

WaveVortex Electrode Rotator User Guide



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1. Preface

1.1 Scope

The WaveVortex is a closed-loop servo-system (utilizing pulse-width modulation) designed to rotate an electrode in an electrochemical cell. This manual describes the proper use of the WaveVortex and covers routine operating procedures, periodic maintenance and calibration, and safety issues.

This user guide assumes that the reader also has some basic knowledge of electronics, electrochemistry, modern voltammetry, and safe practices for working in a chemical laboratory. While some background information is presented in this user guide, the reader is referred to the appropriate scientific literature for more detail regarding the theory and practice of hydrodynamic electrochemistry.

Pine Research Instrumentation provides online support for the rotator at the following URL:

www.pineresearch.com/shop/rotators/standard/wavevortex

1.2 Copyright

This publication may not be reproduced or transmitted in any form, electronic or mechanical, including photocopying, recording, storing in an information retrieval system, or translating, in whole or in part, without the prior consent of Pine Research Instrumentation, Inc. in writing.

1.3 Trademarks

WaveVortex® and AfterMath® are registered trademarks of Pine Research Instrumentation, Inc. (Durham, NC). Viton® is a registered trademark of The Chemours Company. All other trademarks are the property of their respective owners.

1.4 Use Limitations

The WaveVortex rotator system is not designed for use in experiments involving human subjects and/or the use of electrodes inside of the human body or on the surface of the human body.

If the WaveVortex rotator system is used in a manner not specified by the manufacturer in this user guide, then the protection provided by the system may be impaired.

Any use of this instrument for other than its intended purpose is prohibited.



1.5 Harmful or Corrosive Substances

The operator of the WaveVortex rotator system should have prior experience working in a chemical laboratory and knowledge of the safety issues associated with working in chemical laboratory. Electrochemical experiments may involve the use of harmful or corrosive substances, and the operator should wear personal protective equipment while working with these substances. At a minimum, the operator should wear the following items to avoid contact with harmful or corrosive substances:

- Eye protection (safety goggles, face shield, etc.)
- Laboratory coat (flame resistant and solvent resistant)
- Solvent-resistant gloves
- Closed-toe shoes

Additional personal protective clothing and equipment may be required depending upon the nature of the chemicals used in an experiment. A complete discussion of chemical laboratory safety practices is beyond the scope of this user guide, and the reader is directed to the CHEMICAL SAFETY BIBLIOGRAPHY below for additional information.

CHEMICAL SAFETY BIBLIOGRAPHY BIBLIOGRAPHIE DE SÉCURITÉ CHIMIQUE

1. American Chemical Society Committee on Chemical Safety Hazards Identification and Evaluation Task Force, *Identifying and Evaluating Hazards in Research Laboratories: Guidelines Developed by the Hazards Identification and Evaluation Task Force of the ACS Committee on Chemical Safety*; American Chemical Society, 2013.
2. National Research Council (US), Division of Earth and Life Studies, Board of Chemical Sciences and Technology, Committee on Prudent Practices in the Laboratory, *Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards, Updated Version*; National Academies Press, 2011.
3. American Chemical Society Committee on Chemical Safety. *Safety in Academic Chemistry Laboratories*; 7th ed.; American Chemical Society: State College, PA, 2003; Vol. 2.

L'opérateur du WaveVortex doit avoir une expérience préalable de travail dans un laboratoire de chimie et la connaissance des mesures de sécurité associées aux travaux dans un laboratoire de chimie. Les expériences en électrochimie peuvent impliquer l'utilisation de substances nocives ou corrosives, et l'opérateur doit porter des équipements de protection individuelle lorsqu'il travaille avec ces substances. Au minimum, l'opérateur doit porter les articles suivants pour éviter le contact avec les substances nocives ou corrosives :

- Protection des yeux (lunettes de sécurité, masque de protection facial, ect.)
- Blouse de laboratoire (résistante au feu et résistante aux solvants)
- Gants de protection résistants aux solvants
- Chaussures fermées

Des vêtements et équipements de protection individuelle supplémentaires peuvent être requis en fonction de la nature des produits chimiques utilisés dans une expérience. Une discussion complète des pratiques de sécurité de laboratoire chimique est au-delà de la portée de ce guide de l'utilisateur, et le lecteur est dirigé vers la « BIBLIOGRAPHIE DE SÉCURITÉ CHIMIQUE » ci-dessus pour des informations supplémentaires.

1.6 Service and Warranty Information

For questions about proper operation of the WaveVortex or other technical issues, please use the contact information below to contact Pine Research directly.

TECHNICAL SERVICE CONTACT

Pine Research Instrumentation, Inc.
<http://www.pineresearch.com>
 Phone: +1 (919) 782-8320
 Fax: +1 (919) 782-8323

If the WaveVortex or one of its components or accessories must be returned to the production facility for service, please contact Technical Service (see above) to obtain a Return Material Authorization (RMA) form. Include a copy of this RMA form in each carton and ship to the Factory Return Service Address (below).

FACTORY RETURN SERVICE ADDRESS

Pine Instrument Company
 ATTN: RMA # <RMA number>
 104 Industrial Drive
 Grove City, PA 16127
 USA



RETURN MATERIAL AUTHORIZATION REQUIRED!

Do not ship equipment to the production facility without first obtaining a Return Material Authorization (RMA) from Pine Research Instrumentation.

LIMITED WARRANTY

The WaveVortex Electrode Rotator instrument (hereafter referred to as the "INSTRUMENT") offered by Pine Research Instrumentation (hereafter referred to as "PINE") is warranted to be free from defects in material and workmanship for a one (1) year period from the date of shipment to the original purchaser (hereafter referred to as the "CUSTOMER") and used under normal conditions. The obligation under this warranty is limited to replacing or repairing parts which shall upon examination by PINE personnel disclose to PINE's satisfaction to have been defective. The CUSTOMER may be obligated to assist PINE personnel in servicing the INSTRUMENT. PINE will provide telephone support to guide the CUSTOMER to diagnose and effect any needed repairs. In the event that telephone support is unsuccessful in resolving the defect, PINE may recommend that the INSTRUMENT be returned to PINE for repair. This warranty being expressly in lieu of all other warranties, expressed or implied and all other liabilities. All specifications are subject to change without notice.

The CUSTOMER is responsible for charges associated with non-warranted repairs. This obligation includes but is not limited to travel expenses, labor, parts and freight charges.



1.7 Instrument Markings

Labels located on the back panel of each individual WaveVortex rotator include information about the make, model, and serial number of the instrument. These labels also indicate any certifications or independent testing agency marks which pertain to the particular unit (see: Figure 2-5).

1.7.1 Model Number

Model numbers for the Pine Research WaveVortex have the format AF01WVQRST, where Q, R, S, and T are each single characters used to indicate the particular configuration of the rotator. Part numbers for electrodes and other accessories compatible with the WaveVortex rotator are described in more detail later (see: Sections 5 and 6).

Model Number:	A	F	0	1	W	V	Q	R	S	T	Model Name
	A	F	0	1	W	V	1	0			WaveVortex 10

1.7.2 Serial Number

A serial number uniquely identifies each individual WaveVortex rotator. The serial number is encoded as a seven-digit number with format "wwyy1nn" where **wwyy** indicates the week and year of manufacture, and **nn** is a unique number within the indicated week of manufacture.

1.7.3 Certifications and Listings

A particular WaveVortex rotator may bear labels on the back panel which indicate various certifications or independent testing agency marks which pertain to the particular unit. These marks are shown in the table below.

	<p>CE MARK:</p> <p>The CE mark indicates that a WaveVortex rotator complies with one or more European Union directives. These directives are further described in the "CE Declaration of Conformity" attached to the end of this user guide.</p>
	<p>ETL MARK:</p> <p>The ETL mark indicates that a WaveVortex rotator is listed by Intertek to UL 61010-1 (issued 11-MAY-2012; Ed. 3), CSA C22.2 #61010-1 (issued 11-MAY-2012; Ed. 3), and IEC 61010-1 (issued 10-JUN-2010; Corrigendum 1: 11-MAY-2011).</p> <p>Intertek is a Nationally Recognized Testing Laboratory (NRTL) recognized by the United States Occupational Safety and Health Administration (OSHA).</p>

1.8 Specifications

ROTATION RATE	
Rate Accuracy	100 to 200 RPM: Accurate to within ± 2 counts of display reading 200 to 8000 RPM: Accurate to within $\pm 1\%$ of display reading
Rate Display	Four-digit display indicates rotation rate (RPM)
Rate Control (front panel)	10-turn rotation rate control knob
Start/Stop (front panel)	Push-button toggle with LED indicators for "pause" and "run"
Rate Control (external)	Optional rate control via input signal on external I/O port Available control ratios: 1, 2, or 4 RPM/mV, jumper selectable
Start/Stop (external)	Optional digital motor stop input signal on external I/O port Available TTL logic: active high or active low, jumper selectable Front panel LED indicates when external motor stop is active
Enclosure Interlock	Interlock prevents rotation when enclosure window is in raised position. Front panel LED indicates enclosure interlock state.
Rate Output	Optional rate monitoring via output signal on external I/O port Output signal ratio: 2 RPM/mV
MOTOR	
Motor Power	11 W
Control Method	Closed loop servo-system (PWM) Temperature-compensated tachometer mounted on motor shaft
Motor Type	Permanent magnet
Maximum Continuous Torque	18.7 milliNewton-meters
Motor Protection	Motor current is electronically limited
ELECTRICAL	
Power Requirements	AC Mains: 100 – 240 VAC, $\pm 10\%$; 50/60 Hz; 2 A DC Supply: 24 VDC, 1.5 A
Electrode Connections	Disk electrode: red banana jack on motor unit Ring electrode: blue banana jack on motor unit
Chassis Terminal	Metal banana jack on motor unit Second connection provided on external I/O port
DC Common (analog ground)	Connection provided on external I/O port

(specifications table is continued on the next page)

PHYSICAL	
Shipping Information	Shipping weight: 24 pounds (11 kg) Shipping dimensions: 16.0 × 16.0 × 16.0 in (41 × 41 × 41 cm)
Instrument Weight	16 pounds (7.3 kg)
Instrument Dimensions	Width: 15 in (38 cm) Depth: 11 in (28 cm) Height with enclosure window in lower position: 15 in (38 cm) Clearance required to raise enclosure window: 24 in (61 cm)
Materials	Enclosure base and sides: black high-density polyethylene Enclosure window: clear polycarbonate Electronics cabinet: black powder-coated aluminum
Operating Temperature	10 to 40 C (50°F to 104°F)

1.9 Icons (Icônes)

Special icons are used to call attention to safety warnings and other useful information found in this document (see: Table 1-1, Table 1-2, and Table 1-3).

Des icônes spéciales (voir: Tableau 1-1, Tableau 1-2, et Tableau 1-3) sont utilisées pour attirer l'attention sur des avertissements de sécurité et d'autres renseignements utiles disponibles dans ce document.

	<p>STOP:</p> <p>For a procedure involving user action or activity, this icon indicates a point in the procedure where the user must stop the procedure.</p> <p>ARRÊT:</p> <p><i>Dans une opération impliquant l'action ou l'activité d'un utilisateur, cette icône indique l'étape où l'utilisateur doit arrêter l'opération.</i></p>
	<p>NOTE:</p> <p>Important or supplemental information.</p> <p>REMARQUE:</p> <p><i>Renseignements importants ou complémentaires.</i></p>
	<p>TIP:</p> <p>Useful hint or advice.</p> <p>CONSEIL:</p> <p><i>Astuce ou conseil utile.</i></p>

Table 1-1. Special Icons used in this Document.

(Tableau 1-1. Icônes spéciales utilisées dans ce document)

	<p>WARNING: Indicates information needed to prevent injury or death to a person or to prevent damage to equipment.</p> <p>AVERTISSEMENT: Indique les informations nécessaires pour prévenir les blessures ou le décès d'une personne ou pour éviter d'endommager l'équipement.</p>
	<p>ROTATING SHAFT HAZARD: Indicates information needed to prevent injury or death to a person due to a high speed rotating shaft.</p> <p>DANGER LIÉ À LA ROTATION DE L'ARBRE: Indique les informations nécessaires pour prévenir les blessures ou le décès d'une personne à cause de la vitesse élevée de rotation de l'arbre.</p>
	<p>RISK OF ELECTRICAL SHOCK: Indicates information needed to prevent injury or death to a person due to electrical shock.</p> <p>RISQUE DE DÉCHARGE ÉLECTRIQUE: Indique les informations nécessaires pour prévenir les blessures ou le décès d'une personne à cause d'une décharge électrique.</p>
	<p>RISK FROM LASER LIGHT: Indicates information needed to prevent eye injury due to laser beam light.</p> <p>RISQUE LIÉ À LA LUMIÈRE LASER: Indique les informations nécessaires à la prévention des dommages oculaires à cause de la lumière d'un faisceau laser.</p>

Table 1-2. Safety Warning Icons used in this Document.

(Tableau 1-2. Icônes d'avertissement de sécurité utilisées dans ce document)

	<p>CAUTION: Indicates information needed to prevent damage to equipment.</p> <p>ATTENTION: Indique les informations nécessaires pour éviter d'endommager l'équipement.</p>
	<p>RISK OF ELECTROSTATIC DAMAGE: Indicates information needed to prevent damage to equipment due to electrostatic discharge.</p> <p>RISQUE DE DOMMAGES ÉLECTROSTATIQUES: Indique les informations nécessaires pour éviter d'endommager l'équipement à cause d'une décharge électrostatique.</p>
	<p>CHEMICAL INCOMPATIBILITY: Indicates chemical incompatibility information to be considered to prevent damage to equipment.</p> <p>INCOMPATIBILITÉ CHIMIQUE: Indique un renseignement relatif à l'incompatibilité chimique à considérer pour prévenir des dommages à l'équipement.</p>
	<p>TEMPERATURE CONSTRAINT: Indicates when an operation or use of equipment is limited to a specified temperature range.</p> <p>CONTRAINTES DE TEMPÉRATURE : Indique lorsqu'une opération ou un usage de matériel est limité à une plage de températures spécifique.</p>

Table 1-3. Other Safety Warning Icons used in this Document

(Tableau 1-3: Autres Icônes d'avertissement de sécurité utilisées dans ce document)

1.10 Safety Labels (Étiquettes de sécurité)

Specific safety warnings are found on labels attached to the rotator (see: Figure 1-1).

Les avertissements de sécurité spécifiques suivants se trouvent sur les étiquettes apposées sur le rotateur (voir: Figure 1-1).

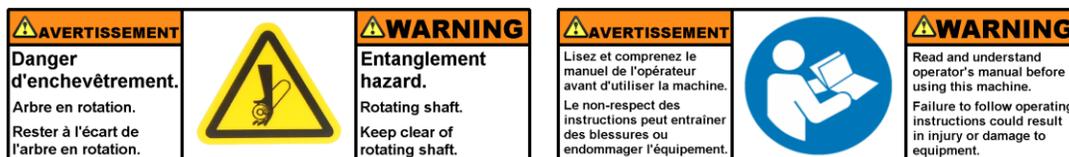


Figure 1-1. Specific Safety Warning Labels on WaveVortex Rotator

1.11 General Safety Warnings (Avertissements de sécurité généraux)

The following safety warnings pertain to general use of the rotator. More specific safety warnings are found in later sections of this document which pertain to particular operations and procedures involving the rotator.

Des avertissements de sécurité plus spécifiques se trouvent dans les sections suivantes de ce document, concernant les opérations et les procédures particulières relatives au rotateur.



WARNING:

Rotating shaft.

The rotator will not operate unless the enclosure window is in the lower position as shown below.

Use extreme caution when operating the rotator at rotation rates above 2000 RPM.

AVERTISSEMENT:

Arbre en rotation.

Le rotateur ne fonctionne que lorsque la fenêtre du boîtier est fermée comme indiqué ci-dessous.

Soyez extrêmement prudent lorsque vous utilisez le rotateur à des vitesses de rotation supérieures à 2000 tr/min.



**WARNING:**

Failure to connect the third prong of the power cord to a proper earth ground may impair the protection provided by the system.

AVERTISSEMENT:

L'absence de connexion de la troisième broche du cordon d'alimentation à une prise de terre appropriée peut altérer la protection fournie par le système.

**WARNING:**

A detachable main power cord is provided with the rotator. Do not replace this cord with an inadequately rated cord.

AVERTISSEMENT:

Un cordon d'alimentation amovible est fourni avec le rotateur. Ne remplacez pas ce cordon par un cordon de calibre inadéquat.

**WARNING:**

A factory-approved power supply is provided with the rotator. Do not use a power supply which is not factory-approved.

AVERTISSEMENT:

Une alimentation électrique approuvée par le constructeur est fournie avec le rotateur. N'utilisez pas une alimentation électrique non approuvée par le constructeur.

**WARNING:**

Risk of electric shock.

Disconnect all power before servicing the rotator.

AVERTISSEMENT:

Risque de décharge électrique.

Déconnectez toutes les sources d'alimentation avant de procéder à l'entretien du rotateur.

**CAUTION:**

Do not exceed the maximum rotation rate for an electrode tip.

Each type of rotating electrode tip has a specific maximum rotation rate limitation. Consult the documentation for the specific electrode being used in order to learn the maximum rotation rate for that electrode.

ATTENTION:

Ne dépassez pas la vitesse de rotation maximale pour un embout d'électrode.

Chaque type d'embout d'électrode tournante possède une vitesse de rotation maximale spécifique.

Consultez la documentation pour connaître la vitesse de rotation maximale de chaque embout.

**WARNING:**

Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before installing or removing an electrode tip on the end of the shaft.

AVERTISSEMENT:

Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant d'installer ou d'enlever un embout d'électrode à l'extrémité de l'arbre.

**WARNING:**

Do not turn on the rotator or rotate the electrode shaft if the shaft is not securely mounted in the motor unit. Inspect the shaft to be certain that it is securely mounted.

AVERTISSEMENT:

Ne mettez pas le rotateur en marche ni l'arbre de l'électrode en rotation si l'arbre n'est pas installé d'une façon sécurisée dans l'unité moteur. Inspectez l'arbre pour vous assurer qu'il est bien fixé.

**WARNING:**

Do not use or attempt to rotate an electrode shaft that has been dropped, bent or otherwise physically damaged. Inspect the shaft to be certain that it is not damaged.

AVERTISSEMENT:

N'utilisez pas et ne tentez pas de mettre en rotation un arbre d'électrode qui est tombé, a été tordu ou a été endommagé physiquement d'une manière ou d'une autre. Inspectez l'arbre pour vous assurer qu'il n'a pas été endommagé.

**WARNING:**

Do not use or attempt to rotate an electrode tip that has been dropped, bent or otherwise physically damaged. Inspect the electrode tip to be certain that it is not damaged.

AVERTISSEMENT:

N'utilisez pas et ne tentez pas de mettre en rotation un embout d'électrode qui est tombé, a été tordu ou a été endommagé physiquement d'une manière ou d'une autre. Inspectez l'embout d'électrode pour vous assurer qu'il n'a pas été endommagé.

