferricyanide/1 M KCl
Initial E:	+600 mV
Final E:	-200 mV
Scan Rate (V):	10 mV/S
Rotation Rate:	100 to 6,000 RPM
Sensitivity:	10^{-5} or 10^{-4} A/V

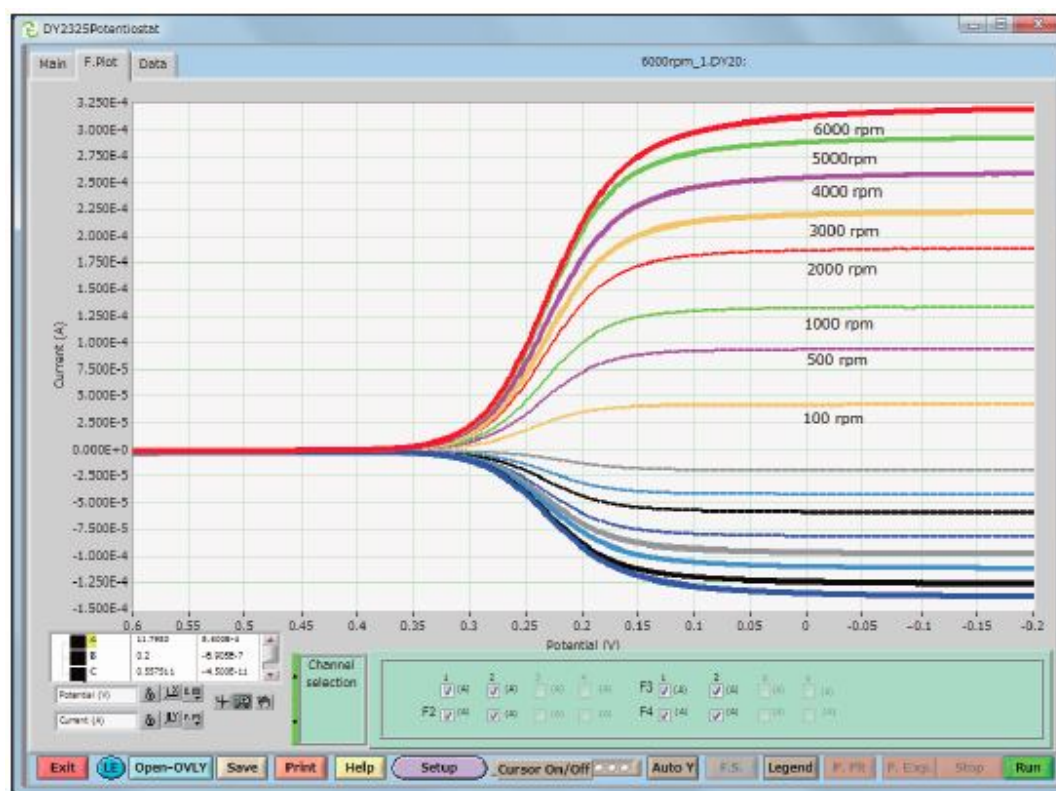


Figure 5-5-1.

5-6 Spin coating

In case of polymer is coated onto the electrode, please use the following way as reference. **RRDE-3A** motor shaft is taken away from Teflon cap. Its motor housing is rotated at 180 degree (**fig.5-6-1**). Its contact pin is positioned at top, and RDE or RRDE electrode is assembled with motor shaft unit. The acrylic cover is mounted at the top of motor housing. After power is ON, and rotation speed is fixed at proper speed, then a droplet of polymer is dropped on the electrode surface. You can get chemically modified electrode by spin coating. Its membrane thickness depends on rotation speed, sample viscosity, applied volume and working temperature.



Figure 5-6-1 Fasten the knob of RRDE-3A, and Teflon plate moves up.



Figure 5-6-2. Fixe the Teflon plate at the top position.



Figure 5-6-3 Rotate at 180 degree the motor housing



Figure 5-6-4 Now the contact pin is positioned at the top. Pin is sharp, handle with care. RDE or RRDE is mounted here.

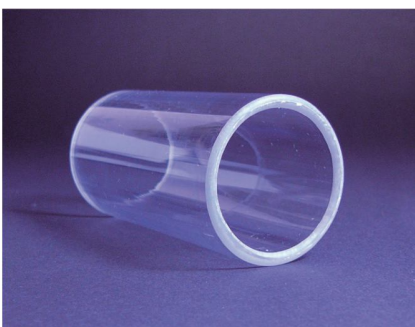


Figure 5-6-5 Acrylic spin coating cover, to be mounted on the top of the motor housing.



Figure 5-6-6 It's ready for spin coating experiment. A sample you want to coat is dropped on RDE or RRDE. The motor is rotated for a few minutes, then the chemically modified electrode with a constant membrane thickness is available.

Chapter 6. Maintenance

6-1. General Maintenance

The **RRDE-3A** is an improving instrument and, with proper care, should give years of service.

The following is a brief list of cautions and general maintenance considerations that will extend the lifetime of the instrument. When turn on the power, please do not touch **RRDE-3A** shaft assembly.

1. Follow customary, good laboratory practices.
2. Clean all spills, especially salt solutions, on or near the cabinet immediately.
3. Avoid placing the unit in a corrosive atmosphere.
4. Avoid dropping, shaking, or other forms of mechanical abuse to prevent loosening of components or subassemblies.
5. Clean gas lines (rinse and wipe dry) after use.
6. Do not bend the auxiliary electrode when removing or placing the cell vial. Repeated bending will cause break of the wire.

6-2. Replacing Working Electrode

Attach the electrode body to the protruding shaft by threading it on lightly and holding the electrode shaft steady with the shaft screw.

DO NOT BEND! Be careful and turn slowly - you should feel the contact pin compress against the electrode material. Turn the electrode until it rests lightly against the rubber O-ring. **DO NOT OVERTIGHTEN!**

If the electrode is over tightened, it will be eccentric and will be broken. By threading the electrode against the O-ring, will assure the conductivity between the shaft contact probe and the electrode.

To remove the electrode, reverse above operations.



Figure 6-2-1 Working Electrode Assembly

Warning: During RDE or RRDE electrode assembly with shaft, be careful to not hurt your finger with the sharp pin.

6-3. Re-polishing Electrodes

1. Carefully remove the electrode as outlined above.
2. Polish the electrode according to instructions using **PK-3 Polishing Kit**. See Appendix **A-6**.

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:

- Chemical species of analyte molecule
- Concentration of the analyte molecule
- Composition of stationary phase
- Applied potential
- Frequency of use

Electrodes used in electrochemistry may need repolishing after each experiment, depending on the application. Rotating ring disk electrode 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 and often deals with lower concentrations of analyte than those in voltammetry methods with non-stirred solutions. The need for polishing varies greatly with the application.

Polishing is justified when a gradual decrease in electrode response is observed.

Electrodes can also accumulate contaminants by adsorption from the environment. The environment such as cigarette smoke, aerosols, and other airborne materials can be adsorbed quite easily.

The polishing process should remove a negligible amount of electrochemically active materials. To refresh the electrode, three different abrasive polish 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. Besides, rare metal (gold, silver, and platinum) electrodes need to polish with either an alumina or a diamond. To determine whether the desired results are obtained, it is best to start with the diamond polishing. Then, chemically modified (gold/mercury amalgam) and glassy carbon electrodes tend to require a two to three step series of abrasives in order to restore the original responsibility.

All polishing steps require extensive rinsing of the electrode before moving onto the next stage.

Without thorough rinsing, minute particulates from the previous polishing step will hinder the progression towards a finely polished surface. A major concern to polishing is that you do not corrode the softer electrode material below 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:

6-4. Spare parts for RRDE-3A

Some spare parts are available for your daily maintenance of RRDE-3A.

Parts No.	Spare Parts	photos
013229	Carbon brush for RRDE-3A Ver.1.2	 <p>Figure. 6-4-1</p>
012624	RRDE-3A Shaft assembly	 <p>Figure. 6-4-2</p>
013342	RRDE-3A Shaft contact probe repair kit	 <p>Figure. 6-4-3</p>
012625	RRDE-3A Bearing assembly	 <p>Figure. 6-4-4</p>
012651	RRDE-3A Upgrade maintenance tool kit	 <p>Figure. 6-4-5</p>

WARNING: For all manipulation in the device, power off the device and unplug it.

6-5. Replacement of Silver-carbon brushes

The electrical resistance between **silver-carbon brushes** and the **shaft** will increase over time. If you get noisy signal or that the current value is smaller than usual please exchange the **silver-carbon brushes** for new ones.