**WARNING:**

Do not use an electrode shaft which appears to wobble, vibrate, or tilt away from the axis of rotation while rotating. Such a shaft is either improperly installed or physically damaged. Turn off the rotator, disconnect electrical power, and remove the shaft immediately.

AVERTISSEMENT:

N'utilisez pas un arbre d'électrode qui semble osciller, vibrer ou dévier de l'axe de rotation pendant la rotation. Un tel arbre est mal installé ou endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'arbre immédiatement.

**WARNING:**

Do not use an electrode tip which appears to wobble, vibrate, or tilt away from the axis of rotation while rotating. Such an electrode tip is either improperly installed or physically damaged. Turn off the rotator, disconnect electrical power, and remove the electrode tip immediately.

AVERTISSEMENT:

N'utilisez pas un embout d'électrode qui semble osciller, vibrer ou dévier de l'axe de rotation pendant la rotation. Un tel embout est mal installé ou endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'embout d'électrode immédiatement.

**WARNING:**

Laser radiation.

Many optical tachometers use a laser beam as a light source. Do not look directly at the laser beam. Do not point the laser beam into the eye.

AVERTISSEMENT:

Rayonnement laser.

Un grand nombre de tachymètres optiques utilisent un faisceau laser comme source de lumière. Ne regardez pas directement le faisceau laser. Ne pointez pas le faisceau laser dans l'œil.

**CAUTION:**

When raising or lowering the motor unit along the support rod, be sure to hold the motor unit carefully so that it does not unexpectedly fall and break the glass cell located below the motor unit.

ATTENTION:

Lors de l'élévation ou l'abaissement du bloc moteur le long de la barre principale, assurez-vous de bien le maintenir pour éviter qu'il tombe brutalement et casse la cellule en verre située sous le bloc moteur.

1.12 Hazardous Material Disclosures

The information provided in Table 1.5 and Table 1.6 is provided in accordance with Chinese Standard SJ/T 11364, *Marking for the Restricted Use of Hazardous Substances in Electronic and Electrical Products*.

Hazardous Material Disclosure Table						
AF01WV10wxyz						
Part Name	Hazardous Substances					
	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Hexavalent Chromium (Cr (VI))	Polybrominated biphenyls (PBB)	Polybrominated diphenyl ethers (PBDE)
PCB	X	O	O	O	O	O
<p>This table is prepared in accordance with the provisions of SJ/T 11364.</p> <p>O: indicates that said hazardous substance contained in all of the homogeneous materials for this part is below the limit requirements of GB/T 26572.</p> <p>X: indicates that said hazardous substance contained in at least one of the homogeneous materials for this part is above the limit requirements of GB/T 26572.</p> <p>Note: The date of manufacture for this item may be coded in the serial number as follows: wwyy1nn: ww indicates week; yy indicates year; and nn is the number of the item, starting with 01 each week.</p> <p>Note: The unit meets EU RoHS requirements; China does not have the exemptions for hazardous materials in components as does the EU; therefore, there may be the above noted materials present in the unit.</p>						

Table 1-4. Hazardous Material Disclosure Table - English

(Tableau 1-4. Le tableau de divulgation des matières dangereuses – en Anglais)

有害物质披露表						
AF01WV10wxyz						
部件名称	有害物质					
	铅 (Pb)	汞 (Hg)	镉 (Cd)	六价铬 (Cr (VI))	多溴联苯 (PBB)	多溴二苯醚 (PBDE)
电路板	X	O	O	O	O	O
<p>本表格依据 SJ/T 11364 的规定编制。</p> <p>○：表示该有害物质在该部件所有均质材料中的含量均在 GB/T 26572 规定的限量要求以下。</p> <p>X：表示该有害物质至少在该部件的某一均质材料中的含量超出 GB/T 26572 规定的限量要求。</p> <p>注：该部件的制造日期可能会按照以下格式出现在序列号码中： wwyy1nn : ww 表示周数； yy 表示年的最后两位数； nn 是该周序列号、每周都从01开始。</p> <p>注：该部件符合欧盟RoHS指标；但是中国不像欧盟那样对零部件的有害物质有豁免，所以该部件可能包含表中列出的物质。</p>						

Table 1-5. Hazardous Material Disclosure Table - Mandarin

(Tableau 1-5. Le tableau de divulgation des matières dangereuses – en Mandarin)

2. Description

The WaveVortex rotator is compatible with a wide variety of rotating disk electrode (RDE) and rotating ring-disk electrode (RRDE) designs offered by Pine Research. Standard electrode materials include glassy carbon, gold, and platinum, but many other electrode materials are available upon request.

The WaveVortex rotator provides excellent steady-state control of constant rotation rates. The rotation rate setpoint may be manually adjusted from 100 to 8,000 *RPM* using a ten-turn potentiometer knob located on the front panel of the control unit. A push-button control is used to toggle the motor between the "run" state and the "pause" state. While the motor is rotating, a built-in tachometer measures the actual rotation rate, and this rate is continuously displayed on the front panel of the control unit.

External control of the rotation rate is possible when the WaveVortex rotator is connected to a potentiostat system capable of supplying an analog rotation rate control signal. While specific details vary from one system to another, the basic idea is that the potentiostat produces an analog signal that is proportional to the target rotation rate. This analog signal is carried by a cable, which is supplied by the potentiostat manufacturer, to an external I/O connector on the side of the control unit. This connection permits the software which controls the potentiostat to control the rotation rate using a constant voltage level for steady-state rotation.

The rotation rate is typically monitored by observing the front panel LED display on the front of the WaveVortex. Optionally, the tachometer measurement may also be monitored via a signal presented at the external I/O connector on the side of the control unit (by connecting an oscilloscope, voltmeter, or other recording device).

Internal connections to the rotating disk and ring electrodes are made mechanically through two spring-loaded silver-carbon brushes. Externally, the connections between the potentiostat cell cable and the rotating electrodes are made by connecting to banana jacks located on the back side of the motor unit. The red jack connects to the disk electrode on an RDE or RRDE, while the blue jack connects to the ring electrode when using an RRDE.

The motor unit and electrochemical cell are enclosed on the back side by a rear wall permanently attached to the enclosure base. The cell and motor are further enclosed on the front side by a transparent enclosure window. The enclosure window may be raised to the upper position to allow access to the electrochemical cell and the motor unit. However, the enclosure window must be placed in the lower position before the control unit will allow the motor to rotate.

2.1 Major System Components

The major components of the WaveVortex rotator are shown below (see: Figure 2-1).

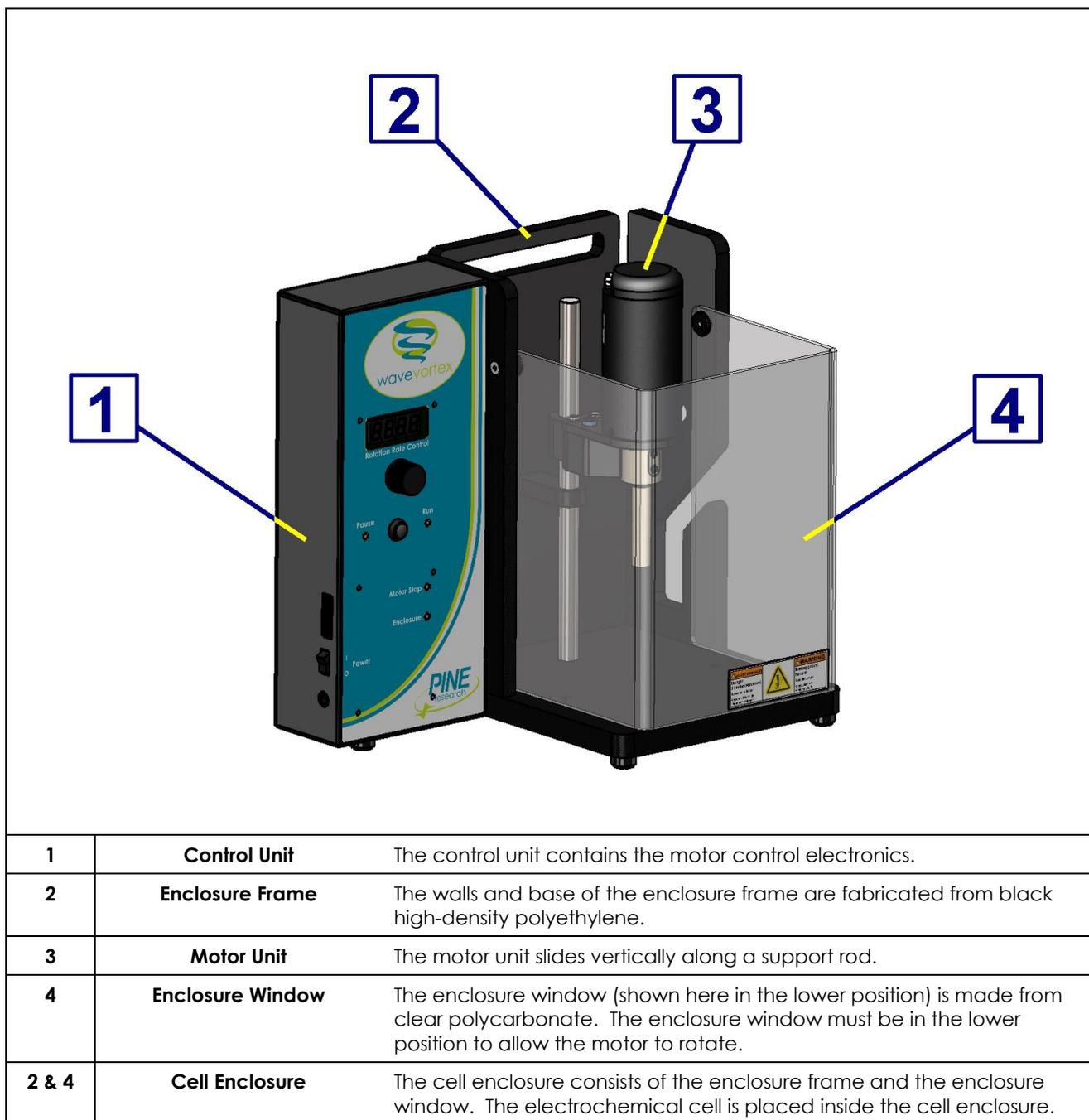


Figure 2-1: Major Components of the WaveVortex Rotator System

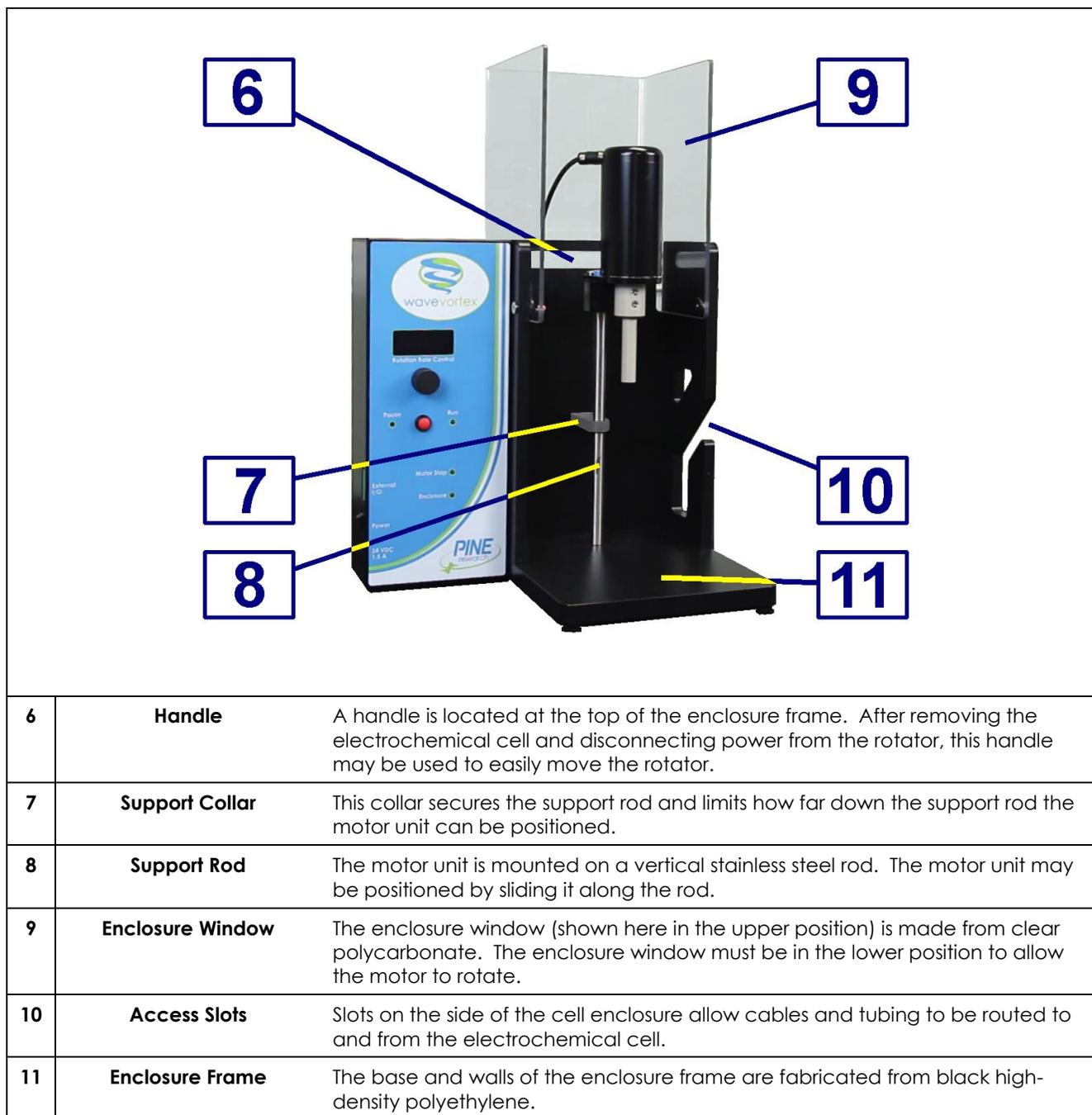
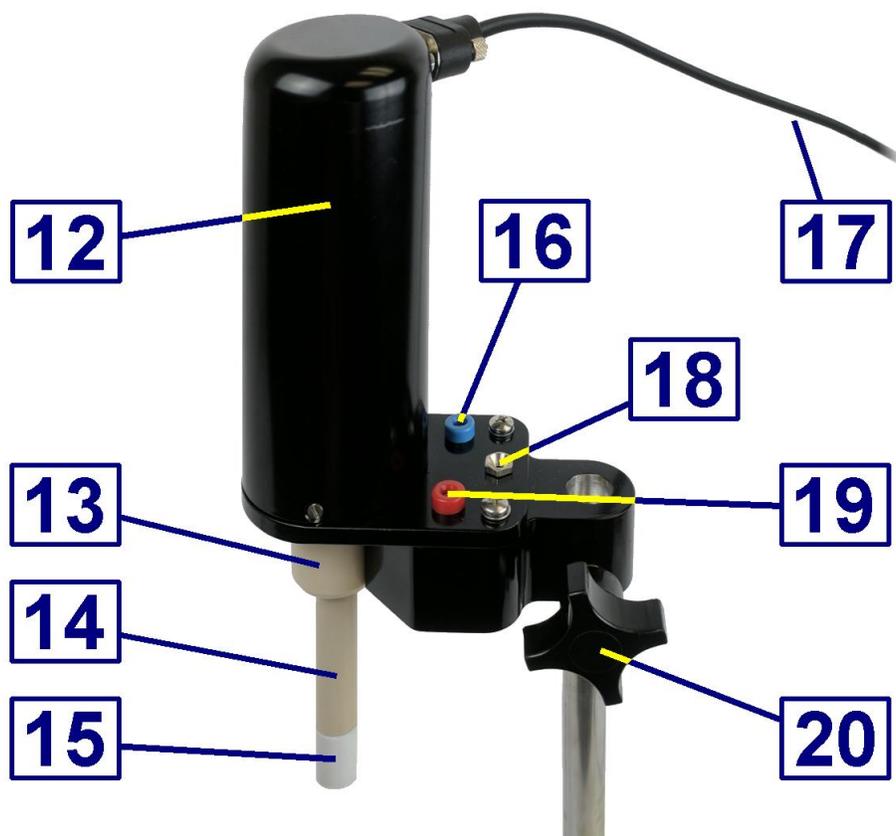


Figure 2-2. Cell Enclosure Details



12	Motor Housing	This sturdy steel housing shields the electrochemical cell from any electromagnetic interference from the motor.
13	Bearing Assembly	This assembly is fabricated from PEEK. At the top and bottom of the assembly there are stainless steel bearings which stabilize the rotating shaft. The portion of the shaft between the two bearings (hidden from view) is where two silver-carbon brushes make electrical contact with the rotating shaft.
14	Shaft	The rotating shaft is fabricated from PEEK and stainless steel.
15	Electrode Tip	Various RDE and RRDE electrode tips (sold separately) thread onto the shaft.
16	Ring Contact Jack (blue)	This blue banana jack makes electrical contact with the ring electrode when an RRDE tip is threaded onto the end of the shaft.
17	Motor Cable	One end of the motor cable connects the male DB-9 connector on the motor unit. The other end of the motor cable connects to the female DB-9 connector on the back of the control unit (see: Figure 2-5).
18	Chassis Terminal	This metal banana jack makes electrical contact with the motor unit chassis. Note that the motor unit chassis is connected to the control unit chassis via the shield line in the motor cable.
19	Disk Contact Jack (red)	This red banana jack makes electrical contact with the disk electrode when an RDE or RRDE tip is threaded onto the end of the shaft.
20	Lift Knob	This knob should be loosened when moving the motor unit up and down the support rod. Tighten the knob to secure the motor unit to the support rod.

Figure 2-3. Motor Unit Details

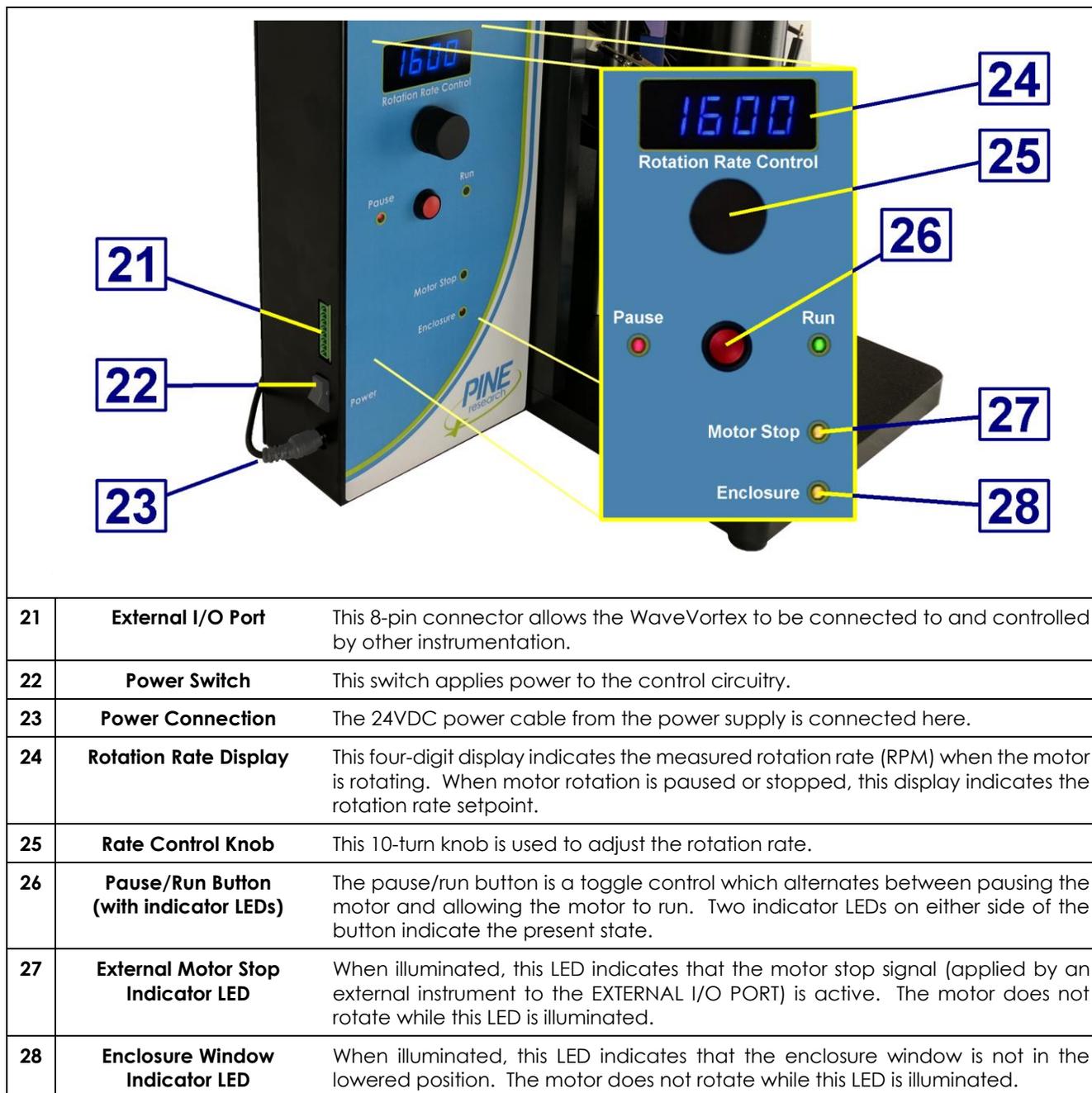


Figure 2-4. Control Unit Details

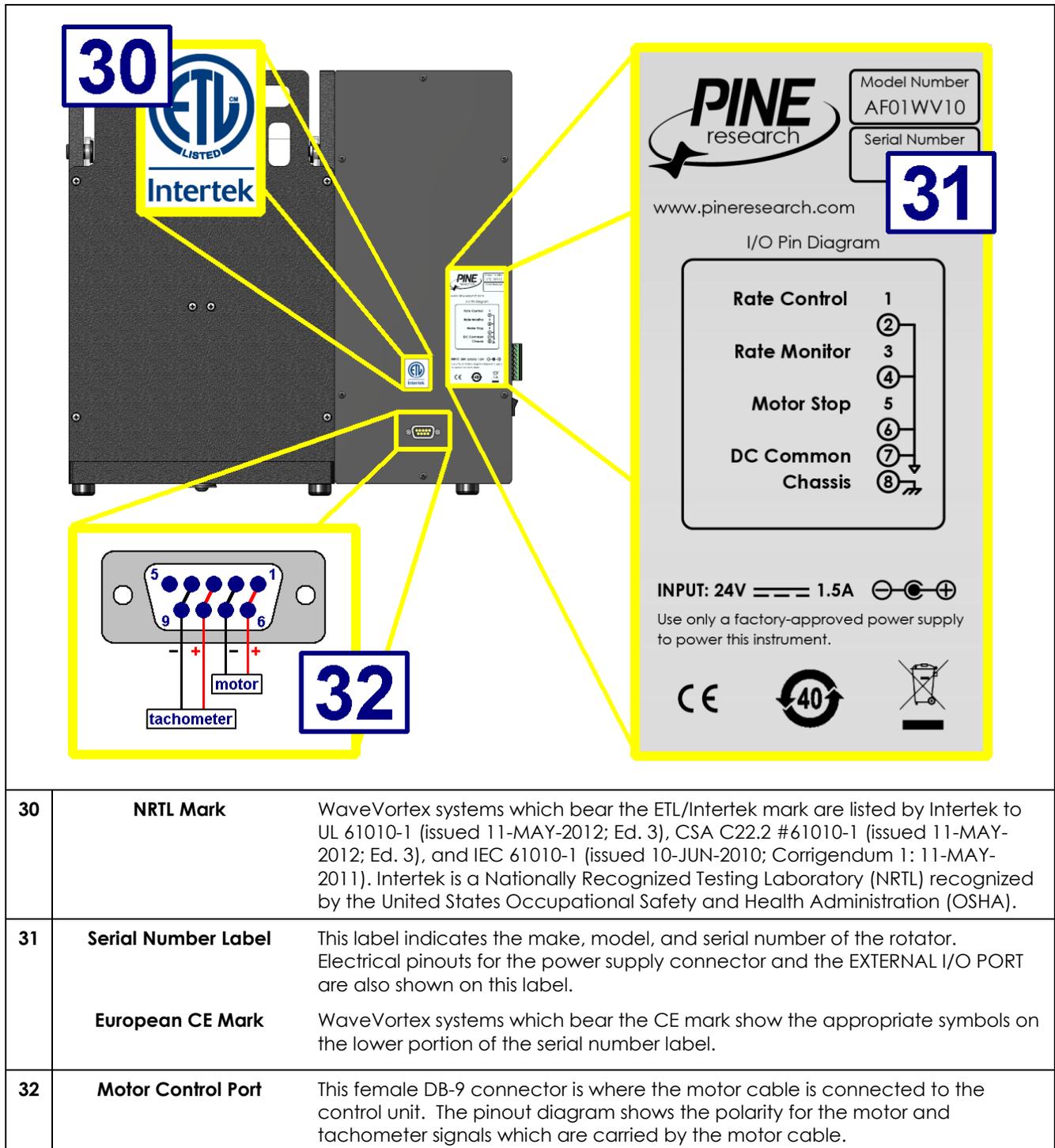


Figure 2-5. Back Panel Details

2.2 Typical Rotating Disk Electrode (RDE) Design

The external threads on Pine Research rotating disk electrode (RDE) tips mate with the internal threads on the WaveVortex's built-in shaft (see: Figure 2-6). Some RDE tips are single-piece, fixed-disk designs which cannot be taken apart. Other designs offered by Pine Research may be taken apart, allowing the disk material to be replaced (see: Section 5).

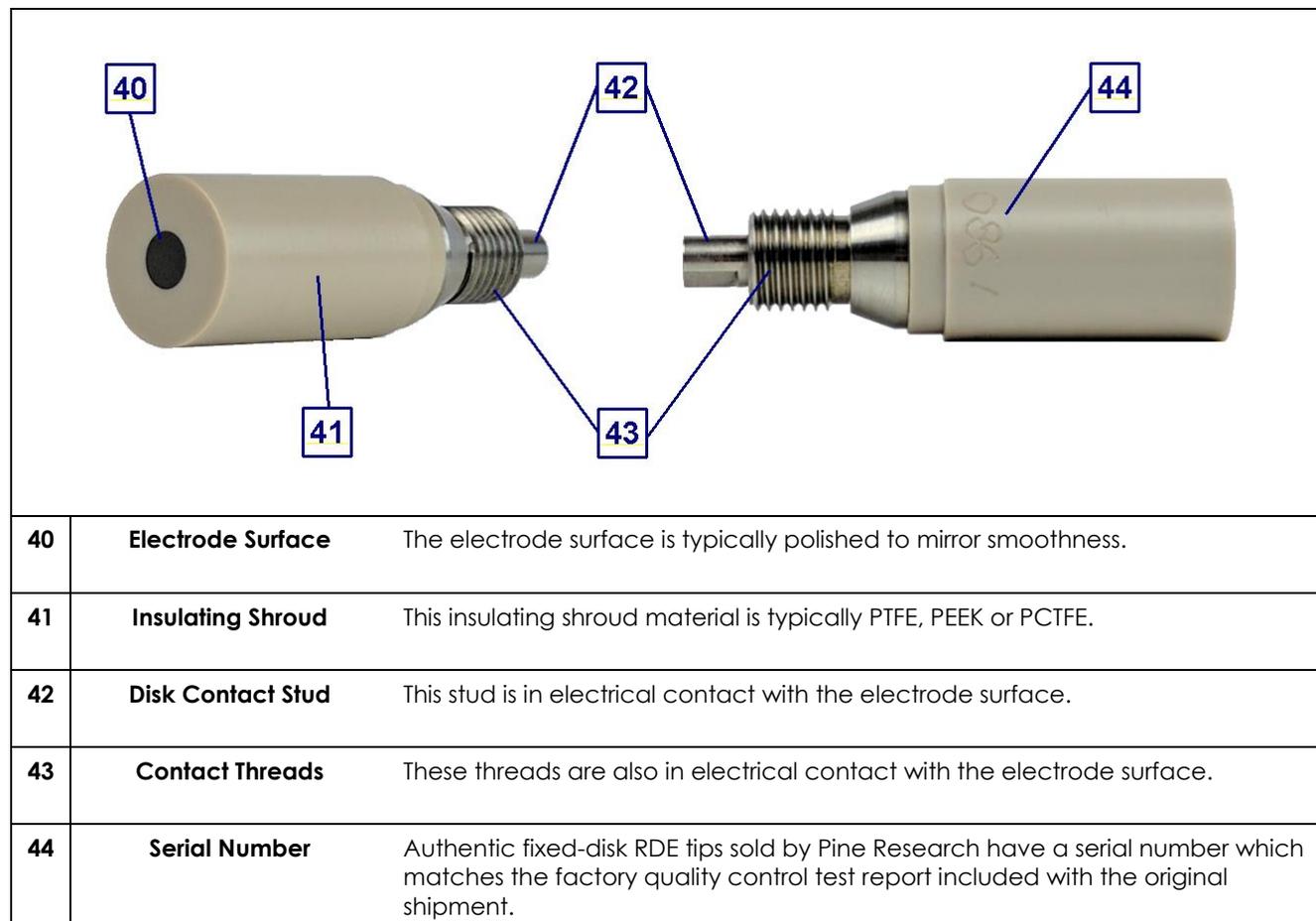


Figure 2-6. Typical Rotating Disk Electrode (RDE) Tip

2.3 Typical Rotating Ring-Disk Electrode (RRDE) Design

The external threads on Pine Research rotating ring-disk electrode (RRDE) tips mate with the internal threads on the WaveVortex's built-in shaft (see: Figure 2-7). Some RRDE tips are single-piece designs which cannot be taken apart. Other designs offered by Pine Research may be taken apart, allowing the disk material to be replaced (see: Section 5).

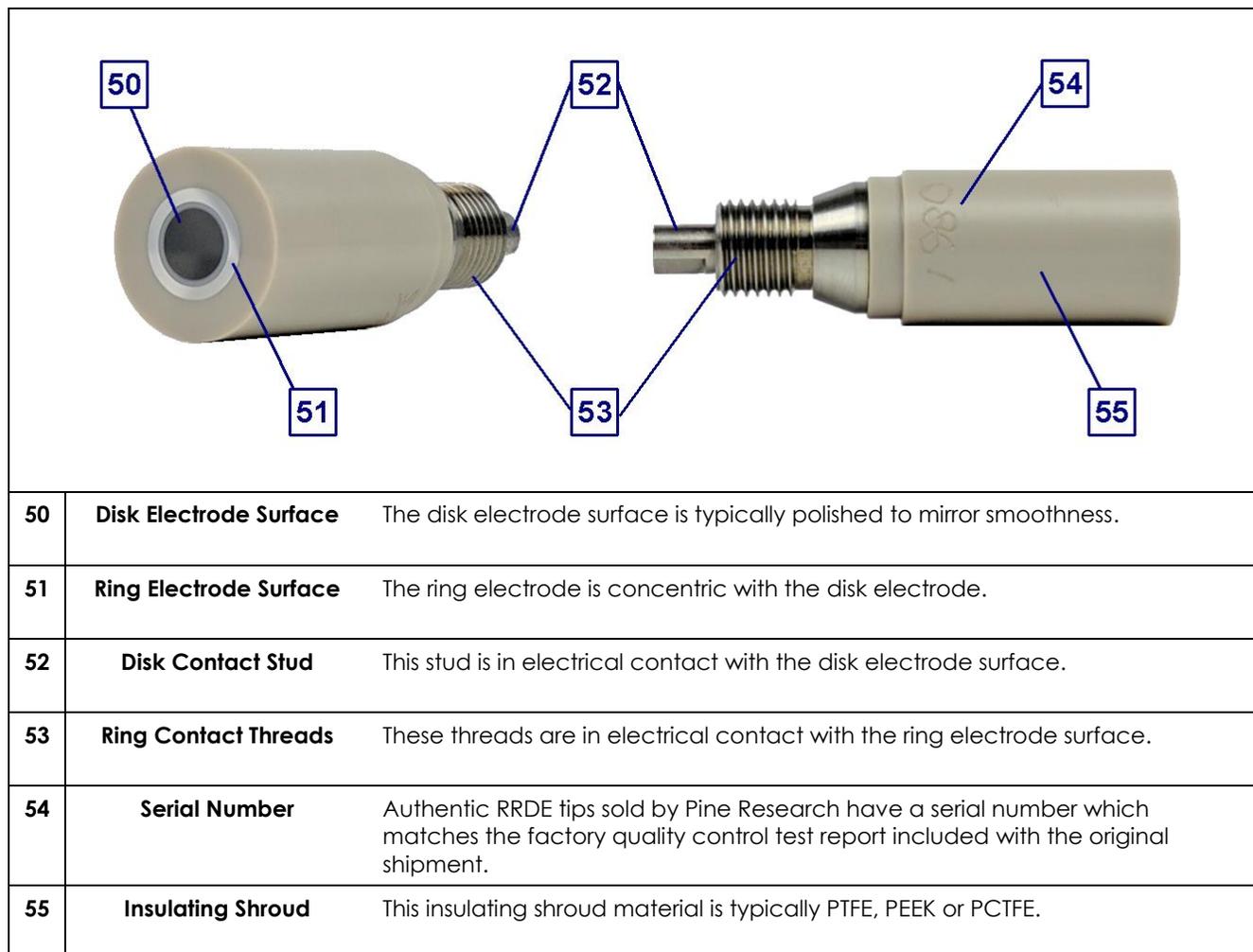


Figure 2-7: Typical Rotating Ring-Disk Electrode (RRDE) Tip

3. Installation

3.1 Site Preparation

The rotator system should be located on a sturdy and level surface (such as a laboratory bench) with ample clearance above and around the perimeter of the rotator. The front of the rotator should be unobstructed.

The rotator has a horizontal footprint which is 38 cm wide by 28 cm deep (15" wide x 11" deep). There should be at least 10 cm (3.9") clearance on each side and 15 cm (5.9") clearance behind the rotator, for a total table space of 58 cm wide by 43 cm deep (23" wide x 17" deep).

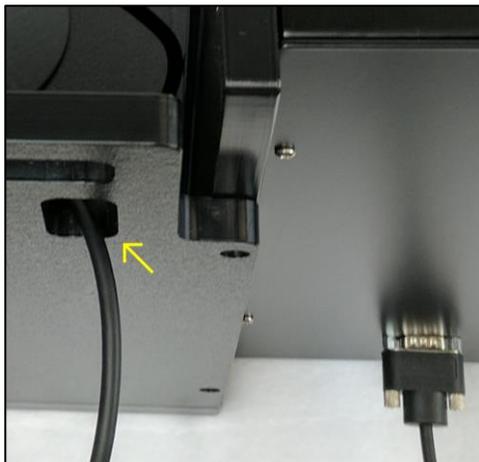
The height of the rotator is 38 cm (15") when the enclosure window is in the lower position. There must be enough vertical clearance above the rotator to allow the enclosure window to be raised to the upper position. During the process of raising the enclosure window, the window follows a path which requires a minimum of 61 cm (24") of vertical clearance with respect to the surface of the table or lab bench which supports the rotator.

3.2 Unpacking and Setting Up the Rotator

The WaveVortex rotator is shipped from the factory fully assembled. Installation requires only a few electrical connections as described below.



Connect the motor cable to the connector on the back side of the control unit.



Feed the motor cable through the opening in the back wall of the cell enclosure.



Connect the other end of the motor cable to the connector on the motor unit.



A power supply is included with the rotator. This power supply converts the laboratory AC power (AC Mains) to the DC power required by the rotator.

The power supply input (AC) is a standard C14 input connector which is compatible with a wide variety of international power cords.

The power supply output (DC) is a round barrel-style connector located at the end of the cable.



Connect the round barrel-style connector to the power input on the side of the control unit.



An appropriate international power cord (10 A rating) is provided with the rotator. Connect this power cord to the C14 connector on the power supply. Then, connect the other end of the power cord to the laboratory AC power (AC Mains).

Note that the AC mains must provide an earth ground connection for the third prong on the power cord and must provide AC power which meets the required specifications for the rotator system (see: Section 1.7.2).



There are three banana jacks located on the rotator unit. Two of these jacks contact the rotating disk and ring electrodes, and the third jack is the chassis connection. If the rotator is being used with a potentiostat cell cable which terminates at banana plugs, then these banana plugs can be directly connected into the banana jacks. However, if the potentiostat cell cable terminates with alligator clips, then it is necessary to install banana studs in the banana jacks so that there is a place to clip the alligator clips. Three banana studs are included with the rotator (see image) and may be installed if needed.

**WARNING:**

Failure to connect the third prong of the power cord to a proper earth ground may impair the protection provided by the system.

AVERTISSEMENT:

L'absence de connexion de la troisième broche du cordon d'alimentation à une prise de terre appropriée peut altérer la protection fournie par le système.

**WARNING:**

A detachable main power cord is provided with the rotator. Do not replace this cord with an inadequately rated cord.

AVERTISSEMENT:

Un cordon d'alimentation amovible est fourni avec le rotateur. Ne remplacez pas ce cordon par un cordon de calibre inadéquat.

4. Operation

This section of the user guide discusses information pertaining to routine operation of the rotator. Users of the rotator should be familiar with all information in this section prior to operating the rotator.

4.1 The Cell Enclosure

The WaveVortex rotator features an enclosure in which the electrochemical cell is placed during an experiment (see: Figure 4-1). The walls and base of the enclosure are made from chemically-resistant high-density polyethylene (black), and the transparent window in the front of the enclosure is made from polycarbonate. The enclosure window may be raised to provide easy access to the electrochemical cell between experiments. The enclosure window must be lowered during the experiment to allow the motor to rotate.