6-5-1. Unscrew four metal screws, which are used for fastening the white motor assembly cover of RRDE-3A, with a screwdriver (#1 or #2).



6-5-2. Remove the motor assembly cover.



6-5-3. Unscrew two tiny screws connecting between the brush base and the bad **silver-carbon brush** unit with a screwdriver (#000).

Never lose tiny screws after the removal and keep them for the



6-5-4. Remove the old **silver-carbon brush** unit and put the new one on the brush base.



6-5-5. Make sure to fix the new **silver-carbon brush** unit with tiny screws.



6-5-6. Put the motor assembly cover and fix it.



6-5-7. Please rotate the rotator by **8000 rpm** for **10 minutes** for making the low resistance between **silver-carbon brushes** and **shaft** and for turning down the noisy sound during rotation.

If **shaft** becomes dirty due to dust of **silver-carbon brush**, please clean it by using cotton swab.

Please do not enforce motor shaft to stop rotation by hand, doing that will trigger the internal resettable fuse for device safety. You may need to wait some time before device gets ready to rotate again.

Note: The life-time of **silver-carbon brush** is depending on its experimental conditions. The RRDE-3A can be used from **100 to 8,000 rpm**.



6-5-8. Silver-carbon brush

However its life-time is directly related to the rotating speed and we recommend to use RRDE-3A with a speed **less than 3,000 rpm** for good performance.

The **silver-carbon brush** is a disposable item; therefore it is not covered for one year of the warranty. If its S/N ratio becomes worse, please replace with new one.

6-6. Replacement of Shaft Assembly

If you find some problem on your **shaft assembly** such as **distortion**, badly dirty surface, or **corrosion**, you should exchange it for a new one.



6-6-1. Unscrew four metal screws, which are used for fastening the white motor assembly cover of RRDE-3A, with a screwdriver (#1 or #2).

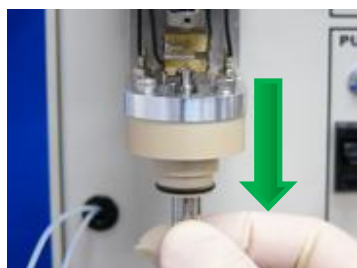


6-6-2. Loosen one screw on the lower side of the **flexible coupling** with hex key (0.89mm = 0.035inch).

*Never loosen screws on the upper side of the **coupling**. They are used for joining the motor unit and the **coupling**.*



6-6-3. Loosen the second screw on the lower side of the **flexible coupling** until the **shaft** is released, while holding the **shaft** with another hand.



6-6-4. Pull the **shaft** gently. If it does not go out smoothly because of **silver-carbon brushes**, please release them by loosen the tiny screws, or bend the leaf spring for **silver-carbon brush** with tweezers, slightly.

*If **shaft** is significantly damaged and hard to pull out, please remove the **bearing assemble** preceding the **shaft**.*



6-6-5. Stick the new **shaft** into the hole of the **bearing** and lift it up carefully while bending the leaf spring of **silver-carbon brush** toward the outside slightly with tweezers.

*If the shaft is lifted without the slight bending, **silver-carbon brushes** would be broken.*

*To prevent any damage on **silver-carbon brushes**, thoroughly remove them from **brass block**.*



6-6-6. Please bend the **silver-carbon brushes** for **ring electrodes** toward the outside slightly with tweezers again, to let pass the gap between Teflon separator and lower side of the **shaft**.

*If the **shaft** is moved without the slight bending, **silver-carbon brushes** would be broken.*



6-6-7. Screw two set screws on the lower side of the **flexible coupling** with hex key (0.89mm = 0.035inch).
Fix the **silver-carbon brushes** back if necessary



6-6-8. Enclose the **shaft** with white cover and then, fix it by four metal screws, with a screwdriver (#1 or #2).

6-7. Replacement of Shaft Contact Probe

There RRDE-3A **shaft assembly probe** has a **shaft contact probe** on the top and the tip of the probe sometimes gets **rusty**. If you can see some **corrosion** (**Figure 6-7-1**, left) or **distortion** of the **probe**, you should change it.

The **corrosion** of the **probe** tip could cause problems for your experiments.

Please also see the information on the dimensions of the **shaft contact probe** (**Figure. 6-7-2, 6-7-3**)

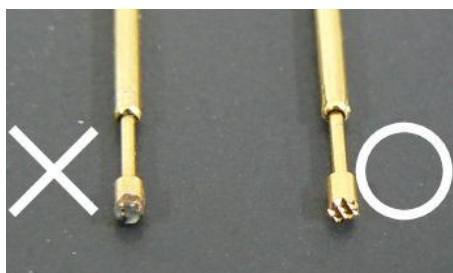


Fig. 6-7-1. Corroded **probe** on the left



Figure. 6-7-2

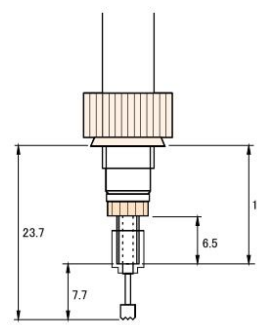
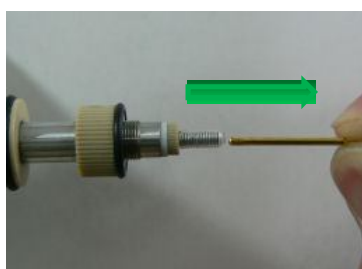


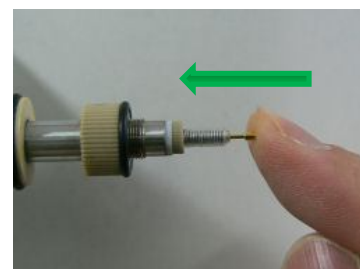
Figure. 6-7-3



6-7-4. Remove the old RRDE-3A shaft **assembly probe** by pulling it.



6-7-5. Stick the new **probe** into the hole of the **shaft**.



6-7-6. Press the **probe** carefully.
If the **heat shrink tubing** is broken during the exchange, you could not attach new **probe** and you should exchange the bad **tubing** for new **tubing** as below.

6-8. Replacement of heat shrink tubing



6-8-1. After taking out the **probe** cut the **Heat shrink tubing** with a knife.



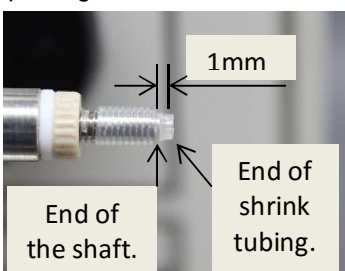
6-8-2. Remove the **Heat shrink Teflon tubing** like peeling.



6-8-3. Put a new **Heat shrink tubing** on the tip of the **shaft assembly**.



6-8-4. Shrink the **tubing** to wrap the shaft with heat gun or a drier (by 150 degrees Celsius)



6-8-5. Cut the **Heat shrink tubing** with some knife at **1mm** far from the tip of the **shaft**.



6-8-6. Attach the **probe** again.

6-9. Replacement of Bearing Assembly

If you feel that the rotator does not work smoothly, it may be caused by the **rusty bearing assembly**. (See also “Replacement of **Shaft Assembly**.”)



6-9-1. Unscrew four metal screws, which are used for fastening the white motor assembly cover of RRDE-3A, with a screwdriver (#1 or #2).



6-9-2. Loosen screw pair on the lower side of the **flexible coupling** with hex key (0.89 mm = 0.035 inch) until the **shaft** is released, while holding the **shaft** with another hand.

Never loosen set screws on the upper side of the coupling. They are used for joining the motor unit and the **coupling**.



6-9-3. Pull the **shaft** gently. If the **shaft** does not go out smoothly because of **silver-carbon brushes**, please release them by loosen the tiny screws, or bend the leaf spring for **silver-carbon brush** with tweezers, slightly.

If your shaft is significantly damaged and hard to pull out, please remove the bearing assemble preceding the shaft removal or remove it with the shaft.



6-9-4. Remove the **bearing assembly** by loosening three set screws connecting between the **aluminum frame** and the **bearing** with hex key (2.5 cm).

Be careful for the silver-carbon brushes when you unscrew the rear side of the set screw.

To prevent any damage on silver-carbon brushes, remove them.



6-9-5. Rotate the motor housing at 180 degree and wipe the bottom of the **aluminum frame** with some swab or paper.



6-9-6. Rotate the housing back to initial position and attach the new **bearing assembly** with hex key carefully.

The screw thread of the bearing assembly is somewhat weak. Don't screw the set screws very tightly.



6-9-7. Stick the new **shaft** into the hole of the **bearing** and lift it up carefully while bending the leaf spring of **silver-carbon brush** toward the outside slightly with tweezers.

If the **shaft** is lifted without the slight bending, **silver-carbon brushes would be broken.**



6-9-8. Screw two set screws on the lower side of the **flexible coupling** with hex key (0.89 mm = 0.035 inch).

Fix the **silver-carbon brushes** back if necessary



6-9-9. Enclose the **shaft** with white cover and then, fix it by four metal screws, with a screwdriver (#1 or #2).

Chapter 7. Trouble shooting

7-1 General

Trouble	No	Cause	Solution	Note
1.Noisy signal	1-1	Bad ground connection	Connect the RRDE-3A ver.1.2 and the potentiostat to the same earth ground.	
	1-2	2 The volume of solution is too much or too few.	When you use "Sample vial (100 ml, Cat. #012632)", 65 ml \pm 5 ml is suggested to use for the measurement to avoid noise.	For minimizing the noise caused by turbulence, The electrode tip should be placed in around 5 mm depth into the solution.
	1-3	Electrode is attached to the shaft in askew direction.	Remove the electrode from the shaft, and connect it to the shaft again.	
	1-4	Air bubble adsorbs on the electrode surface	Take out the electrode from the solution, rotate the electrode over 1000 rpm, and then dip the rotating electrode into the solution to eliminate air bubble with whirling flow.	Air bubble on electrode surface occurs easily after the gas purge. Air bubble is occurred during the electrochemical measurement sometimes.
	1-5	Air bubble on the reference electrode tip	Flip the reference electrode and move off the air bubble.	The alteration of the liquid temperature may cause the generation of air bubble.
	1-6	Dirty or uneven electrode surface	Polish the electrode. If some catalyst is loaded on the disk electrode, please reload the catalyst so that the catalyst layer is thin and flat.	
	1-7	Poor reference electrode.	Exchange the reference electrode for a new one.	
	1-8	After long time use, the contact resistance between silver-carbon brush and shaft has changed.	Remove the silver-carbon brushes from the shaft and clean the carbon powder on the shaft. Place Emery paper UF800, on the surface of the dirty shaft. Then, start rotation for a few seconds at 100rpm. In the case that silver-carbon brushes are consumed greatly by	The silver-carbon brushes damaged may possibly occurred by the big shock during the transportation. [Accessories] 012611 Emery paper UF800 (20 pcs) 013229 Carbon brush for RRDE-3A Ver.1.2

			friction after long time use, please replace them.	
	1-9	Silver-carbon brush shave powder is collected on the bearing part.	Open the motor cover and remove the shave powder with such as a cotton swab etc.	The carbon powder stored on the bearing may possibly cause the ring current noise in most cases.
	1-10	The big vibration of the shaft tip after long time use.	Distorted contact probe on the shaft often cause the vibration. Please replace the shaft contact probe. If the shaft contact probe is straight, please adjust and fix the screw of shaft to motor. If it does not help, please replace the shaft.	013342 RRDE-3A Shaft contact probe repair kit 012624 RRDE-3A Shaft assembly
2. Lower current than usual.	2-1	Different concentration of redox agents	Prepare the test solution again.	
	2-2	High resistance between silver-carbon brushes and shaft.	Remove the silver-carbon brushes, and attach it again or exchange the silver-carbon brush for the new one. Place Emery paper UF800, on the surface of the dirty shaft and rotate for a few seconds at 100 rpm.	013229 Carbon brush for RRDE-3A Ver.1.2 012611 Emery paper UF800 (20pcs)
	2-3	High resistance contact probe on the shaft and disk electrode.	Replace the contact probe or exchange the shaft assembly for a new one.	If the serial number of rotator is after M1131 (purchased after March 2012), you can replace contact probe with 013342 RRDE-3A Shaft contact probe repair kit. If you purchase the RRDE before March 2012, due to the structure, you have to exchange the whole shaft assembly with 012624 RRDE-3A Shaft assembly.
	2-4	Deterioration of the electrode catalysis.	Polish the electrode surface or the replacement of the electrode.	
3. Overflow current	3-1	The reference electrode isn't immersed in the solution.	Dip the tip into the electrolyte. If the volume of the solution is not enough, please increase it until the electrode tip is soaked.	The recommended solution volume is 65 ml \pm 5 ml.
	3-2	Unsuitable sensitivity setting for potentiostat, or potentiostat is in bad condition.	Fix the potentiostat or update the application software of the potentiostat. Adjust the sensitivity setting.	

	3-3	Over-potential of the potentiostat compliance voltage due to unexpected high resistance between rotating electrode and counter electrode.	<p>Measure the resistance with a multimeter from tip of shaft through disk pin, or from shaft near the electrode stopper through ring pin. When the resistance is more than 1000 ohms, inspect the cause of the high resistance and fix it.</p> <p>Replace the rotating electrode.</p> <p>Confirm whether your electrolyte, counter electrode etc. have low resistance. If there is something with high resistance, fix or replace it.</p>	
4.No Current flow.	4-1	The rotating electrode or counter electrode isn't immersed in the solution.	<p>Dip the tip into the electrolyte.</p> <p>If the volume of the solution is not enough, please increase it until the electrode tip is soaked.</p>	The recommended solution volume is 65 ml \pm 5 ml.
	4-2	Disconnection of electrodes with electrode leads of potentiostat.	Connect the electrode leads to electrodes firmly.	
	4-3	Disconnection Between contact probe and disk electrode.	Replace the contact probe or exchange the shaft assembly for a new one.	<p>If the serial number of rotator is after M1131 (purchased after March 2012), you can replace contact probe with 013342 RRDE-3A Shaft contact probe repair kit.</p> <p>If you purchase the RRDE before March 2012, due to the structure, you have to exchange the whole shaft assembly with 012624 RRDE-3A Shaft assembly.</p>
	4-4	Weak electrode catalysis.	<p>Polish the electrode surface or replace it.</p> <p>If some catalyst is loaded on the disk electrode, please check the catalyst activity at first, then active catalyst should reload on the electrode surface.</p>	
5.No ring current (Disk current is available).	5-1	Loosen screw of rotating electrode	Unscrew the rotating electrode, and screw it to the end again.	
	5-2	Broken rotating electrode	Replace the rotating ring-disk electrode.	

7-2 Reported problems

Q1- When I start the rotation after setting a rotation speed, the shaft doesn't rotate and there is no movement (or it move a small bit before stopping).

A1- Please perform the two check inspections below:

1. Inspection of the shaft assembly

Unplug the power cable and switch off the RRDE-3A system.

Remove the motor cover, and check the **shaft assembly**.

The best way to check the **shaft assembly** is to remove it.

First, follow the 3 steps bellow, and if you cannot remove the **shaft assembly**, you can follow the 4th step (See also §6-6 and §6-9).



Fig7-2-1

Step 1

Unscrew four metal screws, which are used for fastening the white motor assembly cover of RRDE-3A, with a screwdriver (#1 or #2).



Fig7-2-2

Step 2

Hold the **shaft** underpart with one hand and loosen the screw pair on underside of coupling with hexagon wrench (0.89 mm) with the other hand.

CAUTION: Do not loosen the two screws on the upper side of coupling. They are used for motor connection.



Fig7-2-3

Step 3

Pull out the **shaft** gently. If the **shaft** gets stuck with the silver-carbon brush, loosen the brush's fixing screws or expand the brush head with tweezers.

(**NOTE:** If the **shaft** is severely damage and cannot be pulled out easily, please do next step in advance, and then remove the **shaft** together with the **bearing**.)

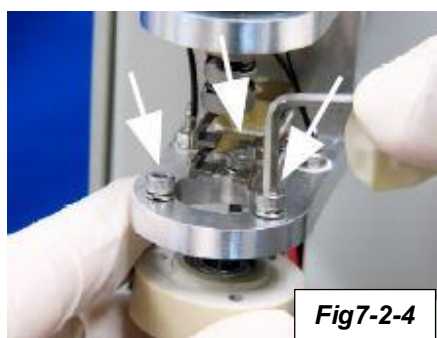


Fig7-2-4

Step 4

Remove the hexagon screws for fixing the **bearing** using the hexagon wrench (2.5 mm)

CAUTION: Be careful for the **silver-carbon brushes** when you unscrew the rear side of the set screw.

To prevent any damage on silver-carbon brushes, thoroughly remove them from brass block.

If you can see damage on the **shaft assembly**, you need to change it.
If you had to remove the **bearing** to remove the **shaft assembly**, then you need to change **the shaft assembly** and the **bearing**.