NOTE:

The enclosure window must be in the **LOWER POSITION** to allow the motor to rotate. A position sensor prevents the motor from rotating when the enclosure window is in the **UPPER POSITION**.

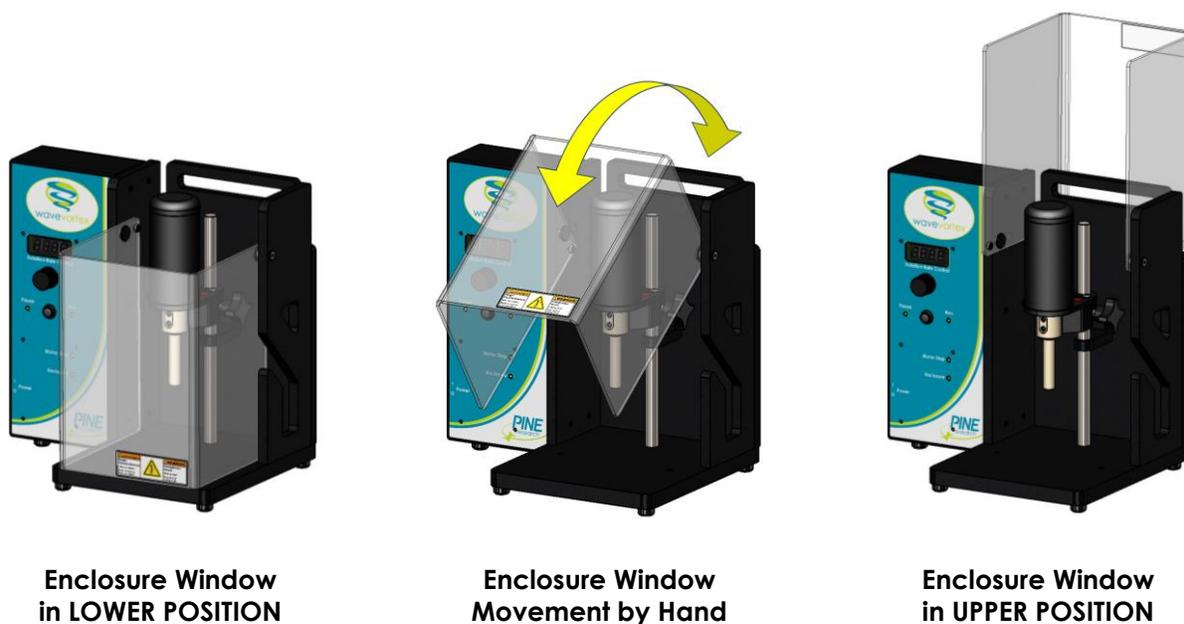


Figure 4-1. Raising and Lowering the Enclosure Window

4.2 The Rotating Shaft

The electrode shaft normally rotates in a clockwise direction as viewed from the top of the rotator. The shaft is compatible with a variety of Rotating Disk Electrode (RDE) or Rotating Ring-Disk Electrode (RRDE) tips offered by Pine Research (see: Section 5). The rotating shaft is designed to be permanently mounted to the motor unit and should not be removed during normal operation of the rotator.

Inside the motor unit, internal electrical connections to the rotating shaft are made using silver-carbon brushes to contact metal surfaces on the upper portion of the rotating shaft. The shaft provides two

internal current paths down to the electrode tip: one path for the disk and the other path for the ring. These current paths are electrically isolated from the chassis of the motor unit.

The standard shaft included with the WaveVortex rotator has a chemically-resistant PEEK shroud along the entire length of the shaft. The bottom of the shaft has internal threads compatible with various Pine Research RDE and RRDE tip designs.



WARNING:

Do not turn on the rotator or rotate the electrode shaft if the shaft is not securely mounted in the motor unit. Inspect the shaft to be certain that it is securely mounted.

AVERTISSEMENT:

Ne mettez pas le rotateur en marche ni l'arbre de l'électrode en rotation si l'arbre n'est pas installé d'une façon sécurisée dans l'unité moteur. Inspectez l'arbre pour vous assurer qu'il est bien fixé.



WARNING:

Do not use an electrode shaft which appears to wobble, vibrate, or tilt away from the axis of rotation while rotating. Such a shaft is either improperly installed or physically damaged. Turn off the rotator, disconnect electrical power, and remove the shaft immediately.

AVERTISSEMENT:

N'utilisez pas un arbre d'électrode qui semble osciller, vibrer ou dévier de l'axe de rotation pendant la rotation. Un tel arbre est mal installé ou endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'arbre immédiatement.

4.3 Gas-Purged Bearing Assembly

Pine Research offers an **optional** gas-purged bearing assembly which fits into a 24/25 center port on an electrochemical cell. The main body of the assembly is made from chemically-resistant PEEK polymer, and the internal bearing is ceramic (see: Figure 4-2). Although the bearing is not perfectly sealed, the inner diameter of the bearing (15 mm ID) allows the precision-machined shaft (15 mm OD) on the motor unit to pass through the bearing assembly with a reasonably tight fit.

Some electrode tips have a small enough diameter that the bearing assembly can easily slide around the tip and onto the shaft. Other electrode tips have a large enough diameter that the bearing assembly must be slid on to the shaft before the electrode tip is threaded into the shaft. In the latter case, follow the instructions for changing the electrode tip (see: Section 4.4), but be sure to slide the bearing assembly onto the shaft before installing the electrode tip.

A small plastic hose barb on the side of the bearing assembly allows the space within the bearing assembly to be purged with an inert gas. A slight positive pressure of inert purge gas helps to protect the contents of the electrochemical cell from the laboratory atmosphere.



Figure 4-2. Gas-Purged Bearing Assembly

4.4 Changing the Electrode Tip

Electrode tips are designed to be easily installed or removed by hand. Each tip has threads which match the internal threads at the bottom of the shaft. Do not use tools to install or remove an electrode tip, and use only those electrode tips recommended by Pine Research (see: Section 5).



INFO:

No tools are required to install or remove an electrode tip. If an electrode tip cannot be easily removed by hand from the shaft, contact Pine Research for assistance.

Before removing or installing an electrode tip, turn off the rotator and disconnect the power cord from the rotator. Move the enclosure window to the upper position and slide the motor unit to the top of the support rod to provide easy access to the end of the shaft.

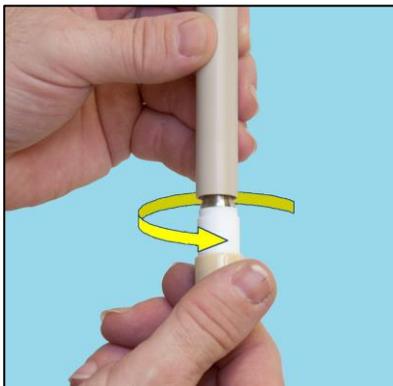
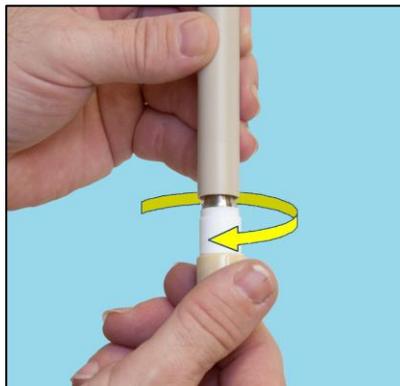


Figure 4-3. Removing and Installing an Electrode Tip

When removing a tip from the shaft (see: Figure 4-3, left), use one hand to prevent the shaft from rotating while using the other hand to gently unscrew the tip from the shaft. When an electrode tip is not in use, store the tip in a safe place (such as the original packaging in which it was shipped).

To install a new tip on the shaft (see: Figure 4-3, middle), gently thread the tip onto the shaft and tighten it by hand. Use one hand to prevent the shaft from rotating while gently tightening the tip onto the shaft.

When a tip is properly hand-tightened onto the shaft, there will be a small but visible gap between tip and shaft (see: Figure 4-3, right). This visible gap (approximately 1.6 mm tall) is part of the design of the shaft and tip. Do not overtighten the tip in an effort to close this gap.



WARNING:

Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before installing or removing an electrode tip on the end of the shaft.

AVERTISSEMENT:

Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant d'installer ou d'enlever un embout d'électrode à l'extrémité de l'arbre.



WARNING:

Do not use or attempt to rotate an electrode tip that has been dropped, bent or otherwise physically damaged. Inspect the electrode tip to be certain that it is not damaged.

AVERTISSEMENT:

N'utilisez pas et ne tentez pas de mettre en rotation un embout d'électrode qui est tombé, a été tordu ou a été endommagé physiquement d'une manière ou d'une autre. Inspectez l'embout d'électrode pour vous assurer qu'il n'a pas été endommagé.



CAUTION:

Do not use tools on the shaft or electrode tip.

Never use a tool to unscrew a tip from a shaft.

If a tip cannot be removed from a shaft by hand, then contact our production facility for further instructions.

ATTENTION:

N'utilisez pas d'outils sur l'arbre ou sur l'embout d'électrode.

N'utilisez jamais un outil pour dévisser un embout d'électrode d'un arbre.

Si un embout d'électrode ne peut être manuellement retirée d'un arbre, contactez notre usine de fabrication pour obtenir des instructions supplémentaires.

4.5 Installing the Electrochemical Cell

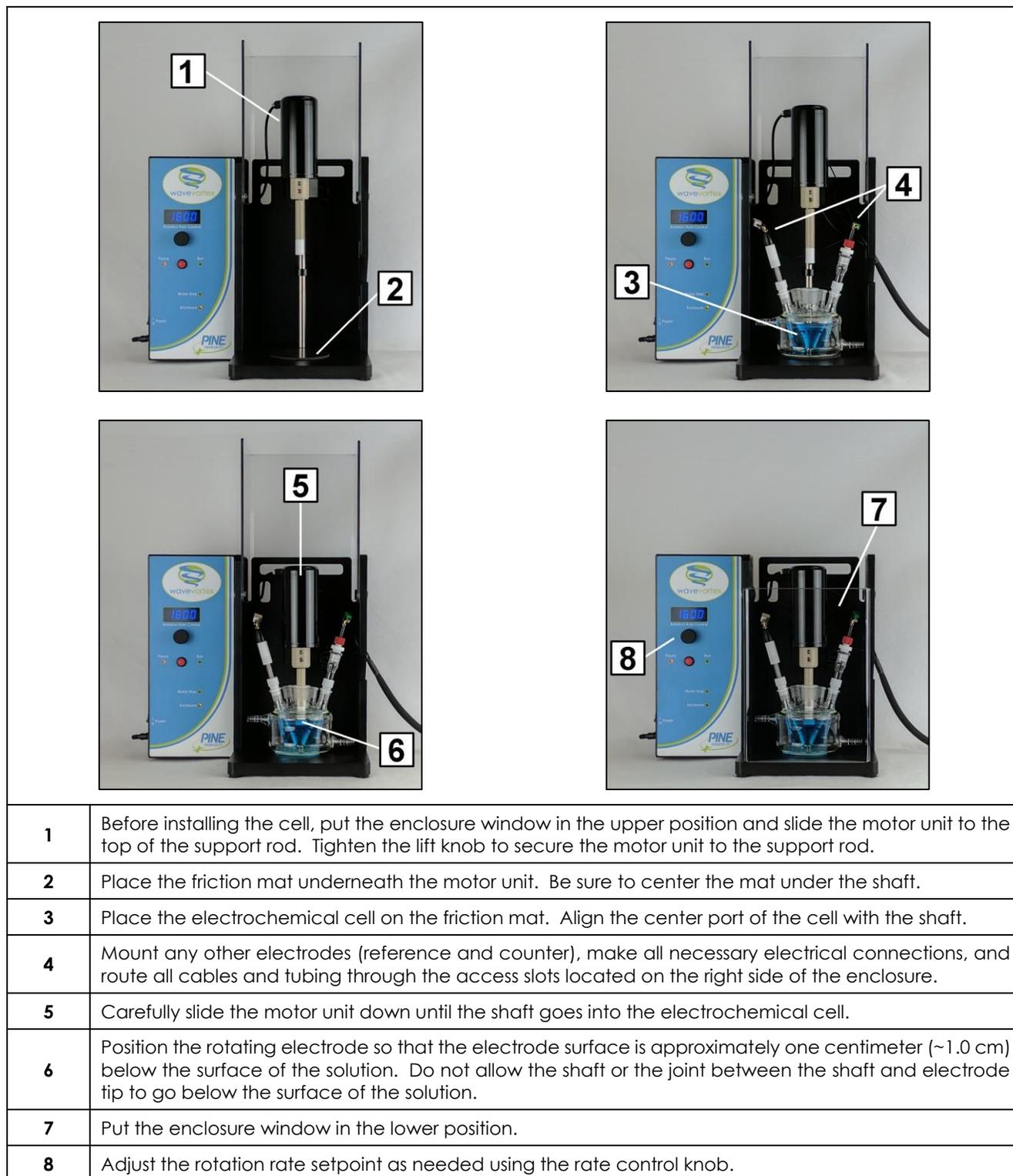


Figure 4-4. Installing the Electrochemical Cell

Proper installation of the electrochemical cell within the enclosure is a multi-step process (see: Figure 4-4). The enclosure is large enough to accommodate an electrochemical cell equipped with various electrodes, tubing connections, sparge/purge tubes, and other accessories.

The cell should be placed on top of the friction mat (see: Figure 4-5). The center opening on top of the electrochemical cell should be aligned with the axis of the shaft on the motor unit.

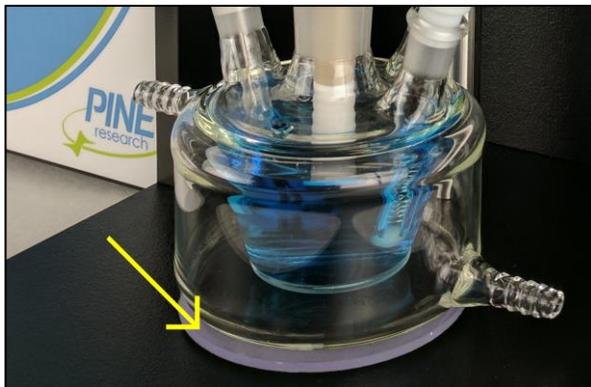


Figure 4-5. Friction Mat under the Cell



Figure 4-6. Access Slots for Cables and Tubing

When installing the cell in the enclosure, careful thought should be given to how the various electrode cables and tubes are routed in and out of the enclosure. The right-hand wall of the enclosure has access slots intended for such cables and tubing (see: Figure 4-6).



CAUTION:

Always place a friction mat below the electrochemical cell.

The friction mat helps to prevent the cell from sliding back and forth while electrodes and tubing are being installed in the cell.

ATTENTION:

Placez toujours un tapis à friction sous la cellule électrochimique.

Ce tapis permet d'éviter le mouvement de va-et-vient alors que les électrodes et les tubes sont installés dans la cellule.

When lowering the rotating electrode down into the cell, the electrode tip should be positioned so that the electrode surface is approximately five to ten millimeters (5 to 10 mm) below the solution surface (see: Figure 4-7). Most electrode tips are at least twenty-five millimeters (25 mm) long, and no more than half the length of the electrode tip should be below the solution surface. It is particularly important that the gap between the shaft and the electrode tip always remains above the solution surface.

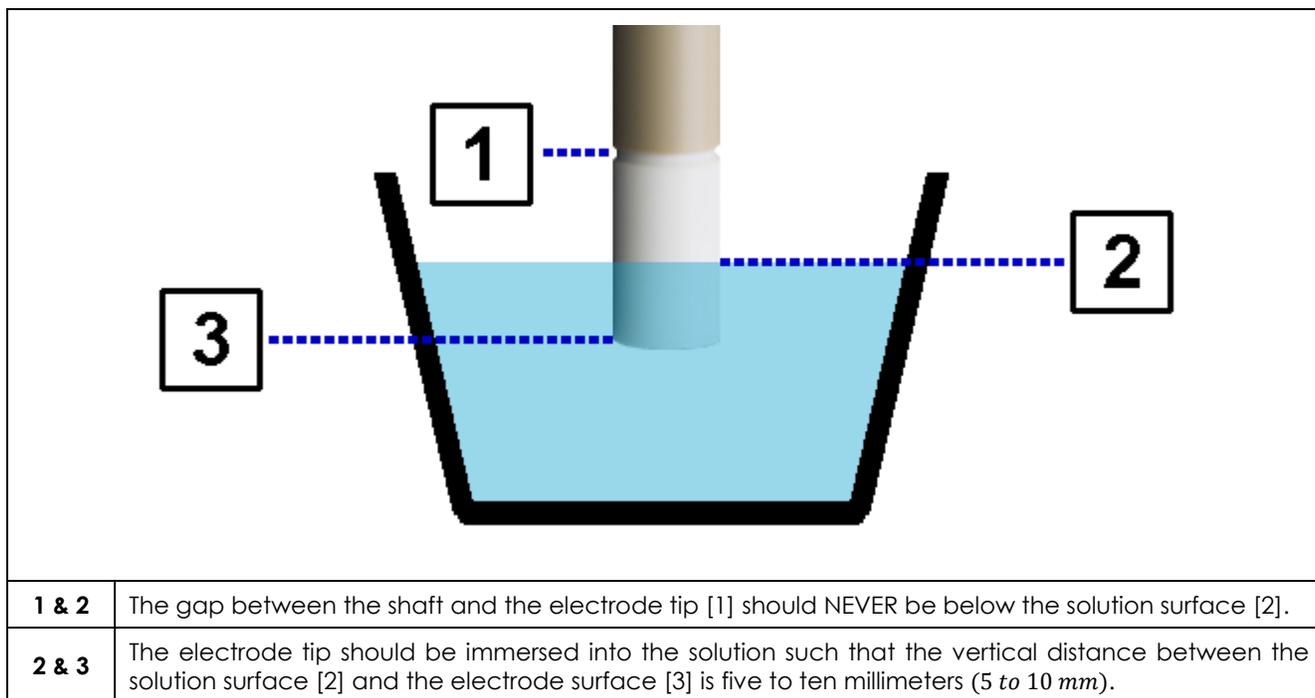


Figure 4-7. Proper Immersion Depth for the Rotating Electrode Tip



CAUTION:

Position the motor unit with respect to the glass cell so that the electrode tip is immersed approximately five to ten millimeters (5 to 10 mm) into the test solution.

Excessive immersion may corrode the shaft or tip by allowing liquids to seep into the gap between the shaft and tip.

ATTENTION:

Positionnez le bloc moteur en fonction de la cellule en verre, de sorte que l'embout d'électrode soit immergé d'environ cinq à dix millimètres (5 to 10 mm) dans la solution d'essai.

Une immersion excessive peut entraîner la corrosion de l'arbre ou de l'embout en permettant aux liquides de s'infiltrer entre eux.



CAUTION:

When raising or lowering the motor unit along the main support rod, be sure to hold the motor unit carefully so that it does not unexpectedly fall and break the glass cell located below the motor unit.

ATTENTION:

Lors de l'élévation ou l'abaissement du bloc moteur le long de la barre principale, assurez-vous de bien le maintenir pour éviter qu'il tombe brutalement et casse la cellule en verre située sous le bloc moteur.

**CAUTION:**

Center the rotating electrode within the opening on the cell so that it does not rub against the walls of the opening.

Damage will occur if the rotating shaft or tip abrades against these walls.

ATTENTION:

Centrez l'électrode tournante dans l'ouverture de la cellule de façon qu'elle ne frotte pas aux bords.

Des dommages se produisent si l'arbre ou l'embout frottent contre les bords de la cellule.

4.7 Rotation Rate Control

The primary control for turning the motor on and off is the RUN/PAUSE push-button on the control unit. This button is flanked on either side by LED indicators (see: Figure 4-8) that indicate whether the rotator is in the “run” state or the “pause” state.

A 10-turn rotation RATE CONTROL KNOB is used to adjust the rotation rate setpoint. A four-digit display shows the setpoint (in *RPM*) when the rotator is in the “pause” state. When the rotator is in the “run” state, the display shows the actual rotation rate measured by the tachometer inside the motor unit.

Two additional LED indicators signal other conditions that can cause the motor to stop rotating. The ENCLOSURE indicator is illuminated when the enclosure window is raised. The MOTOR STOP indicator is illuminated when an external instrument (such as a potentiostat) sends a digital signal to the WaveVortex to halt rotation (see: Section 4.14.3). If either of these LED indicators is illuminated, then the motor will not rotate.

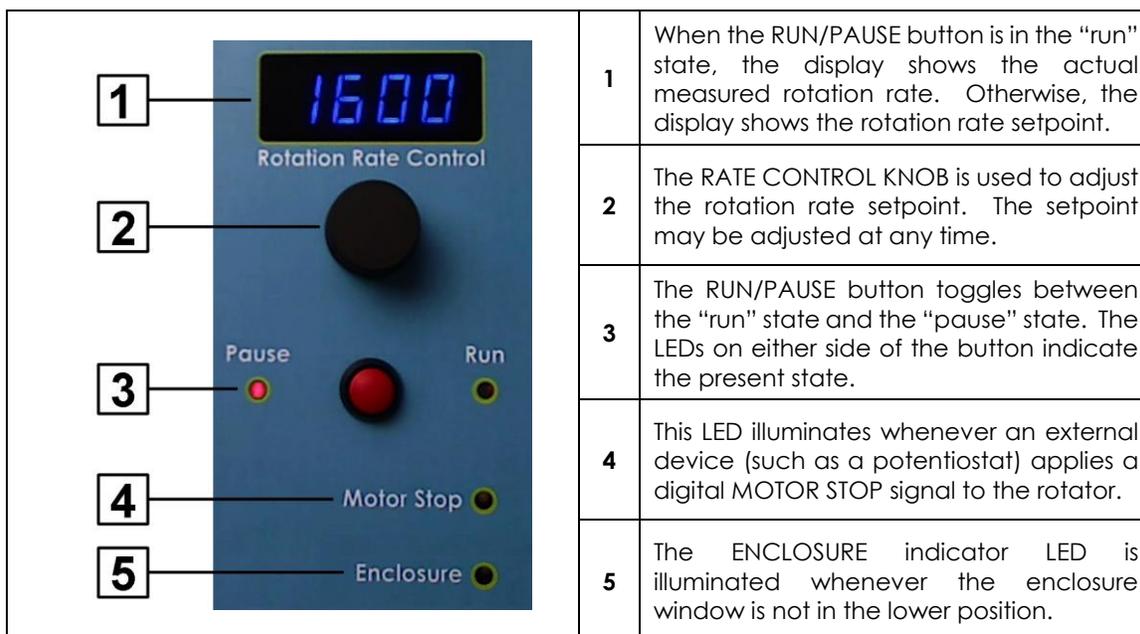


Figure 4-8. Rotation Rate Controls

4.7.1 Initial Power Sequence

When the WaveVortex is initially powered on, the RUN/PAUSE button is in the “pause” state to prevent the motor from rotating. The number initially shown in the display is the rotation rate setpoint; that is, it is the target rate at which the motor will rotate once the motor begins to run (see: Figure 4-9).

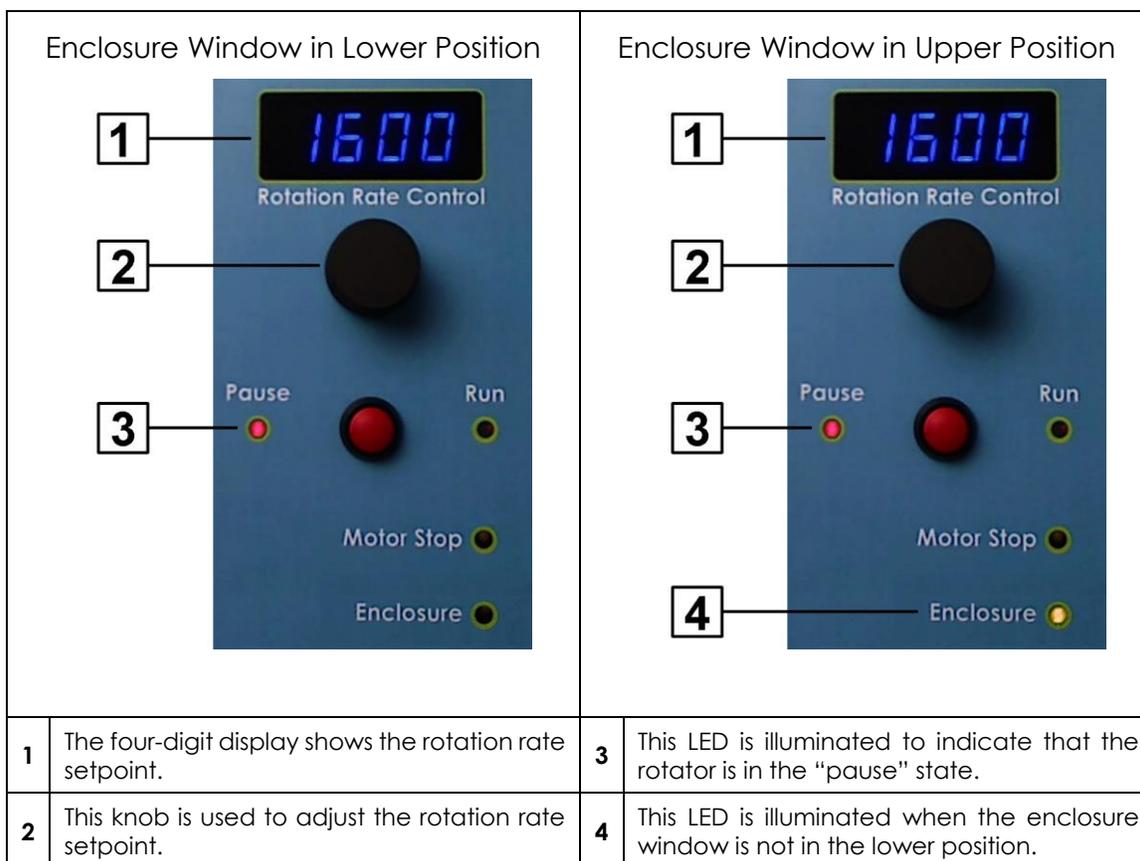


Figure 4-9. Appearance of Controls when Rotator is in the “Pause” State

After the rotator has been powered on and while the RUN/PAUSE button is still in the “pause” state, the rate control knob should be used to select the desired rotation rate. In general, it is a good idea to start with a slow rotation rate (*i.e.*, less than 200 RPM) at the beginning of an experiment. Then, after verifying that the electrochemical cell, signal cables, tubing, and other accessories are securely positioned, measurements can be made at higher rotation rates as needed.

4.7.2 Rotating the Electrode

Before attempting to rotate the electrode, use the RATE CONTROL KNOB to select a slow rotation rate (*i.e.*, less than 200 RPM). Then, make certain that the enclosure window is in the lower position (see: Figure 4-1), and then press the RUN/PAUSE button to put the rotator in the “run” state (see: Figure 4-10).

While the shaft is slowly rotating, inspect the rotating shaft and tip to assure that both the shaft and electrode tip are rotating properly about the axis of rotation. If either the shaft or the tip is wobbling, vibrating, or tilting away from the axis of rotation, then turn off the rotator and repair or replace the shaft (or tip) immediately.

If the shaft is rotating properly along the axis of rotation, then the rotation rate may be slowly increased to the desired value using the RATE CONTROL KNOB (see: Figure 4-10, left). Note that while the rotator

is in the “run” state, the four-digit display shows the actual rotation rate measured by the tachometer inside the motor unit. This measured value may vary slightly over time, but it should remain within the specified tolerance for the rotation rate (see: Section 1.7.2).

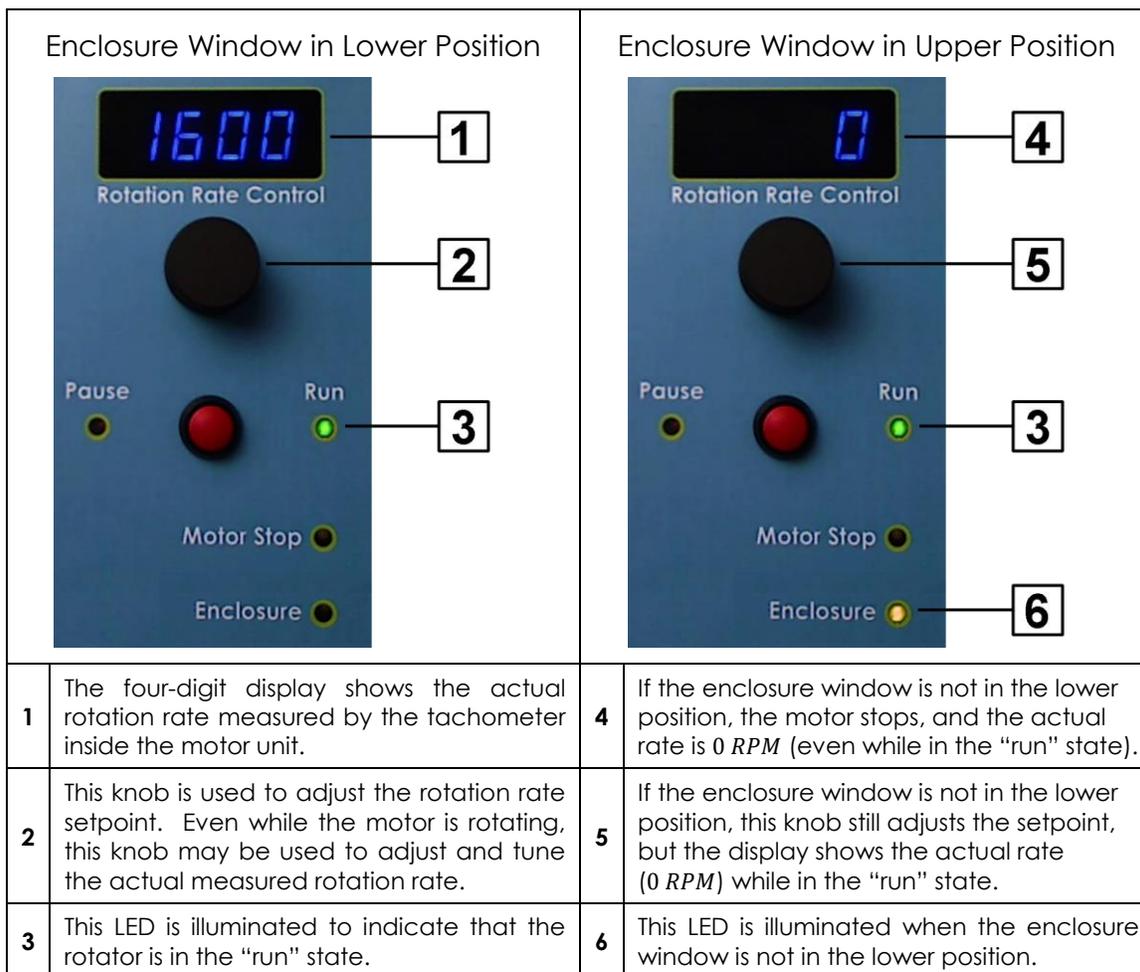


Figure 4-10. Appearance of Controls when Rotator is in the “Run” State

While the electrode is rotating at the desired rotation rate, electrochemical measurements may be made using an appropriate potentiostat system. Note that some potentiostat systems have the ability to control the rate of rotation via a signal cable from the potentiostat to the rotator (see: Section 4.14).

4.8 Stopping the Motor

The preferred method for stopping the motor is to press the RUN/PAUSE button and place the rotator in the “pause” state. Note that there are two other cases described below that cause the motor to stop, even if the rotator is in the “run” state.

The first case is when the enclosure window is raised (*i.e.*, not in the lower position) while the motor is running. If the window is raised, the control unit will stop the motor. Because the rotator is in the “run” state, the display shows the rotation rate of the stopped motor (0 *RPM*) as measured by the tachometer (see: Figure 4-11, left). If the enclosure window is returned to the lower position, the motor will immediately resume rotating, and the display will show the rotation rate.

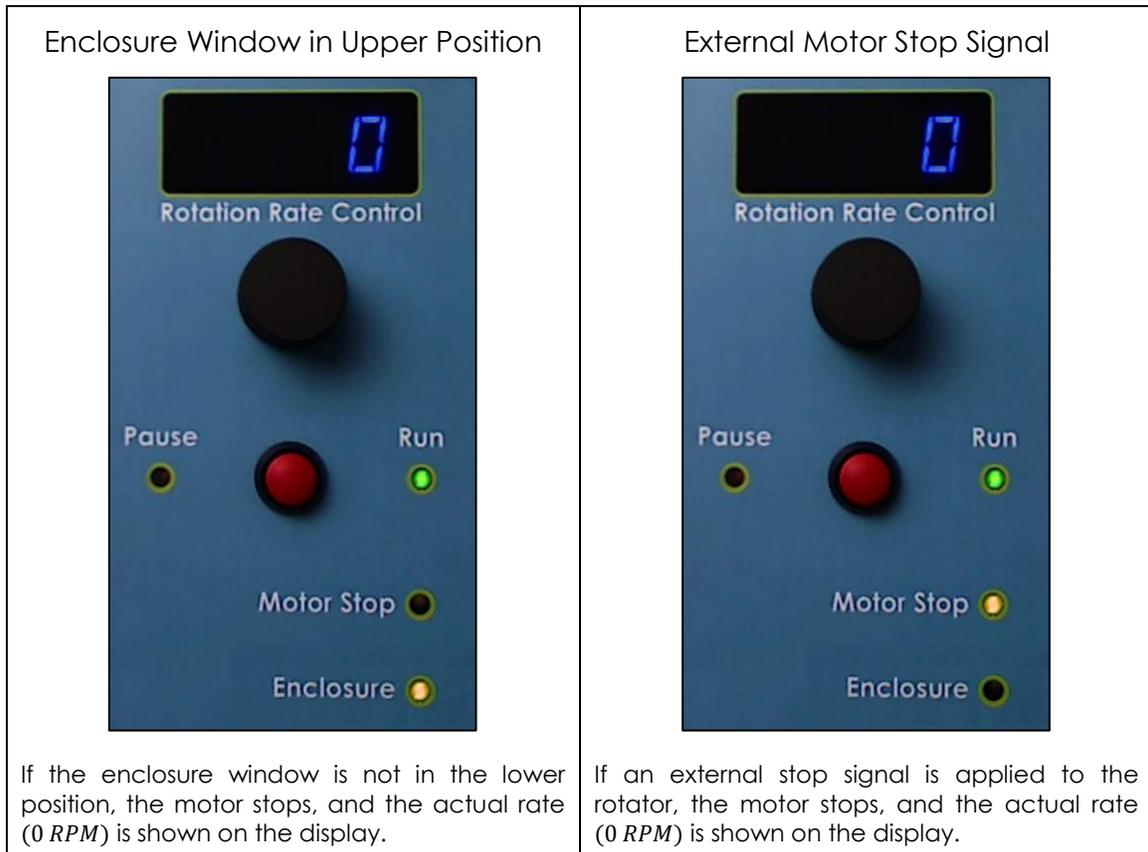


Figure 4-11. Motor Stop Conditions when Rotator is in the “Run” State

If the rotation rate is under the control of an external potentiostat system (via a special signal cable connected from the potentiostat to the EXTERNAL I/O PORT on the side of the rotator), then it is possible for the potentiostat to send a signal to the rotator to halt rotation. Whenever this external signal is applied to the rotator, the MOTOR STOP indicator on the front panel is illuminated. Even though the rotator remains in the “run” state, the display will show the actual rotation rate of the stopped motor (0 RPM) as measured by the tachometer (see: Figure 4-11, right).



CAUTION:

When the rotator is in the “run” state but the motor is stopped, the display shows the actual rotation rate of the stopped motor (0 RPM) rather than the rotation rate setpoint.

In such a condition, adjustments to the ROTATION RATE CONTROL knob are not recommended because the setpoint is not visible.

ATTENTION:

Lorsque le rotateur est en état de "marche" mais que le moteur est en arrêt, l'affichage montre le taux de rotation réel du moteur arrêté (0 tr/min) plutôt que la consigne de la vitesse de rotation.

Dans une telle situation, les réglages du bouton "ROTATION RATE CONTROL" ne sont pas recommandés car la consigne n'est pas visible.

4.9 Cell Connections

The counter electrode and the reference electrode are usually mounted in appropriate side ports on the electrochemical cell (see: Figure 4-12). The counter electrode is often a simple platinum wire or carbon rod to which an alligator clip is easily affixed.



Figure 4-12. Connection of Counter and Reference Electrodes

Always consult the manual for the potentiostat system to determine which cell cable leads should be connected to the counter and reference electrodes. Many commercially available reference electrodes have a sturdy pin connector on the top end which can mate with a reference electrode cable with a matching pin socket. Alternately, an alligator clip can be connected to the pin connector. The cable that connects the reference electrode to the potentiostat should be shielded (coaxial), and care should be taken to route this cable well away from noise sources such as power cords, networking cables, or video monitors.



NOTE:

Cell cables on newer model Pine Research potentiostats use **GREEN** to mark the counter electrode connection and **WHITE** to mark the reference electrode connection (see: Figure 4-12).

Older Pine Research bipotentiostats use **RED** to mark the counter electrode and use a BNC connector for the reference electrode.



TIP:

There is no universally accepted color coding scheme for marking potentiostat cell cable connections. If you are using the rotator with a third-party potentiostat, consult the potentiostat documentation for information about the cell cable markings.

4.10 Rotating Disk Electrode (RDE) Connections

There are three banana jacks located on the motor unit. The red jack is used to make electrical contact with a rotating disk electrode (RDE). Connect the working electrode cable(s) from the potentiostat directly to the red banana jack (see: Figure 4-13).