If you are in this case:

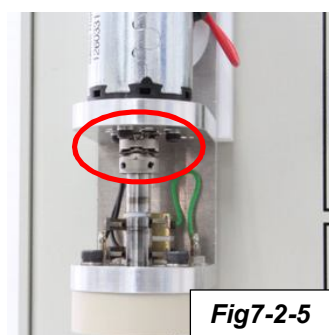


Fig7-2-5

Motor blocked by coupling.

1mm space should be present between the coupling (motor side) and the motor support.



Fig7-2-6

After removing shaft (and bearing) as described above, remove the motor by unscrewing the 3 screws with an Allen wrench 1.5 mm. Hold the motor in your hand to prevent it falling due to its own weight.



Fig7-2-7

Do not unplug the motor, it is not necessary. Now you have released the motor, you have access to the coupling's screws. Remove the coupling from the motor by loosen the 2 screws of the coupling with a hex wrench.

Place the motor back with the 3 screws and continue the inspection.

1 mm space should be present between the coupling (motor side) and the motor support.

2. Inspection of the RRDE-3A system

Once removed the **shaft assembly** and **bearing** (the 4th step above), check the motor.

Set the rotation to 1,000 rpm, and turn the rotation switch to "LOCAL", and check if the rotation can display 1,000 rpm.

If any problem occurs, contact us at: service@bas.co.jp

Q2- The purge button does not work. It is always purging the solution when the purge button is on SET or REMOTE position.

A2- Check the purging tubing inside the device, maybe they have been dislocated from the valve system. That can be occurred if the purging tubing were pulled out to far from the device.

Remove the right side panel in order to access inside the device.



Fig7-2-8

Remove the right side panel in order to access inside the device.

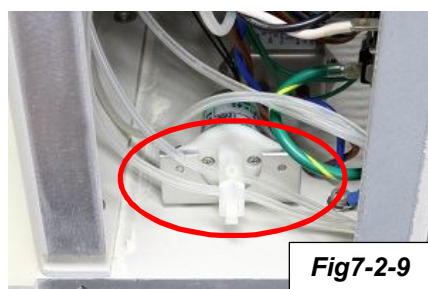


Fig7-2-9

Check the purging tubing, they should be inserted into the valve system.

If tubing are not inserted into the valve system as **Fig 7-2-10**, just insert them gently.



Fig7-2-10



Fig7-2-11

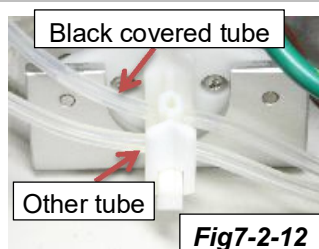
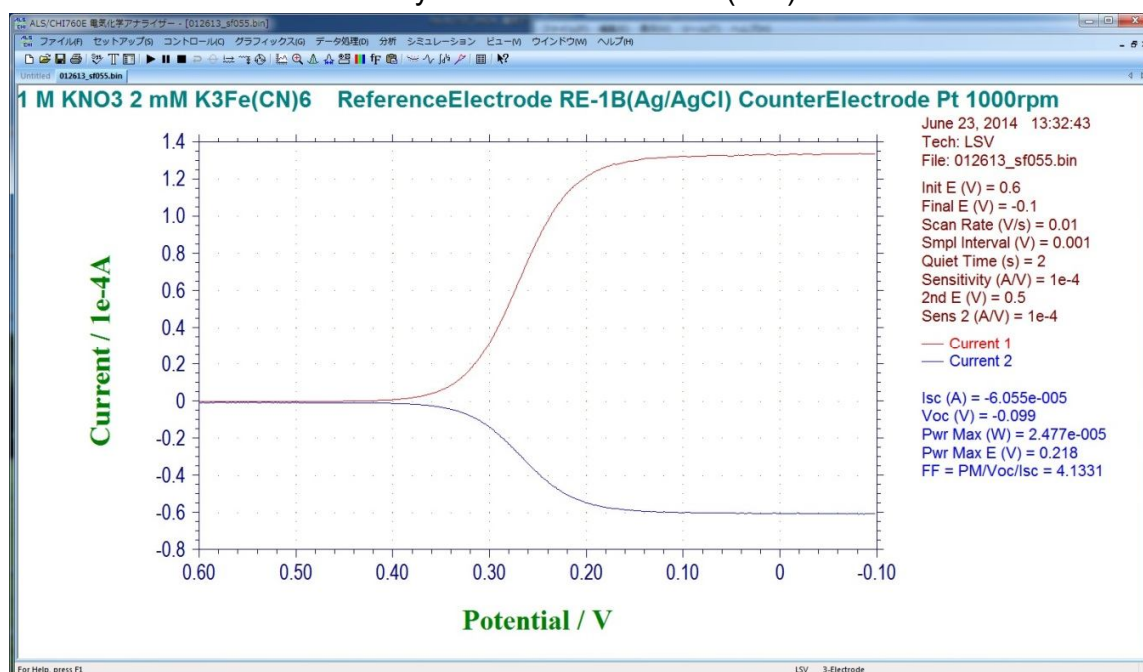


Fig7-2-12

Insert the black covered tube into the pinch nearest the valve system and the other tube into the more distant pinch from the valve system.

Q3- The acquired data are not good**A3-** Please repeat the following experiment with exactly same steps:**Step1: Polish** the electrode with the PK-3 0.05 μm alumina polishing kit as described in **A-6****Step2: Use a fresh** potassium ferricyanide sample solution1M KNO_3 2mM $\text{K}_3\text{Fe}(\text{CN})_6$ If the solution is not fresh, Potassium ferricyanide may be turned into Potassium ferrocyanide ($\text{K}_4\text{Fe}(\text{CN})_6$)**Step3: Proceed the experiment with a 1000 rpm.**

Reference Electrode:	Ag/AgCl
Counter Electrode:	Platinum Wire
Initial E:	+600 mV
Final E:	-100 mV
Scan Rate :	0.01 V/S
Sample interval :	0.001 V
Sensitivity:	10^{-4} (A/V)

If you don't get this kind of curves, contact our service: service@bas.co.jp.

Appendix

A-1 Working Electrodes

A-1-1 RRDE/RDE

Products listed in below are working electrodes for rotating ring disk electrode system, **RRDE-3A**.

Organic solvent-resistant resin, PEEK, is used as an insulator, and is able to be polished with **PK-3 Electrode polishing kit**.



Fig A-1-1

The diameters of regular rotating electrodes (Glassy carbon, platinum, and gold) are decided. **Diameter of Disk electrode of RDE is 3 mm. Diameter of Disk electrode of RRDE is 4 mm. The inner and outer diameter of Ring electrode of RRDE is 5 mm and 7 mm.** Electrodes of other materials and sizes will be made on a custom request.

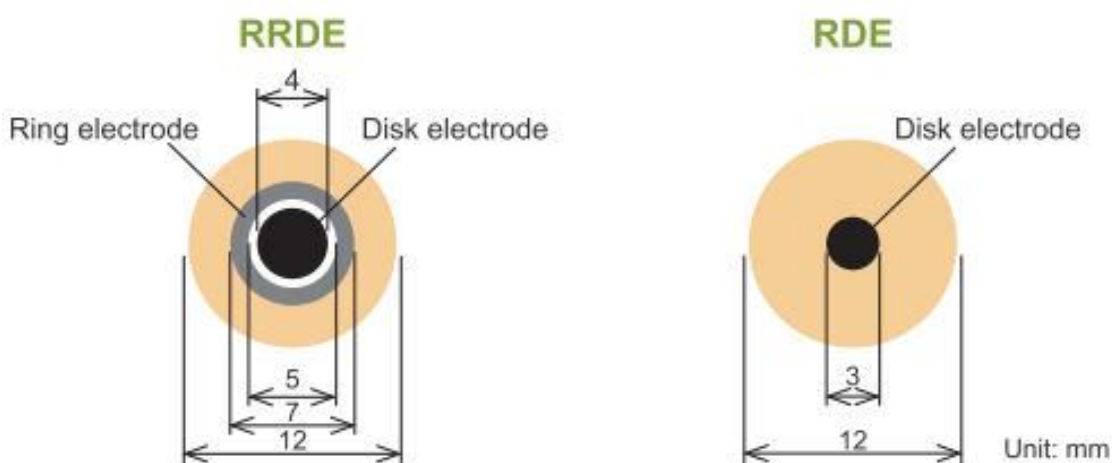


Fig A-1-2

Cat. #	Description
Ring/Disk Electrode	
012614	RRDE Pt Ring/Pt Disk Electrode
012613	RRDE Pt Ring/GC Disk Electrode
012616	RRDE Au Ring/GC Disk Electrode
012615	RRDE Pt Ring/Au Disk Electrode
Disk Electrode	
011169	RDE GCE Glassy carbon Disk Electrode
013490	RDE GCET Glassy carbon disk electrode
013482	RDE GCE Glassy carbon disk electrode ID:5mm
013491	RDE GCET Glassy carbon disk electrode ID:5mm
011170	RDE PTE Platinum Disk Electrode
011171	RDE AUE Gold Disk Electrode
011966	RDE ALE Aluminum Disk Electrode
011967	RDE AGE Silver Disk Electrode
011968	RDE CUE Copper Disk Electrode
011969	RDE NIE Nickel Disk Electrode
011970	RDE TAE Tantalum Disk Electrode
011971	RDE TIE Titanium Disk Electrode
011972	RDE WE Tungsten Disk Electrode
011973	RDE CPE Carbon paste Disk Electrode

Table A-1-1

A-1-2 DRE – Disk Replaceable Electrode

The Disk Replaceable Electrode (DRE) has the Disk electrode removable. The replacement of the disk electrode is possible from both sides: front side and rear side. It makes possible to choose, according to the condition required for your research purpose.

Features:

1. Assessment of the disk electrode using the same ring electrode, could avoid the influence of the ring material and dimension.
2. Removable disk and ring assembly make possible modification of the electrode surface and polishing process, separately.
3. Disposable disk could be used.



Fig A-1-2-1

Full detailed manual:

http://usr.bas.jp/dl_sub/?id=518c47a52982c

Cat.#	Description
013336	DRE-PGK Pt ring/GC disk replaceable electrode kit
Component	
013337	DRE-PTR Pt ring assembly
013338	DRE-GCD GC disk
013339	DRE-SPC Teflon spacer (3pcs)

Optional items		For the DRE- PGK Pt
013366	DRE-AUD Au disk	
013367	DRE-PTD Pt disk	

ring/GC disk replaceable electrode kit:

Table A-1-2-1

For the DRE-GCK disk replaceable electrode kit:

Cat.#	Description
013362	DRE-GCK GC disk replaceable electrode kit
013364	DRE-AUK Au disk replaceable electrode kit
013365	DRE-PTK Pt disk replaceable electrode kit
Components common for the kits	
013361	DRE-DAS Disk assembly
013339	DRE-SPC Teflon spacer (3pcs)
Optional items	
013338	DRE-GCD GC disk (included in 013362)
013366	DRE-AUD Au disk (included in 013364)
013367	DRE-PTD Pt disk (included in 013365)

Table A-1-2-2

For the DRE-DCP Disk electrode polishing and exchanging tool kit:

Cat.#	Description
013340	DRE-DCP Disk electrode polishing and exchanging tool kit
Component	
-	DRE-BLK Base block

-	DRE-STP Stopper
-	DRE-DRS Disk remove tool
-	DRE-SPS Spacer push tool
-	DRE-DPS Disk push tool
-	DRE-EPH Electrode polishing holder

Table A-1-2-3

A-2 Reference Electrodes

Reference electrodes are widely used for electrochemical measurements (CV, LSV, DPV etc.) and electrochemical devices (electrochemical detectors for HPLC, electrochemical biosensor etc.). Various kinds of reference electrodes such as aqueous, non-aqueous, calomel types are available.



Fig A-2-1

Cat. #	Description	Length approx. (mm)	OD (mm)	Porous junction	Solution
Ag/AgCl type					
012167	RE-1B Reference electrode (Ag/AgCl)	78	6	IPPG ¹	3 M NaCl
013393	RE-1S Reference electrode (Ag/AgCl)	52	4.5	IPPG ¹	3 M NaCl
013503	RE-1CP Reference electrode (Ag/AgCl/Saturated KCl)	92	6	Ceramics	Saturated KCl
013488	RE-3VT Reference electrode screw type (Ag/AgCl)	52	10	IPPG ¹	3 M NaCl
Calomel type					
013458	RE-2BP Calomel Reference electrode	92	6	Ceramics	Saturated KCl
Hg type					
013459	RE-2CP Reference electrode	92	6	Ceramics	Saturated K ₂ SO ₄
Alkaline type					
013456	RE-61AP Reference electrode for alkaline solution	92	6	Ceramics	Use 1 M NaOH

1: Ion Permeability Porous Glass, 2:ACN: acetonitrile

3:TBAP: tetrabutylammonium perchlorate.

Table A-2-1

A-3 Non Aqueous reference electrode: RE-7

We have developed new Non-Aqueous Reference electrodes RE-7 and RE-7S as successor products of our former Non-Aqueous Reference electrode RE-5 and RE-5S. In order to overcome fear of inner electrolyte's evaporation during their storage or transportation, we separated them into body and inner electrolyte. This revolutionary modification enables users to refill the solution and to use it repeatedly.



Fig A-3-1

We are firmly sure these cost-effective products satisfy you.

RE-7:

Cat. #	Description
012171	RE-7 Non Aqueous reference electrode (Ag/Ag ⁺)
Contents	
012057	RE-7 Teflon cap with Ag wire
012549	RE-7 Electrolyte solution (10mL)
(012176)	Sample holder dia 6mm (2pcs)

Table A-3-1

RE-7S:

Cat. #	Description
013394	RE-7S Non Aqueous reference electrode (Ag/Ag ⁺)
Contents	
012058	RE-7S Teflon cap with Ag wire
012549	RE-7 Electrolyte solution (10mL)
-	Sample holder dia 4.5mm

Table A-3-2

A-4 RHEK – Reversible Hydrogen Electrode Kit

Reversible Hydrogen Electrode (RHE) is a reversible electrode which the hydrogen gas is generating by electrolysis of strong acid and trapping, while it is used. It makes unnecessary the complicated and dangerous set-up of the hydrogen cylinder and avoids the large volume of hydrogen gas exhaustion, even though it has a good potential stability.

The RHE has a large application field, it can be used in a strong acid solution (<pH 2), such as 0.5 mol/L sulfuric acid, also in a neutral solution, when it is applied with a double junction chamber.

Full detailed manual:

http://usr.bas.jp/dl_sub/?id=529d7ecacf927

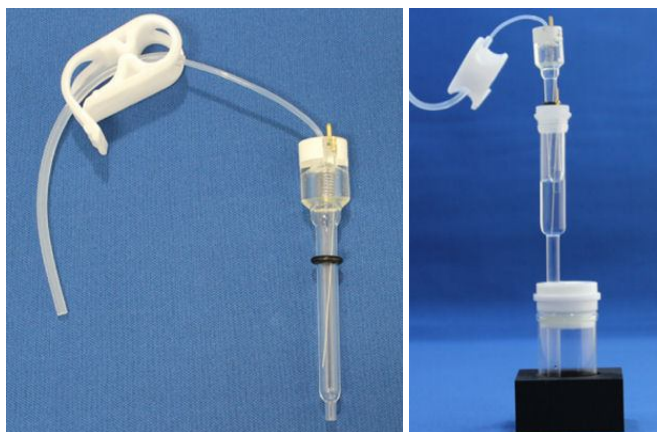
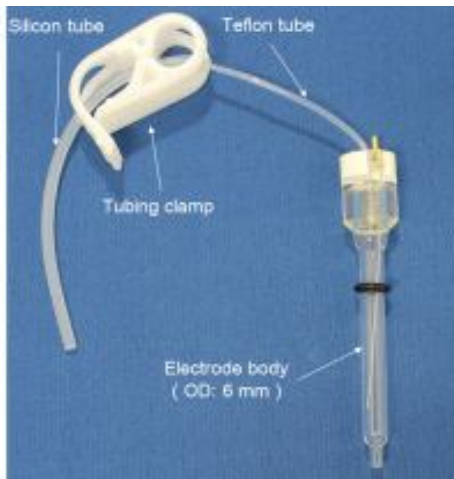


Fig A-4-1 For application in an acid solution (left), and RHE with a double junction chamber, for application in a neutral solution (right).

Reversible hydrogen electrode kit

The RHEK Reversible hydrogen electrode kit can be used without Double junction chamber when the strong acid (<pH 2, such as 0.5 mol/L sulfuric acid) is used as an electrolyte solution.