Figure 4-13. Electrode Connections for a Rotating Disk Electrode (RDE)



TIP:

Most modern potentiostats provide separate cable connections for the working electrode “drive” line and for the working electrode “sense” line. The drive line carries current while the sense line measures the potential. Both of these lines must be connected to the disk electrode jack (red) on the motor unit.



NOTE:

Cell cables on newer model Pine Research potentiostats use RED to mark the working electrode “drive” line and ORANGE to mark the working electrode “sense” line. Both of these should be to the disk electrode jack (red) on the motor unit (see: Figure 4-13).



NOTE:

Older Pine Research bipotentiostats use only one cable connection for both the “drive” and the “sense” signals. This connection is marked with a YELLOW banana jack on the bipotentiostat front panel.

4.11 Rotating Ring-Disk Electrode (RRDE) Connections

There are three banana jacks located on the motor unit. When using a rotating ring-disk electrode (RRDE), the red jack makes electrical contact with the disk, and the blue jack makes contact with the ring. Connect the various working electrode cables from the potentiostat directly to these jacks (see: Figure 4-14).



Figure 4-14. Electrode Connections for a Rotating Ring-Disk Electrode (RRDE)



TIP:

A bipotentiostat is required when working with a rotating ring-disk electrode. A bipotentiostat provides independent control of two different working electrodes in the same electrochemical cell.



NOTE:

Cell cables on newer model Pine Research bipotentiostats use RED and ORANGE to mark the first working electrode drive and sense lines, respectively. Both of these cables must be connected to the red banana jack on the motor unit.

Cell cables on newer model Pine Research bipotentiostats use BLUE and VIOLET to mark the second working electrode drive and sense lines, respectively. Both of these cables must be connected to the blue banana jack on the motor unit.



NOTE:

Older Pine Research bipotentiostats use only one cable for both the “drive” and the “sense” signals. The YELLOW connection corresponds to the disk, and the BLUE connection corresponds to the ring.

4.12 Proper Grounding

The general goal of a grounding strategy is to reduce the level of signal noise in the electrochemical measurement caused by noise sources in the laboratory environment. To avoid issues with laboratory noise sources, it is important to properly ground all metal objects near an electrochemical cell and to make proper grounding connections between the rotator, the potentiostat and any other electronic equipment being used as part of the experiment.

A modern laboratory is often full of noise sources that can interfere with the measurement of small amplitude electrochemical signals. Computers, LCD displays, video cables, network routers, network cables, ovens, hotplates, stirrers, and fluorescent lighting are all examples of common laboratory equipment that may electromagnetically interfere with a delicate electrochemical measurement.

In general, the electrochemical cell, the cell cable, the rotator, and the potentiostat should be located as far away from such noise sources as possible. It is especially important that the cell cable be located well away from any digital noise sources such as mouse or keyboard cables, network cables, video cables, USB cables, cell phones, etc. The reference electrode cable is particularly sensitive to picking up noise from the environment. Also, any piece of laboratory equipment which intermittently draws a lot of current, such as an oven or hotplate under thermostatic control, should not be powered using the same branch circuit as the potentiostat. When such a piece of equipment goes through a power cycle, it may induce noise or a glitch in the electrochemical measurement.

When using a rotator in conjunction with a potentiostat or other electronic instrumentation, it is often necessary to make grounding connections between the various instruments. A proper approach to making such connections begins with a good understanding of the terminology associated with grounding. In an electrochemical experiment, there are three general kinds of grounding connections, and these three are often confused with one another: the **earth ground**, the **chassis terminal**, and the **DC Common**. These are discussed in more detail below.

4.12.1 Earth Ground



EARTH GROUND:

An earth ground connection is a direct physical and electrical connection to the Earth.

An earth ground connection is available in most modern laboratories via the third prong on the power receptacle for the local power system (see: Figure 4-15). The power system infrastructure for a laboratory building usually has a long metal probe buried in the earth, and the third prong of the electrical outlets in the building wiring is connected to this earth connection. Many scientific instruments have a three-prong power cord which brings the earth ground connection to the instrument's power supply. Depending upon the design of the instrument, the earth ground connection may or may not pass through the power supply to the circuitry inside the instrument.

The power supply for the WaveVortex rotator is connected to the earth ground via the third prong on the power cord. The power supply converts the higher voltage supplied by the AC Mains to the lower DC voltage required by the rotator, and the earth ground connection is one of the protective safety features associated with the higher voltage portion of the WaveVortex power supply.

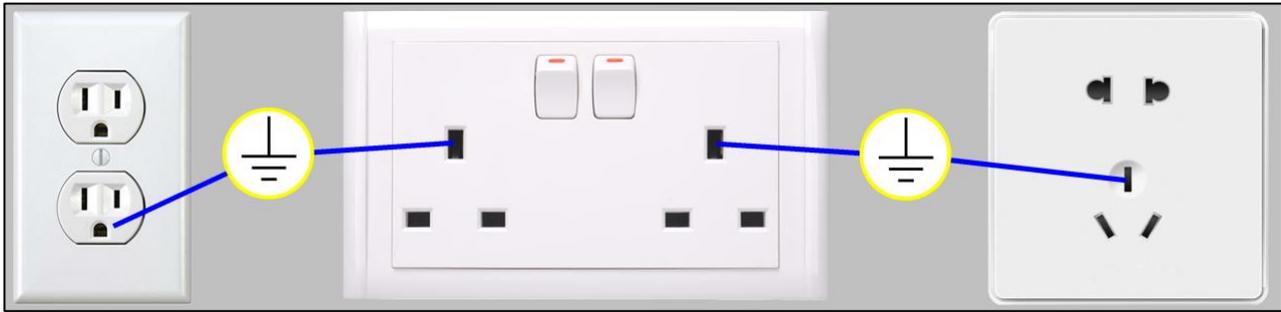


Figure 4-15. Location of Earth Ground on Common Electrical Outlets

The lower DC voltage produced by the power supply is carried to the rotator via a two-conductor cable which does not include a connection to the earth ground. This means that there is no permanent, direct connection between the rotator and the earth ground via the power supply. This arrangement is by design and gives the researcher maximum flexibility in deciding how or whether to make a connection between the experimental apparatus and the earth ground.

In general, a connection to earth ground need only be made if it helps to reduce (or, at least, has no effect on) the amount of noise in the electrochemical signals being measured. In some laboratory environments, connections to earth ground may actually increase the amount of signal noise. Some trial-and-error experimentation may be necessary to decide whether to make a connection to earth ground.

When required for a given experiment, the researcher can choose to make a deliberate connection between the experimental apparatus and the earth ground. Conversely, in those cases where the researcher does not desire to make a connection to earth ground, careful consideration must be given to possible indirect connections to earth ground via other equipment (see: Section 4.12.5).

4.12.2 Chassis Terminal



CHASSIS TERMINAL:

A metal case which surrounds and protects electronic circuitry is called a chassis. A convenient connection point to this chassis is called a chassis terminal.

The WaveVortex control unit is a metal cabinet containing the motor control circuitry. The cabinet helps protect the circuitry from possible damage and shields the circuitry from environmental noise sources. The WaveVortex motor unit has a metal cover around the motor to prevent any electromagnetic interference produced by the motor from reaching the electrochemical cell. The motor cable which connects the control unit to the motor unit has a mesh shield which makes an electrical connection between the control unit cabinet and the metal cover around the motor.

Collectively, the electronics cabinet, the mesh shield in the cable, and the metal cover around the motor are the "chassis" of the WaveVortex rotator. A metal banana jack located on the motor unit provides a convenient connection point to the chassis. In addition, pin #8 on the EXTERNAL I/O PORT provides a connection point to the chassis (see: Figure 4-16).

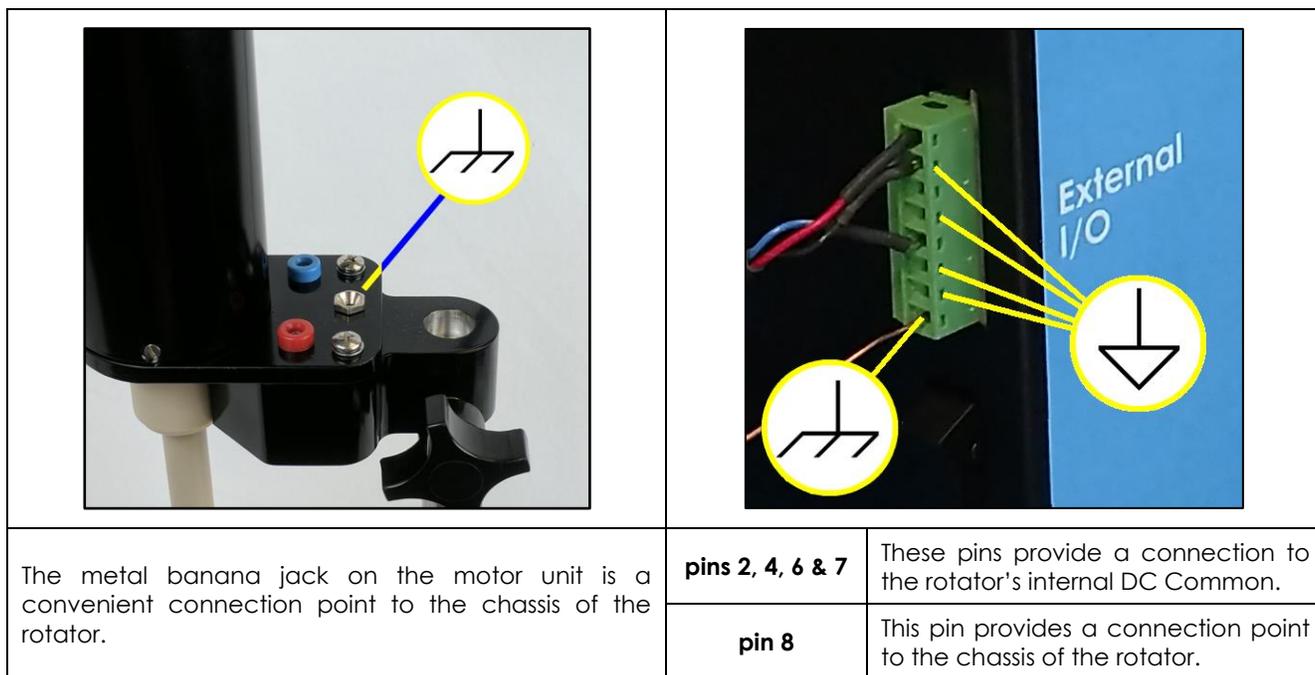


Figure 4-16. Connection Points for the Chassis and DC Common

A rotator is almost always used with a potentiostat, and most potentiostats offer a chassis connection point. It is very common to make a connection between the chassis of the potentiostat and the chassis of the rotator so that the electronic circuitry in both instruments share the same overall protection from environmental noise sources (see: Figure 4-17).

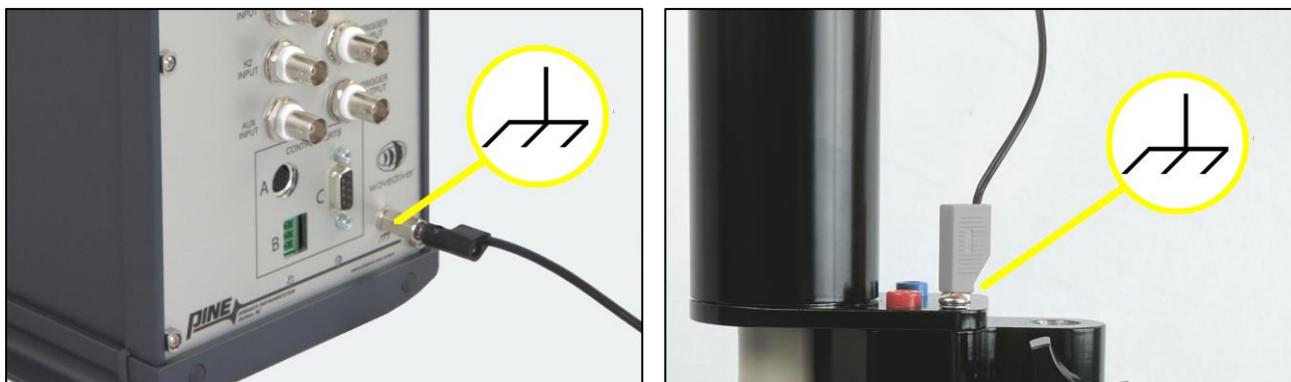


Figure 4-17. Connection between Chassis of Potentiostat and Chassis of Rotator

In some electrochemical experiment configurations, various metal objects are placed near or around the electrochemical cell. Examples include ring stands, three-prong laboratory clamps, and Faraday cages. In most cases, connecting each of these other metal objects to the chassis of the potentiostat (and rotator) can help reduce or eliminate interference from environmental noise sources.

While not a requirement for normal operation, in some circumstances it may be desirable for the potentiostat (and rotator) chassis to be connected to earth ground. A deliberate and direct connection from the chassis to the earth ground may (in some cases) reduce the amount of interference from environmental noise sources. Some experimentation may be required to determine whether a direct connection to earth ground is of any benefit in a particular laboratory environment.

4.12.3 DC Common

**DC COMMON:**

In an analog circuit, like those found in a potentiostat or rotator, the DC Common is the zero reference point against which signal voltages are measured. This point is also known as the analog ground, signal ground, or signal common.

The DC Common for the WaveVortex rotator is the zero volt (0.0 V) reference point used by the motor control circuit. The WaveVortex rotator may send or receive analog signals to and from other electronic instruments such as a waveform generator, an x-y recorder, a digital oscilloscope, or a potentiostat. All of these other instruments also have a DC Common line which represents the common "zero volt" analog signal level.

In general, the act of connecting a signal cable from the WaveVortex to another instrument connects the DC Common lines for each of the instruments together. Such connections are made using the EXTERNAL I/O PORT on the side of the control unit. This connector has several pins which provide connection points to the rotator's internal DC Common (see: Figure 4-16).

The distinction between the DC Common and the instrument chassis is important to maintain and preserve whenever possible. Just like the WaveVortex rotator, many potentiostats offer separate connection points for the chassis terminal and the DC Common because it is often desirable to allow the DC Common to "float" with respect to the chassis. By having separate chassis and DC common connections, it is possible to connect the potentiostat chassis to the rotator chassis (to reduce the effect of environmental noise) while maintaining a separate DC Common connection between the potentiostat and rotator (so that the DC Common continues to float).

Determining whether a floating DC Common is a requirement in a given experimental configuration often requires some trial-and-error experimentation. The act of deliberately shorting the DC Common signal to the chassis may or may not reduce the amount of environmental noise picked up by the potentiostat. In general, the preferred configuration is to allow the DC Common to float, but there may be times when shorting the DC Common to the chassis (or to earth ground) reduces the amount of interference from environmental noise.

4.12.4 Typical Grounding Strategies

While the details of proper grounding in any given instance may differ, a very common strategy is to connect the chassis connections for all of the instruments involved in the experiment together (e.g., potentiostat, rotator, stirrer, heater, etc.). All such connections should be brought together to a single point. In addition, any other metal objects located near the electrochemical cell (e.g., ring stands, Faraday cage, clamps, etc.) should be connected to the same single point. The reason that all connections are brought together to a single point is to avoid creating a grounding loop.

**NOTE:**

A grounding loop is often accidentally created when ground connections are made in series from one instrument to the next. The resulting loop can act as a large antenna which injects environmental noise into sensitive signal measurements.

In general, in an electrochemical experiment, it is ideal to maintain as much isolation between the DC Common, the earth ground, and the instrument chassis as possible. Modern potentiostats are usually designed so that electrode connections (working, counter, and reference) and the DC Common are all able to “float” with respect to the instrument chassis and the earth ground. This floating configuration is considered ideal because it gives the researcher maximum flexibility when working with electrochemical cells that may contain a component which is connected to earth ground or an instrument chassis.

But in almost all cases, an electrochemical cell containing a rotating electrode does not have any electrodes which are connected to earth ground or the instrument chassis. This gives the researcher some additional options for reducing environmental noise by making additional grounding connections. A few examples which may reduce or eliminate environmental noise are listed below:

- Make a deliberate connection from the potentiostat (and rotator) chassis to the earth ground
- Make a deliberate connection between the DC Common of the potentiostat (and rotator) to the instrument chassis and/or the earth ground.
- Wrap the electrochemical cell and/or reference electrode with thick aluminum foil, and then connect the foil to the instrument chassis and/or the earth ground.

As always, some trial-and-error experimentation is usually required to find the best grounding configuration in any given laboratory.

4.12.5 Indirect Grounding Connections

When using a rotator alongside other electronic equipment, it is important to be aware of possible indirect grounding connections. Such indirect (and often hidden) connections may occur (either deliberately or inadvertently) when connecting signal lines from the rotator to other instruments.

A good example of an indirect connection is when a personal computer (desktop or tower) is connected to a potentiostat which is, in turn, connected to the rotator. The chassis of a desktop or tower computer is almost always connected to earth ground via the computer's power supply. When the computer is connected to the potentiostat, the chassis of the potentiostat becomes connected to the chassis of the computer (usually via the shield line of the USB cable which connects the potentiostat to the computer). This causes the chassis of the potentiostat to be (indirectly) connected to earth ground. If the chassis of the rotator is then connected to the chassis of the potentiostat, then the rotator chassis is connected to earth ground (indirectly, via the potentiostat, the USB cable, and the computer).

It is also important for users to be aware of cases where the “floating DC Common” feature is indirectly compromised by a hidden connection. Such a compromise can occur when the rotator, the potentiostat, a computer and/or other instrumentation are interconnected as part of a larger experimental configuration. If any one of the interconnected instruments makes a (possibly hidden or undocumented) connection between the DC Common and the chassis or earth ground, then the potentiostat's “floating DC Common” feature may be inadvertently compromised.

The best way to investigate suspected issues with indirect grounding is to use an ohmmeter. An ohmmeter can easily detect interconnections (whether intentional, hidden, or accidental) between the earth ground, the chassis, and/or the DC Common.

4.13 Using the Rotator in a Glove Box

The rotator may be placed in a glove box when working with air-sensitive or moisture-sensitive compounds. The small form factor of the WaveVortex allows for easy transfer into the glove box through the antechamber. Some minor disassembly outside the glove box and subsequent assembly inside the glove box may be required, depending upon the size of the antechamber.

It is important to understand that the low humidity environment found in most glove boxes increases the rate of wear for the various brush contacts inside the motor unit. Prolonged use of the rotator in a glove box may require more frequent replacement of both the motor and the brushes which contact the rotating shaft. To address the latter case, Pine Research offers special low-humidity brushes (sold separately) for contacting the rotating shaft.



CAUTION:

Using the rotator in a dry environment such as a low humidity glove box will increase the wear rate of various brushes located inside the motor unit.

ATTENTION:

L'utilisation du rotateur dans un environnement sec, tel qu'une boîte à gants à faible taux d'humidité augmente la vitesse d'usure des balais internes du moteur.