Cat.#	Description
013373	RHEK Reversible hydrogen electrode kit
Content	
013374	RHE Reversible hydrogen electrode
-	Tubing clamp
-	Silicone tube
-	O-ring
-	Female lure fitting
-	Disposable syringe

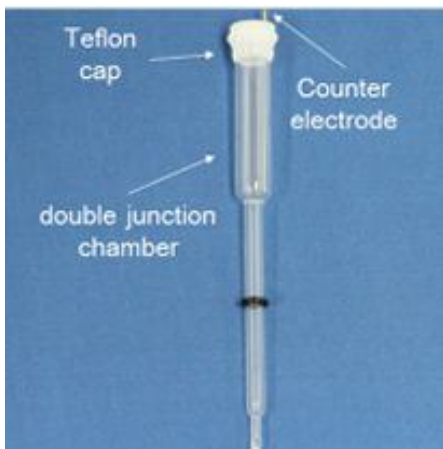


The five items, without the catalog number, are available as a kit.
013378 RHEK accessories kit

Double junction chamber kit

The Double junction chamber kit is used when the electrolyte solution is a weak acid, neutral solution, solution containing scum etc.

Cat.#	Description
013375	Double junction chamber kit
Content	
013376	Double junction chamber
-	O-ring
013377	PTFE Cap for double junction chamber
002222	Platinum counter electrode 5.7cm



A-5 Counter electrodes

Please select suitable counter electrodes according to your application.

Custom made counter electrode is also available.
Please do not hesitate to contact us.



Fig A-5-1

Cat. #	Description
002222	Platinum counter electrode 5.7 cm
002233	Platinum counter electrode 5 cm
012961	Platinum counter electrode 23 cm
012962	Gold counter electrode 23 cm
012963	Nickel counter electrode 23 cm

Table A-5-1

A-6 PK-3 Electrode Polishing kit

PK-3 is a polishing kit to refresh the working electrode surface for CV/Flow cell. Contents of PK-3 can be provided separately. In that case, each polishing pad (012600 Alumina polishing pad/012601 Diamond polishing pad) includes 20 pieces of them in per sales unit.

Cat. #	Description
013223	PK-3 Electrode polishing kit
Components	
012620	0.05 μm polishing alumina (20 mL)
012621	1 μm polishing diamond (10 mL)
-	Alumina polishing pad (10 pcs)
-	Diamond polishing pad (10 pcs)
013222	Replacement glass plate for PK-3 (1 pc)

Table A-6-1

How to use:

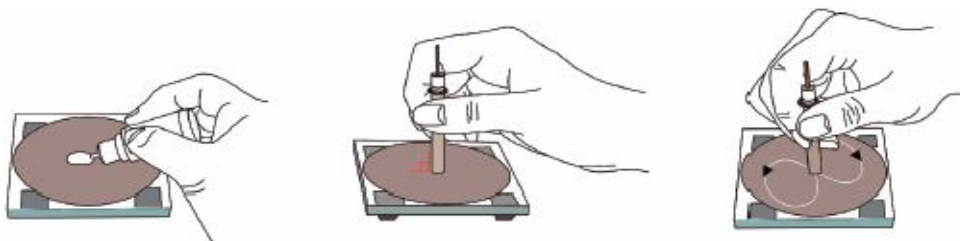


Fig A-6-1

1. Prepare the glass plate, put drops of polishing diamond on diamond polishing pad.
2. Hold the CV electrode at right angle to the pad, polish in a circular motion.
3. Replace by an alumina polishing pad, put drops of polishing alumina and polish to be mirror surface. Rinse the electrode surface with distilled water, dry in air before use.

Attention: Polishing with PK-3 is the most appropriate way to clean up electrode surface. Please consult us when other methods are applied.

A-7 Connecting other potentiostat

A-7-1 CHInstrument

Remote connection

The RRDE-3A **rotator speed** and **purge** can be controlled by **CHI** Electrochemical Analyzer.

- Rotation speed control:
Link the CHI and RRDE-3A devices with a banana jack to wire cable (**Fig A-7-1-1**). Contact CHI to purchase the cable.

ALS/CHI	RRDE-3A
RDE	IN
GND	GND (next to IN)



Figure A-7-1-1 Rotation remote control cable

- Purge control:
Link the CHI and RRDE-3A devices with an optional cable (**Fig A-7-1-2**). Contact CHI to purchase the cable.

ALS/CHI		RRDE-3A
Cell Control	White	Purge
	Black	GND (next to Purge)

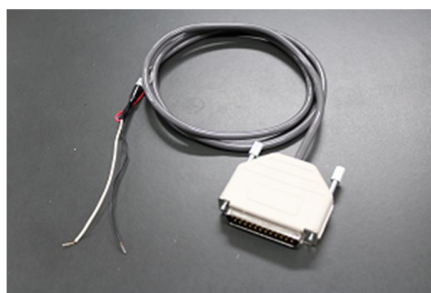


Figure A-7-1-2 Purge remote control cable

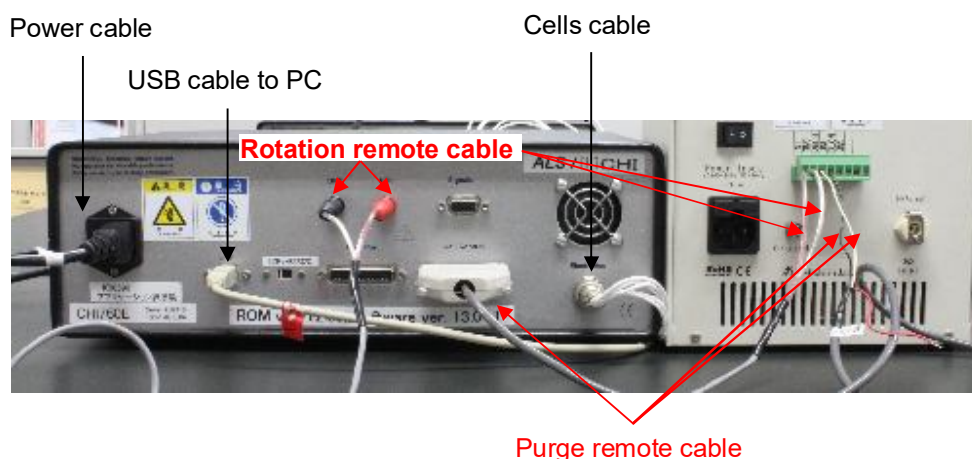


Figure A-7-1-3 Remote connection between CHI and RRDE-3A

Cell Connections

The **reference electrode** and **counter electrode** lead wires extend from the potentiostats. The connectors are press-on type. Simply push the connector over the corresponding pin in the electrode to make the connection (**Figure A-7-1-4**). Alligator clips are color coded to the electrode it attaches.

The **ring electrode** and the **disk electrode** connector are located under the motor shaft assembly.

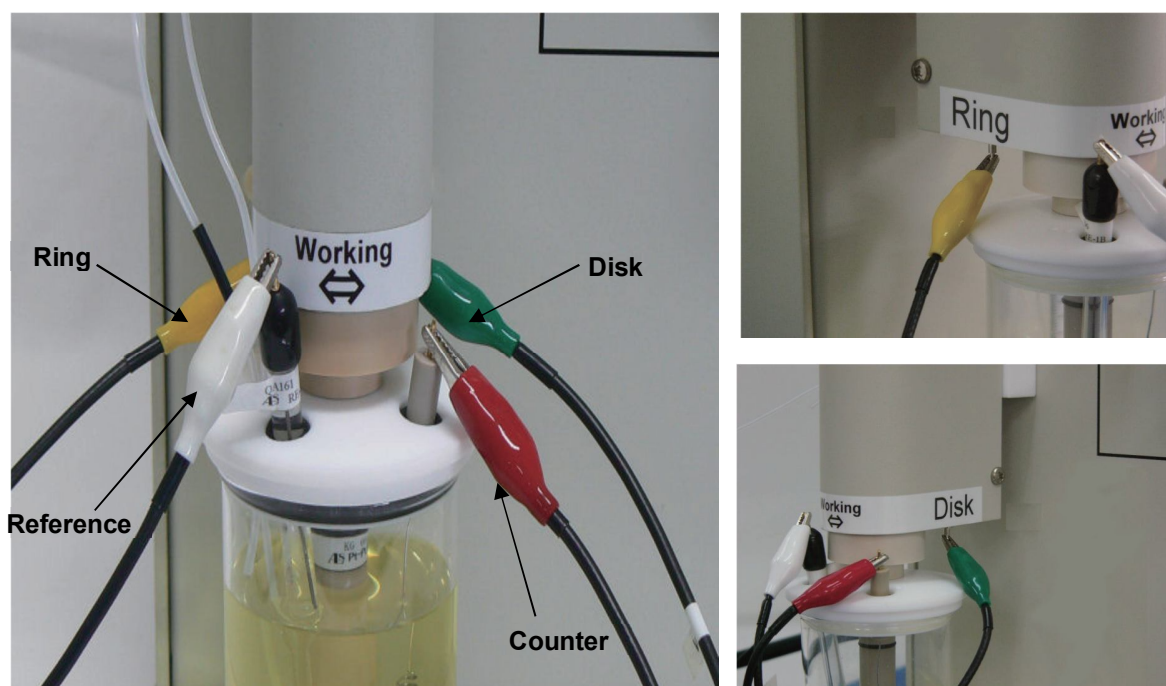


Figure A-7-1-4. Electrode lead connections

Wire color	Electrode
Green	Disk
White	Reference
Red	Counter
Yellow	Ring
Black	Not used

A-7-2 BioLogic Potentiostat

In case of coupling RRDE-3A with any Bio-Logic potentiostats:

BioLogic Instrument	RRDE-3A input
Pin 2 (analog Out)	IN
Pin 7 (Ground)	GND (next to IN)
Pin 5 (trigger Out)	PURGE

A dedicated cable with the appropriate wiring is available. It is the **PN: 092-22/11**. It has a DB9 connector on one end and three wires named Analog Out, Trigger Out and 1 Ground on the other end

One volt corresponds to 1,000 rpm. It is possible to set from 90 to 8,000 rpm from 0 to 8 DC Volt.