4.14 External Rotation Rate Control

Another instrument, such as potentiostat, can control the rotation rate of the WaveVortex rotator. Other instruments can also monitor the actual rotation rate (as measured by the rotator's tachometer) if needed. Connections between the rotator and other instruments are made via the EXTERNAL I/O PORT.

4.14.1 Monitoring the Rotation Rate

When the rotator is in the "run" state, the actual rotation rate measured by the rotator's tachometer is displayed on the front panel. The actual rotation rate can also be monitored using a signal presented at pin #3 on the EXTERNAL I/O PORT. A voltmeter, oscilloscope, or other recording device can monitor the potential difference between pin #3 (RATE MONITOR) and pin #4 (DC Common), and the observed signal is proportional to the rotation rate. The ratio for this output signal is 2 RPM/mV.

4.14.2 External Control of the Rotation Rate

Many potentiostats can provide a signal to control the rotation rate while simultaneously performing electrochemical measurements. A special cable is available to connect Pine Research potentiostats to the WaveVortex Rotator (see: Figure 4-18). One line of this cable carries an analog rate control signal from the potentiostat to pin #1 (RATE CONTROL) on the EXTERNAL I/O PORT. A second line carries a digital control signal from the potentiostat to pin #5 (MOTOR STOP) on the EXTERNAL I/O PORT. The third line connects to the DC Common.

The software which controls a potentiostat usually offers a way to specify the rotation rate to be used during an electrochemical experiment. The researcher simply types the desired rotation rate into the software, and when the experiment is initiated, the potentiostat sends out a voltage signal which is proportional to the specified rotation rate. This output signal is usually presented at a connector on the back panel of the potentiostat, and a special cable is required to connect this signal to the EXTERNAL I/O PORT on the WaveVortex rotator.



When the WaveVortex receives the rate control signal from the potentiostat, the voltage level of this signal determines the rotation rate. The rotation rate is proportional to the voltage. When shipped from the factory, the rotator is initially configured to use a ratio that is compatible with Pine Research potentiostats (1.0 *RPM/mV*). Other ratios are available for use with other potentiostat brands (contact Pine Research for details).

To configure the rotator for external rate control, first put the rotator in the “pause” state. Then, use the ROTATION RATE CONTROL knob to adjust the rotation rate setpoint to zero (0.0 *RPM*). Then, connect the potentiostat to the rotator using the special rate control cable. Finally, put the rotator in the “run” state to allow external control of the rotation rate by the potentiostat’s control software.



NOTE:

External control of the rotation rate may involve a signal connection between a potentiostat from one manufacturer being connected to a rotator from another manufacturer. The signals on these various instruments may have been calibrated to different tolerances by each manufacturer. Small signal level differences within these tolerances can add up. The sum of these tolerances may cause the actual rotation rate, as displayed on the control unit, to differ slightly from the specified rotation rate, as entered by the user of the potentiostat software.



Figure 4-18. Connecting the Rotation Rate Control Cable

4.14.3 External Motor Stop Control

An external digital signal can be applied across pin #5 (MOTOR STOP) and pin #6 (DC Common) on the EXTERNAL I/O PORT. This digital signal can be used by a potentiostat or other external instrument to assure that the rotation rate is actually zero. The logic for this digital signal may be either “active HIGH” or “active LOW”.

When shipped from the factory, the WaveVortex rotator is initially configured to use “active HIGH” logic. If desired, a jumper setting inside the control box can be configured to use the opposite logic (contact Pine Research for details).

If the logic is configured to be “active HIGH”, then the motor is allowed to rotate if a signal greater than 2.0 V is applied to pin #5 (MOTOR STOP) on the EXTERNAL I/O PORT. But if pin #5 is shorted to pin #6 (i.e., if the MOTOR STOP signal is driven to DC Common), then the motor stops rotating.

If the logic is configured to be “active LOW”, then the motor will stop rotating if a signal greater than 2.0 V is applied to pin #5 (MOTOR STOP) on the EXTERNAL I/O PORT. But if pin #5 is shorted to pin #6 (i.e., if the MOTOR STOP signal is driven to DC Common), then the motor is allowed to rotate.



NOTE:

When the control unit is configured for “active HIGH” logic and when no connections are made to the pins on the EXTERNAL I/O PORT, then the motor is allowed to rotate. An internal “pull up” circuit assures that the motor stop signal remains “high” in this case.

When the external motor stop signal causes the motor to stop running, the motor stop indicator on the front panel of the WaveVortex is illuminated (see: Figure 4-11, right).

5. Electrodes

5.1 Electrode Handling Precautions

Rotating electrode tips are precision research tools machined to tight specifications for proper balance while rotating at high rotation rates. When not in use, an electrode tip should be cleaned, dried, and stored in its original case. Special care should be taken not to drop an electrode tip or allow it to roll off a lab bench onto the floor. Damage from such a fall will likely throw the tip off balance.



WARNING:

Do not use or attempt to rotate an electrode tip that has been dropped, bent or otherwise physically damaged. Inspect the electrode tip to be certain that it is not damaged.

AVERTISSEMENT:

N'utilisez pas et ne tentez pas de mettre en rotation un embout d'électrode qui est tombé, a été tordu ou a été endommagé physiquement d'une manière ou d'une autre. Inspectez l'embout d'électrode pour vous assurer qu'il n'a pas été endommagé.



WARNING:

Do not use an electrode shaft which appears to wobble, vibrate, or tilt away from the axis of rotation while rotating. Such a shaft is either improperly installed or physically damaged. Turn off the rotator, disconnect electrical power, and remove the shaft immediately.

AVERTISSEMENT:

N'utilisez pas un arbre d'électrode qui semble osciller, vibrer ou dévier de l'axe de rotation pendant la rotation. Un tel arbre est mal installé ou endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'arbre immédiatement.



WARNING:

Do not use an electrode tip which appears to wobble, vibrate, or tilt away from the axis of rotation while rotating. Such an electrode tip is either improperly installed or physically damaged. Turn off the rotator, disconnect electrical power, and remove the electrode tip immediately.

AVERTISSEMENT:

N'utilisez pas un embout d'électrode qui semble osciller, vibrer ou dévier de l'axe de rotation pendant la rotation. Un tel embout est mal installé ou endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'embout d'électrode immédiatement.

**CAUTION:**

Do not exceed the maximum rotation rate for an electrode tip.

Each type of rotating electrode tip has a specific maximum rotation rate limitation. Consult the documentation for the specific electrode being used in order to learn the maximum rotation rate for that electrode.

ATTENTION:

Ne dépassez pas la vitesse de rotation maximale pour un embout d'électrode.

Chaque type d'embout d'électrode tournante possède une vitesse de rotation maximale spécifique.

Consultez la documentation pour connaître la vitesse de rotation maximale de chaque embout.

**CAUTION:**

Do not apply excessive twisting force to the shroud of an electrode tip when threading it onto the shaft, as this may cause a leak between the shroud and the electrode.

ATTENTION:

N'appliquez pas une force de torsion excessive à l'enveloppe de protection d'un embout d'électrode lorsque vous la vissez sur l'arbre, car cela pourrait provoquer une fuite entre l'enveloppe de protection et l'électrode.

**TEMPERATURE LIMITATIONS:**

Electrode tips with PTFE shrouds are designed for use at room temperature (10°C to 30°C). Exposing these tips to colder or warmer temperatures may compromise the seal provided by the shroud.

Electrode tips with PEEK or PCTFE shrouds are available and are better suited for use at elevated temperatures.

**NOTE:**

After each use of a rotating electrode tip, clean and dry the electrode tip, and then return it to the plastic storage box in which it was originally shipped. Do not place the electrode tip into an oven to dry.

**NOTE:**

A polishing kit is available for use in restoring the electrode surface to its original mirror smooth finish. A slurry of microscopic abrasive particles may be used to routinely repolish the electrode surface (usually at the end of each day). In the event of very serious damage to the electrode surface, it is generally better to return the electrode to Pine Research for professional repolishing.

5.2 RDE Tips (E5 Series with PTFE shroud)



These RDE tips feature a 15 mm OD PTFE shroud around a 5 mm OD disk electrode. These tips fit the WaveVortex shaft and may be used at rotation rates up to 3000 RPM.

Standard disk materials include gold, platinum, and glassy carbon. Additional disk materials are available on a custom basis.

Part numbers for these RDE tips are listed below. If a part number is not listed, contact Pine Research for more details.



CAUTION:

Maximum Rotation Rate: 3000 RPM

Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 TR/MIN

Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

The operating temperature range of this electrode is 10°C to 30°C

Do not use this electrode outside the operating temperature range.

Carbon Materials

glassy carbon
edge-plane pyrolytic graphite
basal-plane pyrolytic graphite

Part Number

AFE5T050GC
AFE5T050GE
AFE5T050GB

Precious Metals

gold (Au)
platinum (Pt)
palladium (Pd)
silver (Ag)

Part Number

AFE5T050AU
AFE5T050PT
AFE5T050PD
AFE5T050AG

Metal Alloys

1018 carbon steel
303 stainless steel
316 stainless steel
316L stainless steel
321 stainless steel
410 stainless steel
430 stainless steel
2205 stainless steel
Zeron 100 stainless steel

Part Number

AFE5T050S1
AFE5T050T1
AFE5T050T3
AFE5T050T6
AFE5T050T9
AFE5T050T4
AFE5T050T5
AFE5T050T7
AFE5T050T8

Common Metals

aluminum (Al)
cobalt (Co)
copper (Cu)
iron (Fe)
niobium (Nb)
nickel (Ni)
lead (Pb)
tin (Sn)
tantalum (Ta)
titanium (Ti)
tungsten (W)
zinc (Zn)

Part Number

AFE5T050AL
AFE5T050CO
AFE5T050CU
AFE5T050FE
AFE5T050NB
AFE5T050NI
AFE5T050PB
AFE5T050SN
AFE5T050TA
AFE5T050TI
AFE5T050W
AFE5T050ZN

5.3 RDE Tips (E5 Series with PEEK shroud)



These RDE tips feature a 15 mm OD PEEK shroud around a 5 mm OD disk electrode. These tips fit the WaveVortex shaft and may be used at rotation rates up to 3000 RPM.

Standard disk materials include gold, platinum, and glassy carbon. Additional disk materials are available on a custom basis.

Part numbers for these RDE tips are listed below. If a part number is not listed, contact Pine Research for more details.



CAUTION:

Maximum Rotation Rate: 3000 RPM. Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 TR/MIN. Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

The operating temperature range of this electrode is 10°C to 80°C. Do not use this electrode outside the operating temperature range.



CHEMICAL INCOMPATIBILITY:

Shrouds fabricated from PEEK may be discolored by exposure to concentrated acids.

Carbon Materials

glassy carbon
edge-plane pyrolytic graphite
basal-plane pyrolytic graphite

Part Number

AFE5T050GCPK
AFE5T050GEPK
AFE5T050GBPK

Precious Metals

gold (Au)
platinum (Pt)
palladium (Pd)
silver (Ag)

Part Number

AFE5T050AUPK
AFE5T050PTPK
AFE5T050PDPK
AFE5T050AGPK

Metal Alloys

1018 carbon steel
303 stainless steel
316 stainless steel
316L stainless steel
321 stainless steel
410 stainless steel
430 stainless steel
2205 stainless steel
Zeron 100 stainless steel

Part Number

AFE5T050S1PK
AFE5T050T1PK
AFE5T050T3PK
AFE5T050T6PK
AFE5T050T9PK
AFE5T050T4PK
AFE5T050T5PK
AFE5T050T7PK
AFE5T050T8PK

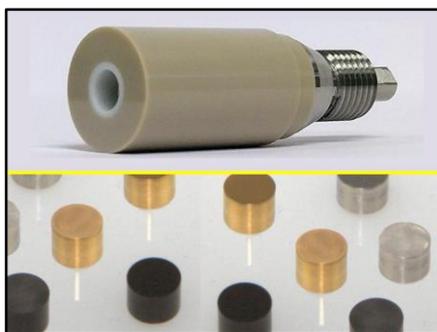
Common Metals

aluminum (Al)
cobalt (Co)
copper (Cu)
iron (Fe)
niobium (Nb)
nickel (Ni)
lead (Pb)
tin (Sn)
tantalum (Ta)
titanium (Ti)
tungsten (W)
zinc (Zn)

Part Number

AFE5T050ALPK
AFE5T050COPK
AFE5T050CUPK
AFE5T050FEPK
AFE5T050NBPK
AFE5T050NIPK
AFE5T050PBPK
AFE5T050SNPK
AFE5T050TAPK
AFE5T050TIPK
AFE5T050WPK
AFE5T050ZNPk

5.4 RDE Tips (ChangeDisk E5TQ Series)



These RDE tips feature a 15 mm OD shroud around a 5 mm OD disk electrode. The disk insert is removable, and a variety of disk insert materials are available separately (see: Section 5.10).

A thin, hydrophobic PTFE seal called a U-Cup surrounds the disk insert when it is installed into the RDE tip. The shroud material may be PEEK or PTFE.

These tips fit the WaveVortex shaft and may be used at rotation rates up to 3000 RPM. Part numbers for these ChangeDisk RDE tips, tools, and consumables are listed below.



CAUTION:

Maximum Rotation Rate: 3000 RPM.

Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 TR/MIN

Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

Consult the description of the electrode tip (see table below) for temperature range.

Do not use this electrode outside the operating temperature range.



CHEMICAL INCOMPATIBILITY:

Shrouds fabricated from PEEK may be discolored by exposure to concentrated acids.

Description

Part Number

ChangeDisk RDE tip (15 mm OD PEEK shroud, 5 mm OD disk, for use from 10°C to 80°C)

AFE5TQ050PK

ChangeDisk RDE tip (15 mm OD PTFE shroud, 5 mm OD disk, for use from 10°C to 30°C)

AFE5TQ050

E5/E6 Series ChangeDisk Toolkit (for removing, installing, and polishing disk inserts)

AFE6K050

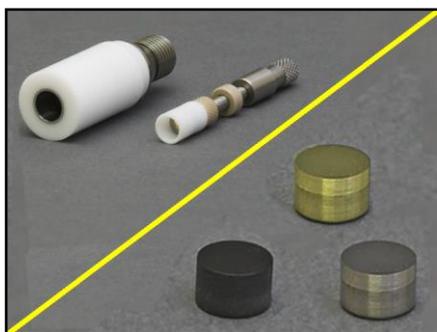
U-Cups (pack of three, for 5 mm OD disk inserts, PTFE)

AKUCUP

These ChangeDisk RDE tips accept 5 mm OD × 4 mm thick disk inserts (sold separately).

Refer to Section 5.10 for a list of common disk inserts.

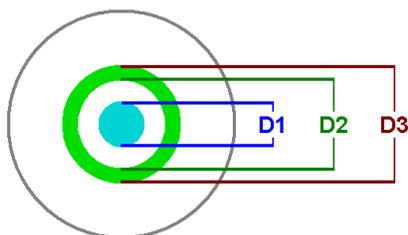
5.5 RRDE Tips (ChangeDisk E6R1 Series with PTFE shroud)



These RRDE tips feature a removable disk electrode surrounded by a ring electrode and a PTFE shroud. The disk insert is sold separately (see: Section 5.10), allowing the same ring electrode to be used with a variety of different disk materials. The seal between the disk and the ring (known as a “U-Cup”) is made from hydrophobic PTFE.

These tips thread directly onto the WaveVortex shaft and may be used at rotation rates up to 3000 *RPM*. Part numbers for these RRDE tips, related tools, and consumables are listed below.

The geometric parameters for this RRDE tip are as follows:



- Disk outer diameter (**D1**): 5.0 mm
- Ring inner diameter (**D2**): 6.5 mm
- Ring outer diameter (**D3**): 7.5 mm
- Shroud outer diameter: 15.0 mm
- Theoretical collection efficiency: 25%



CAUTION:

Maximum Rotation Rate: 3000 *RPM*. Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 TR/MIN. Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

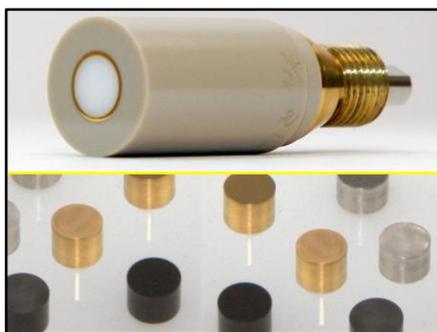
The operating temperature range of this electrode is 10°C to 30°C. Do not use this electrode outside the operating temperature range.

<u>Description</u>	<u>Part Number</u>
ChangeDisk RRDE tip (15 mm OD PTFE shroud, platinum ring, accepts 5 mm OD disks)	AFE6R1PT
ChangeDisk RRDE tip (15 mm OD PTFE shroud, gold ring, accepts 5 mm OD disks)	AFE6R1AU
ChangeDisk RRDE tip (15 mm OD PTFE shroud, glassy carbon ring, accepts 5 mm OD disks)	AFE6R1GC
E5/E6 Series ChangeDisk Toolkit (for removing, installing, and polishing disk inserts)	AFE6K050
U-Cups (pack of three, for 5 mm OD disk inserts, PTFE)	AKUCUP

These ChangeDisk RRDE tips accept 5 mm OD × 4 mm thick disk inserts (sold separately).

Refer to Section 5.10 for a list of common disk inserts.

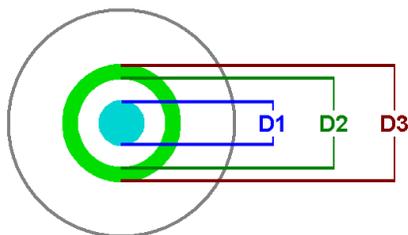
5.6 RRDE Tips (ChangeDisk E6R1 Series with PEEK shroud)



These RRDE tips feature a removable disk electrode surrounded by a ring electrode and a PEEK shroud. The disk insert is sold separately (see: Section 5.10), allowing the same ring electrode to be used with a variety of different disk materials. The seal between the disk and the ring (known as a “U-Cup”) is made from hydrophobic PTFE.

These tips thread directly onto the WaveVortex shaft and may be used at rotation rates up to 3000 *RPM*. Part numbers for these RRDE tips, related tools, and consumables are listed below.

The geometric parameters for this RRDE tip are as follows:



- Disk outer diameter (**D1**): 5.0 mm
- Ring inner diameter (**D2**): 6.5 mm
- Ring outer diameter (**D3**): 7.5 mm
- Shroud outer diameter: 15.0 mm
- Theoretical collection efficiency: 25%



CAUTION:

Maximum Rotation Rate: 3000 *RPM*. Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 *TR/MIN*. Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

The operating temperature range of this electrode is 10°C to 80°C. Do not use this electrode outside the operating temperature range.



CHEMICAL INCOMPATIBILITY:

Shrouds fabricated from PEEK may be discolored by exposure to concentrated acids.