The purge and pinch valve in the RRDE-3A is controlled by TTL level active low.

The appropriate settings are already available in **EC-Lab® software**.

Controlling RRDE-3A through EC-Lab® software:

In the “**External device**” tab, select “**RDE**” for “**Device Type**” and then “**ALS RRDE-3A**” for “**Device Name**”.

Figure A-7-2-1. External device tab screen shot

Cell Connections

- For RDE case (one potentiostat is used), the **red leads** have to be stacked together and then connected to the **disk** plug. The **white leads** and **blue leads** have to be connected to the **reference** and the **counter** electrodes, respectively.

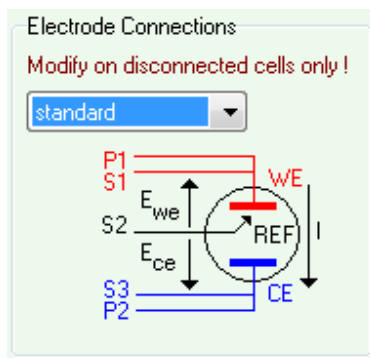


Figure A-7-2-2. RDE connection

- For RRDE studies, the user has to first select the “**CE to ground**” mode for “**Electrode Connections**” (in the “**Advanced Settings**” experiment). This connection mode enables the multi-electrode measurements. According to the instrument type, the connection of the instrument to the cell is different:

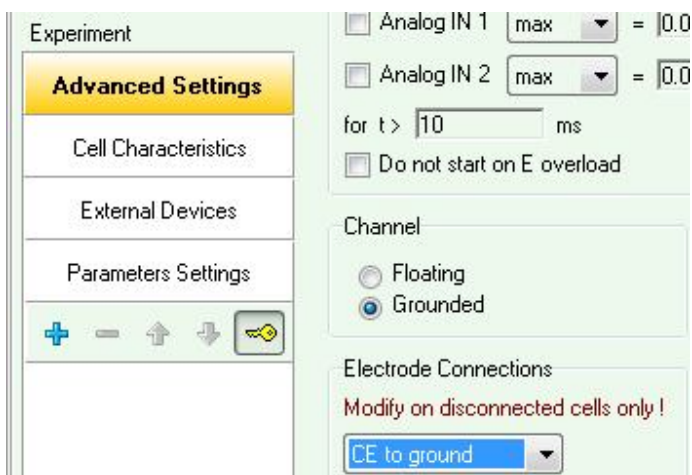


Figure A-7-2-3. RRDE connection

- For the **VMP3** based instrument, the **blue leads (CA1 and Ref3)** of **one channel** has to be connected to the **disk** and the two **blue leads (CA1 and Ref3)** of the **other channel** have to be connected to the ring. Then the **two white leads (Ref2)** have to be connected to the **reference** electrode and the two **Ref1** leads and the **ground** leads have to be connected to the **counter-electrode**.

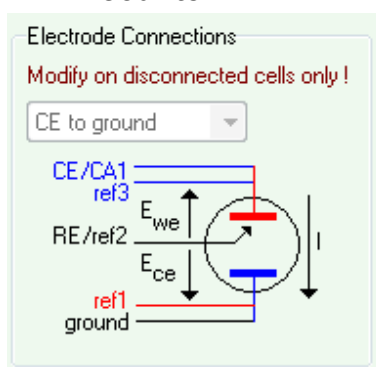


Figure A-7-2-4. RRDE for VMP3

- For the **SP-300** based instruments, the **red leads (P1 and S1)** of **one channel** has to be connected to the **disk** and the **two red leads (P1 and S1)** of the **other channel** has to be connected to the **ring**. Then **the two white leads (S2)** have to be connected to the **reference** electrode and the **two S3 blue leads** and the **ground** leads have to be connected to the **counter-electrode**.

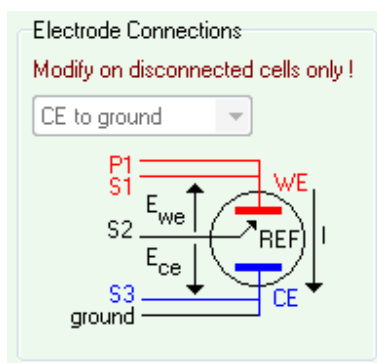


Figure A-7-2-5. RRDE for SP-300

A-7-3 Gamry R600

Gamry potentiostats can be used in BiPotentiostat configuration for the purpose of rotating Ring-Disk electrode experiments. Special cables and scripts are required to synchronize the data acquisition of the potentiostats. In this configuration one potentiostat is the “**Master**” and the other is the “**Serf**”. The **Master** will connect to the **Disk** electrode and the **Serf** will connect to the **Ring** electrode.

Editing scripts

For RRDE-3A, 1V corresponds to 1,000 rpm, but the R600 is set up with the ratio 0.5V for 1,000 rpm. Therefore we have to edit the R600 script from the R600 framework software.

To do that, follow this procedure:

#1- Open the file “classElectrode.exp”.

Location:

Win XP:

C:\Document and Settings\All Users\Application Data\Gamry Instruments\Framework 5\Scripts\

Win 7:

C:\ProgramData\GamryInstruments\Framework\Scripts\

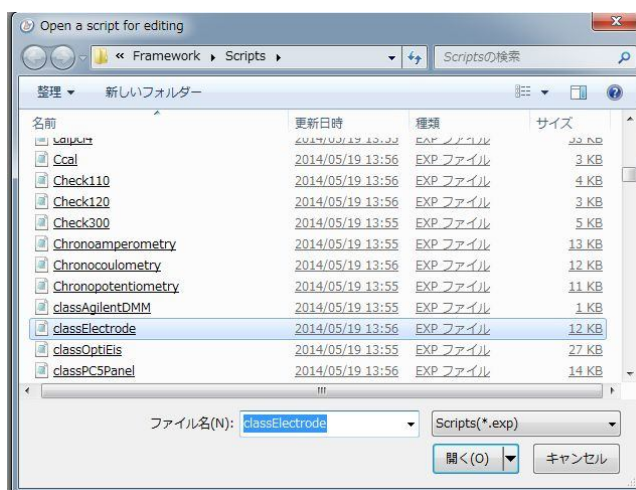
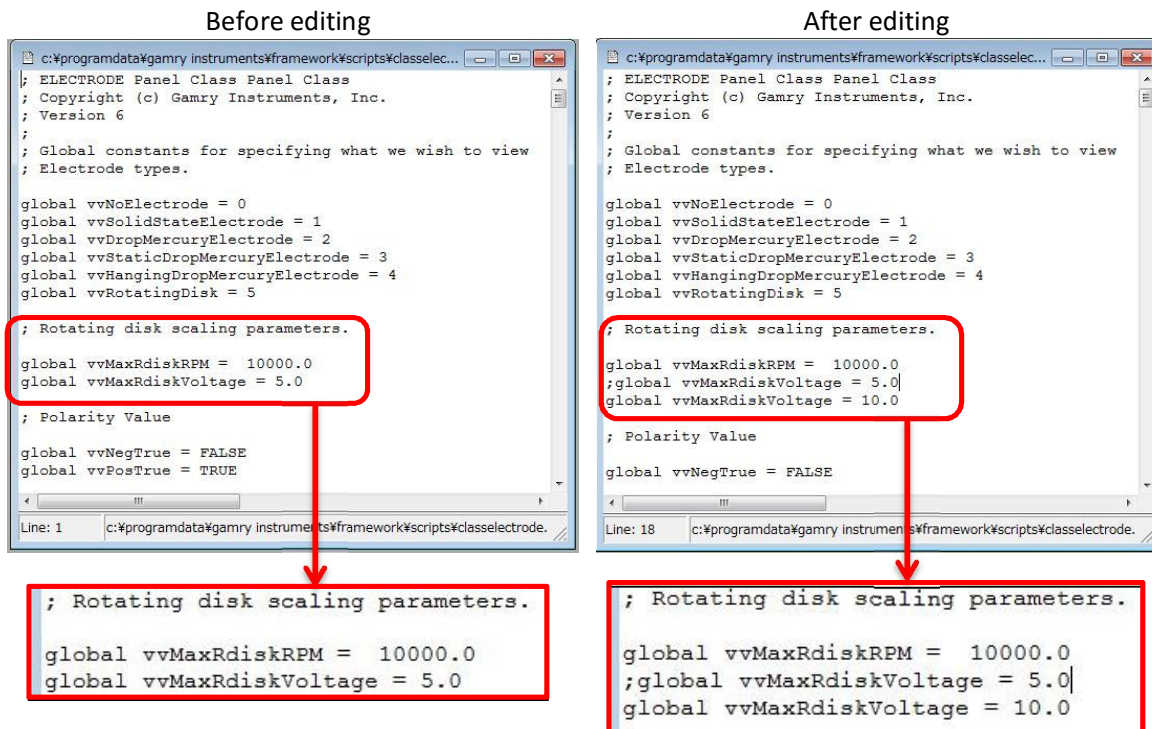


Figure A-7-3-1. Win 7 screen shot

#2. Rewrite the script as below

Place a semicolon “;” at the beginning of the line:
global vvMaxRdiskVoltage= 5.0.

And add this line:
global vvMaxRdiskVoltage= 10.0

Connecting the Potentiostats

In order to synchronize the Disk scan with the Ring data acquisition, an interconnection is required between the two potentiostats.

To do this, just link **pin 6** each other from the I/O port to link each potentiostat sync ground (green cable on **Fig. A-7-3-2**). Then, link the **Master's pin 4** to the **Serf's pin 3** (black cable on **Fig. A-7-3-2**).

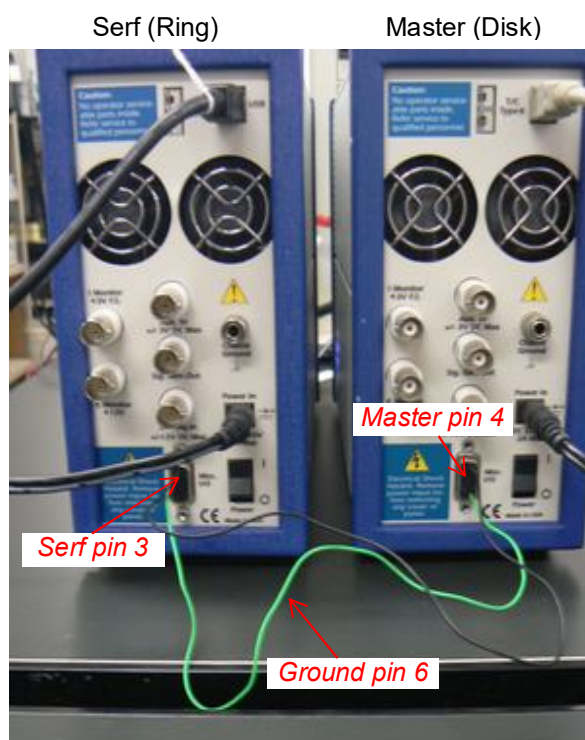


Fig. A-7-3-2

Cell Connections

Two standard Gamry Cell Cables are be used to connect to the Cell to the Gamry Potentiostats. There are many wires! Be certain that unwanted interconnections are not made accidentally!

The single **Reference** Electrode should be connected to **BOTH** of the **White**, Reference Electrode pin jack sockets. The single **Counter** Electrode should be connected to **BOTH** of the **Red**, Counter Electrode banana plugs. Banana-to-Alligator or Pin Jack-to-Alligator adapters may be useful here.

The **Blue (Working Sense)** and **Green (Working)** leads of the **Master** potentiostat should be connected to the **disk** electrode of the RRDE-3A.

The **Blue (Working Sense)** and **Green (Working)** leads of the **Serf** potentiostat should be connected to the **ring** electrode of the RRDE-3A.

The “Counter Sense” (Orange) leads of both Gamry Potentiostats should be left disconnected. They are not used.

The Ground wire (“Floating” ground) or the Ground “jack” of the Disk Potentiostat may be connected together to provide better noise rejection. The Ground wire and Grounding jack of the Ring Potentiostat must both remain unconnected to any other ground or cell lead.

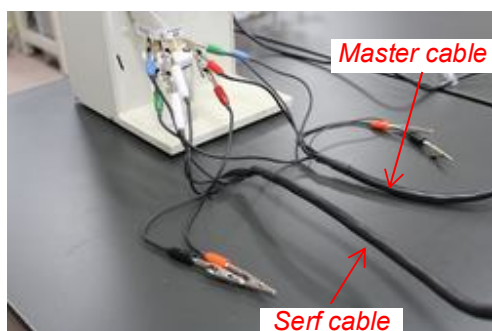


Fig. A-7-3-3



Fig. A-7-3-4

Remote connection

Gamry R600 can remotely control the **RRDE-3A** rotation.

Just link the I/O port **pin 1** to the **IN** of the RRDE-3A remote terminal port and the I/O port **pin 2** to the **GND** of the RRDE-3A remote terminal port.

A-7-4 Ivium potentiostat

Most Ivium potentiostats are equipped with a peripheral port for connection to, and communication with, external equipment. For example the **CompactStat** and **IviumStat** are equipped with a DB37 connector (**Fig. A-7-4-1**).



Fig. A-7-4-1. DB37 connector

To connect the peripheral port to the RRDE-3A:

Ivium peripheral port*		RRDE-3A input
DB37 pin#	Signal	
7	An-out1	IN
25	AGND	GND (next to IN)
21	Dig out1	PURGE
24	DGND	GND
*To verify the exact configuration and specifications of the peripheral port of your Ivium potentiostat, refer to the Ivium user manual.		

Controlling RRDE-3A through IviumSoft:

The rotator RPM and purge controls are integrated in the Ivium potentiostat control software 'IviumSoft'. In IviumSoft activate the advanced method options and check AUX-parameter:

- Purging can be controlled by entering a purging time (s); When time > 0s is selected, a TTL Level active low is given for that duration on Digital output1, prior to the start of the experiment.
- The RPM can be controlled from 0 to 100% which corresponds to 0% and 100% of the maximum voltage output capability of the Analog output1 port. Note that this voltage may be 0-4V max. or 0-10V max., depending on the type of your instrument. In case your peripheral port has a 4V max. output, a PLT module can be supplied by Ivium to increase this voltage to 10V max.

For example:

0-4V max. output --> 90 to 4,000 RPM (at 100% setting)

0-10V max. output --> 90 to 8,000 RPM (at 80% setting)

Cell Connections

Ivium potentiostats are delivered with a standard cell cable that has 6 leads (**Fig. A-7-4-2**).



Fig. A-7-4-2. Ivium standard cell cable

To connect to the RRDE-3A cell stand use the delivered alligator clips on the cell cable and connect as follows:

Ivium potentiostat cell cable			Connect to Electrode of RRDE-3A
Banana Color	Lead code	Connection	
BLACK	C	counter electrode	COUNTER
RED (big)	W	working electrode	DISK
RED (small)	W2	second working* electrode	RING
BLUE	R	reference electrode	REFERENCE
WHITE	S	sense electrode	DISK**
GREEN	T	ground	-

*The second working electrode, or Bipotentiostat, is available on most Ivium potentiostats as an optional extra.

**The sense electrode acts as a reference to the counter electrode and can be connected directly to the (primary) working electrode.



DECLARATION OF CONFORMITY

BAS INC

No. 1-36-6, Oshiage, Sumida-ku, Tokyo, 131-0045, Japan

Declare under our sole responsibility that the product;

Product : Rotating Ring Disk Electrode Apparatus
Model : RRDE-3A

To which this declaration relates is in conformity with the following standards or other normative documents;

EN61326: 1997 + A1: 1998 + A2: 2001 + A3: 2003 (Class A)

EN61000-3-2: 2000

EN61000-3-3: 1995 + A1: 2001

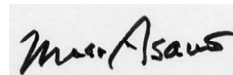
following the provisions of Directives;

2004/108/EC Electromagnetic Compatibility Directive
and its amending directives: 92/31/EEC, 93/68/EEC

Tokyo, Japan
(Place)

Jan 27, 2009
(Date of issues)

Masao Asano
name


signature