Description

ChangeDisk RRDE tip (15 mm OD PEEK shroud, platinum ring, accepts 5 mm OD disks)

ChangeDisk RRDE tip (15 mm OD PEEK shroud, gold ring, accepts 5 mm OD disks)

ChangeDisk RRDE tip (15 mm OD PEEK shroud, glassy carbon ring, accepts 5 mm OD disks)

E5/E6 Series ChangeDisk Toolkit (for removing, installing, and polishing disk inserts)

U-Cups (pack of three, for 5 mm OD disk inserts, PTFE)

Part Number

AFE6R1PTPK

AFE6R1AUPK

AFE6R1GCPK

AFE6K050

AKUCUP

These ChangeDisk RRDE tips accept 5 mm OD × 4 mm thick disk inserts (sold separately).

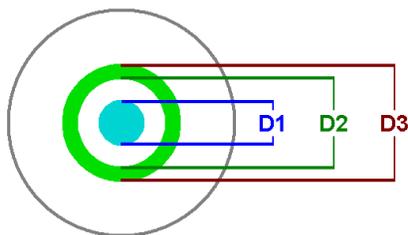
Refer to Section 5.10 for a list of common disk inserts.

5.7 RRDE Tips (E6R2 Series with PEEK shroud and PEEK ring-disk seal)



These RRDE tips feature a permanently mounted disk electrode surrounded by a ring electrode and a PEEK shroud. The seal between the disk and the ring is also made from PEEK. PEEK has excellent high temperature stability.

These tips thread directly onto the WaveVortex shaft and may be used at rotation rates up to 3000 *RPM*. Part numbers for these RRDE tips are listed below.



The geometric parameters for this RRDE tip are as follows:

- Disk outer diameter (**D1**): 5.5 *mm*
- Ring inner diameter (**D2**): 6.5 *mm*
- Ring outer diameter (**D3**): 8.5 *mm*
- Shroud outer diameter: 15.0 *mm*
- Theoretical collection efficiency: 38%



CAUTION:

Maximum Rotation Rate: 3000 *RPM*. Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 TR/MIN. Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

The operating temperature range of this electrode is 10°C to 80°C. Do not use this electrode outside the operating temperature range.



CHEMICAL INCOMPATIBILITY:

Shrouds fabricated from PEEK may be discolored by exposure to concentrated acids.



PEEK vs. PTFE HYDROPHOBICITY

Drop casting films on to a disk electrode surrounded by an insulating polymer is typically easier when the polymer is hydrophobic. PEEK is not as hydrophobic as PTFE.

Description

RRDE tip (glassy carbon disk, platinum ring)
 RRDE tip (gold disk, platinum ring)
 RRDE tip (platinum disk, platinum ring)
 RRDE tip (glassy carbon disk, gold ring)
 RRDE tip (gold disk, gold ring)
 RRDE tip (platinum disk, gold ring)

Part Number

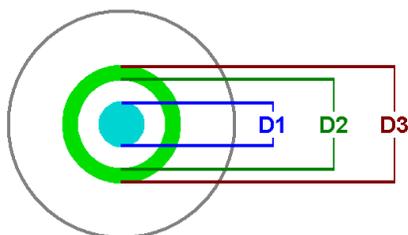
AFE6R2GCPT
 AFE6R2AUPT
 AFE6R2PTPT
 AFE6R2GCAU
 AFE6R2AUAU
 AFE6R2PTAU

5.8 RRDE Tips (ThinGap E7R8 Series with PTFE shroud and PTFE ring-disk seal)



These RRDE tips feature a permanently mounted disk electrode surrounded by a ring electrode and a PTFE shroud. PTFE has excellent chemical resistance and abrades easily when hand-polishing the RRDE tip. The PTFE seal between the disk and the ring is very thin, allowing a short transit time for species travelling from the disk to the ring.

These tips thread directly onto the WaveVortex shaft and may be used at rotation rates up to 3000 *RPM*. Part numbers for these RRDE tips are listed below.



The geometric parameters for this RRDE tip are as follows:

- Disk outer diameter (**D1**): 4.57 mm
- Ring inner diameter (**D2**): 4.93 mm
- Ring outer diameter (**D3**): 5.38 mm
- Shroud outer diameter: 15.0 mm
- Theoretical collection efficiency: 22%



CAUTION:

Maximum Rotation Rate: 3000 *RPM*

Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 *TR/MIN*

Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

The operating temperature range of this electrode is 10°C to 30°C.

Do not use this electrode outside the operating temperature range.

Description

RRDE tip (gold disk, gold ring)

RRDE tip (platinum disk, platinum ring)

Part Number

AFE7R8AUAU

AFE7R8PTPT

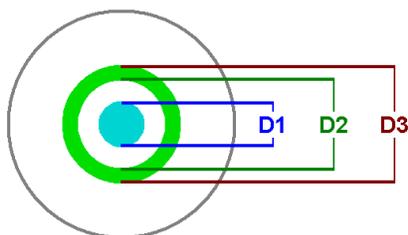
5.9 RRDE Tips (ThinGap E7R9 Series with PTFE shroud and PTFE ring-disk seal)



These RRDE tips feature a permanently mounted disk electrode surrounded by a ring electrode and a PTFE shroud. PTFE has excellent chemical resistance and abrades easily when hand-polishing the RRDE tip. The PTFE seal between the disk and the ring is very thin, allowing a short transit time for species travelling from the disk to the ring.

These tips thread directly onto the WaveVortex shaft and may be used at rotation rates up to 3000 *RPM*. Part numbers for these RRDE tips are listed below.

The geometric parameters for this RRDE tip are as follows:



- Disk outer diameter (**D1**): 5.61 mm
- Ring inner diameter (**D2**): 6.25 mm
- Ring outer diameter (**D3**): 7.92 mm
- Shroud outer diameter: 15.0 mm
- Theoretical collection efficiency: 37%



CAUTION:

Maximum Rotation Rate: 3000 *RPM*. Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximale: 3000 *TR/MIN*. Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximale.



TEMPERATURE LIMITATIONS:

The operating temperature range of this electrode is 10°C to 30°C. Do not use this electrode outside the operating temperature range.

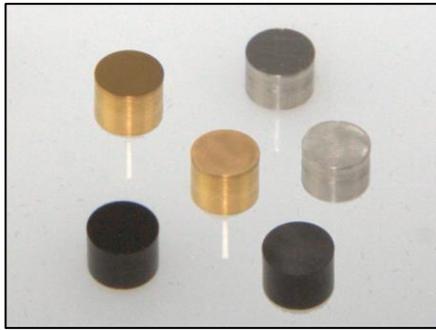
Description

RRDE tip (glassy carbon disk, platinum ring)
 RRDE tip (glassy carbon disk, gold ring)
 RRDE tip (glassy carbon disk, glassy carbon ring)

Part Number

AFE7R9GCPT
 AFE7R9GCAU
 AFE7R9GCGC

5.10 Disk Inserts (for use with ChangeDisk Electrode Tips)



The standard size of a disk insert is 5 mm OD × 4 mm thick. Pine Research offers many types of disk materials, including carbon materials, precious metals, steels and alloys, and other common metals. Most disk inserts are shipped from the production facility with a mirror polish.

Part numbers for standard disk inserts are listed below. Disk inserts made from custom materials or with custom dimensions and finish are also available. Contact Pine Research for pricing and lead times.

<u>Carbon Materials</u>	<u>Part Number</u>	<u>Precious Metals</u>	<u>Part Number</u>
glassy carbon	AFED050P040GC	gold (Au)	AFED050P040AU
edge-plane pyrolytic graphite	AFED050P040GE	platinum (Pt)	AFED050P040PT
basal-plane pyrolytic graphite	AFED050P040GB	palladium (Pd)	AFED050P040PD
		silver (Ag)	AFED050P040AG
<u>Metal Alloys</u>	<u>Part Number</u>	<u>Common Metals</u>	<u>Part Number</u>
1018 carbon steel	AFED050P040S1	aluminum (Al)	AFED050P040AL
303 stainless steel	AFED050P040T1	cobalt (Co)	AFED050P040CO
304 stainless steel	AFED050P040T2	copper (Cu)	AFED050P040CU
316 stainless steel	AFED050P040T3	iron (Fe)	AFED050P040FE
316L stainless steel	AFED050P040T6	niobium (Nb)	AFED050P040NB
NI321 stainless steel	AFED050P040T9	nickel (Ni)	AFED050P040NI
410 stainless steel	AFED050P040T4	lead (Pb)	AFED050P040PB
430 stainless steel	AFED050P040T5	tin (Sn)	AFED050P040SN
2205 stainless steel	AFED050P040T7	tantalum (Ta)	AFED050P040TA
Zeron 100 stainless steel	AFED050P040T8	titanium (Ti)	AFED050P040TI
brass	AFED050P040BR	tungsten (W)	AFED050P040WU
		zinc (Zn)	AFED050P040ZN

6. Parts and Accessories

6.1 Mechanical Parts and Hardware

There are several moving parts on the rotator which are subject to normal wear during routine use. This section describes these parts in more detail.



Replacement Brushes

Brushes should be ordered and replaced in pairs. Special low-humidity brushes are available for use in dry environments such as inside a glove box.

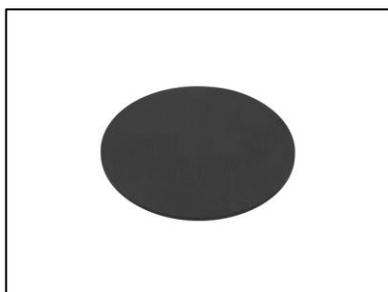
Description	Part Number
One Standard Brush	AC01WV10S05
One Low-Humidity Brush	AC01WV10S05LH



Gas-Purged Bearing Assembly

This assembly may be used to provide a partial seal to help protect the contents of the electrochemical cell from the lab atmosphere.

Description	Part Number
Bearing Assembly	AC01TPA6M



Friction Mat

The friction mat is placed underneath the electrochemical cell to help prevent the cell from sliding on the enclosure base.

Description	Part Number
Friction Mat	KAAS400V



Motor Control Cable

The motor control cable is 91 cm long and has a male DB-9 connector on one end and a female DB-9 connector on the other end.

Description	Part Number
Motor Control Cable	EWCP09DMFL



WaveVortex Maintenance Toolkit

This toolkit contains the hex drivers and screwdrivers needed for repair and maintenance of the WaveVortex rotator.

Description

WaveVortex Toolkit

Part Number

AK01WV10TK1



WaveVortex Rotating Shaft

This shaft is for use with the WaveVortex rotator only. It is compatible with various Pine Research RDE and RRDE tips (see: Section 5).

Description

WaveVortex Shaft

Part Number

AC01WV10S07



WaveVortex Bearing Assembly

The rotating shaft passes through this bearing assembly and the brushes are mounted in the side wall of this assembly.

Description

WaveVortex Bearing Assembly

Part Number

AC01WV10M12



WaveVortex Power Supply

This 24 VDC power supply for the WaveVortex rotator requires 100 – 240 VAC, $\pm 10\%$; 2 A; 50/60 Hz input power. A wide variety of international power cords (sold separately) may be used with this supply.

Description

WaveVortex 24 VDC Supply

Part Number

EE24V17A

6.2 Power Cords

The AC power entry on the power supply accepts a power cord with a standard C13 connector (see: Figure 6-1). Pine Research offers a variety of international power cords, each rated at 10 A (minimum), which terminate at a standard C13 connector (see: Table 6-1).



Figure 6-1. Standard C13 Connector (on Power Cord) and C14 Connection (on Power Supply)

Table 6-1. Power Cord Options

 <p>This cord is for use in the USA, Canada, Mexico, Brazil, Columbia, Mexico, Saudi Arabia, and Taiwan.</p> <p>Description Power Cord (USA)</p> <p>Part Number EWM18B7</p>	 <p>This cord is for use in continental Europe, Korea, Russia, and Indonesia.</p> <p>Description Power Cord (Europe)</p> <p>Part Number EWM18B8EU</p>
 <p>This cord is for use in the United Kingdom, Ireland, Kuwait, Malaysia, Oman, Hong Kong, and Singapore.</p> <p>Description Power Cord (UK)</p> <p>Part Number EWM18B8UK</p>	 <p>This cord is for use exclusively in China.</p> <p>Description Power Cord (China)</p> <p>Part Number EWM18B8CN</p>

(table is continued on next page)



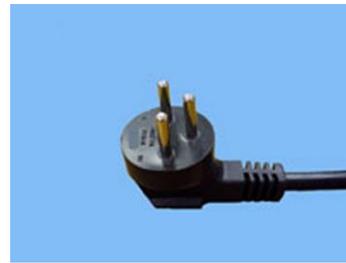
This cord is for use in India and South Africa.

Description

Power Cord (South Africa)

Part Number

EWM18B8IN



This cord is for use exclusively in Israel.

Description

Power Cord (Israel)

Part Number

EWM18B8IL



This cord is for use in Japan.

Description

Power Cord (Japan)

Part Number

EWM18B8JP



This cord is for use in Argentina.

Description

Power Cord (Argentina)

Part Number

EWM18B8AR



This cord is for use in Denmark.

Description

Power Cord (Denmark)

Part Number

EWM18B8DK



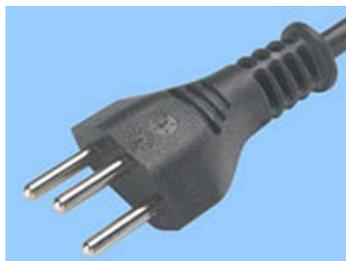
This cord is for use in Australia and New Zealand.

Description

Power Cord (Australia)

Part Number

EWM18B8NZ



This cord is for exclusively in Switzerland.

Description

Power Cord (Switzerland)

Part Number

EWM18B8CH



This cord is for use exclusively in Italy

Description

Power Cord (Italy)

Part Number

EWM18B8IT

7. Troubleshooting

This section describes some basic troubleshooting considerations when working with a rotator. If problems with the rotator persist, contact Pine Research for further assistance (see: Section 1.6).

Problem/Issue	Corrective Action
No Rotation	<ul style="list-style-type: none"> • Confirm that the unit is connected to a live power outlet. • Confirm that the power switch is in the "on" position. • Check the motor control cable which connects the control unit to the motor unit. The connectors at both ends of this cable must be secured using the two screws on each connector. • The rotation rate knob may be set to full counterclockwise position. If this is the case, then rotate the knob clockwise to increase the rotation rate. • The motor, the shaft or one of the bearings may be frozen due to corrosion, or one of the boards or cables may be loose. Turn off the rotator, disconnect power, and then try the following procedure: <div data-bbox="605 865 1468 1381" style="border: 1px solid black; padding: 10px; margin-top: 10px;"> <div data-bbox="626 884 708 961" style="float: left; text-align: center;">  </div> <div data-bbox="743 884 1398 995" style="clear: both;"> <p>WARNING: Risk of electric shock. Disconnect all power before servicing the rotator.</p> </div> <div data-bbox="743 1016 1425 1157" style="clear: both;"> <p>AVERTISSEMENT: <i>Risque de décharge électrique. Déconnectez toutes les sources d'alimentation avant de procéder à l'entretien du rotateur.</i></p> </div> <ul style="list-style-type: none"> • Check for freedom of rotation of the shaft by manually attempting to rotate the shaft. • Look inside the control unit and confirm that the printed circuit board and all connector cables are securely mounted. </div>
Continuous Rotation at a High Rate	<ul style="list-style-type: none"> • Check the motor control cable which connects the control unit to the motor unit. The connectors at both ends of this cable must be secured using the two screws on each connector. • Faulty connection or wire – contact Pine Research. • Faulty circuitry – contact Pine Research.

Problem/Issue	Suggested Cause or Action
Rotator Spins Backwards	<ul style="list-style-type: none">• When the rotation rate knob is in the full counterclockwise position, there may be a small residual rotation rate, and often this is in the reverse direction. This behavior is normal.• If a negative voltage signal is applied to the RATE CONTROL input on the EXTERNAL I/O PORT, then the rotator will spin backwards. If this is undesirable, reverse the polarity of the applied signal.
Excessive Audible Noise	<ul style="list-style-type: none">• The bearing inside the bearing assembly may be corroded. If corroded, replace the entire bearing assembly.
Electrical Noise in Electrochemical Response (environmental)	<ul style="list-style-type: none">• Make sure that working, reference, and counter electrode cables do not cross or travel near power cords, video cables, or network lines.• Make sure that the potentiostat and rotator are located as far as possible from hotplates, ovens, video monitors, computers, network hubs, wireless devices, or cellular telephones.
Electrical Noise in Electrochemical Response (grounding issues)	<ul style="list-style-type: none">• Refer to Section 4.12 for detailed information on grounding.
Electrical Noise in Electrochemical Response (brush wear)	<ul style="list-style-type: none">• After being used for several hours, the internal brushes which contact the rotating shaft should have a concave groove worn in them which exactly mates with the rotating shaft. The depth of this concave groove naturally increases over the lifetime of the brush. Eventually, the brush wears out and must be replaced.
Electrical Noise in Electrochemical Response (cell connections)	<ul style="list-style-type: none">• Confirm that the reference electrode has low impedance and is in good contact with the main test solution. High impedance at the reference electrode is often caused by a plugged frit, which impedes current between the inner chamber of the reference electrode and the main test solution. High impedance may also be encountered when working with low dielectric media, such as non-aqueous solvents.• Use working, reference, and counter electrode cables which are shielded (coaxial) cables.• Confirm that any alligator clips being used for connection to the electrodes are not corroded and are securely fastened to the electrodes.

8. Storage and Shipment

In the event that the rotator system is not going to be used for a long period of time, it should be stored in the original packaging material to prevent damage. It should be stored at temperatures between -17°C and 37°C, and at humidity levels less than 95% non-condensing.

Retain the original packing materials for future use. These packing materials were designed to provide both protection in shipment, and to minimum size and weight for efficient shipment.

9. Theory

9.1 Forced Convection

The current signal recorded during an electrochemical experiment is easily influenced or disturbed by the convection of various molecules and ions due to bulk movement of the solution. Proper interpretation of the current signal must accurately account for any contributions (desired or undesired) from solution convection. Thus, the control of solution movement is a critical part of any electrochemical experiment design, and the issue of convection cannot be ignored. Two opposing approaches are typically used to address the convection issue. At one extreme, an experiment can be conducted in a quiescent solution, so that convection makes little or no contribution to the observed current. The opposite extreme involves forced convection, where the solution is actively stirred or pumped in a controlled manner.

At first glance, it may seem that the simplest and most obvious way to account for convection is to try to eliminate it entirely by using a quiescent (non-moving) solution. This is the approach used in many popular electroanalytical techniques¹ (including cyclic voltammetry, chronoamperometry, square wave voltammetry, and differential pulse voltammetry). The timescale for these methods is generally less than 30 seconds, and on such short timescales, the influence of convection in an unstirred solution is generally negligible. On longer timescales, however, even unstirred solutions are prone to convective interference from thermal gradients and subtle environmental vibrations.

For long duration (steady-state) experiments, convection is unavoidable, so actively forcing² the solution to move in a well-defined and controlled manner is the preferred approach. An entire family of electroanalytical methods (broadly categorized as hydrodynamic voltammetry) couples precise control of solution flow with rigorous mathematical models defining the flow. Some of the many examples of hydrodynamic voltammetry include placing an electrode in a flow cell,³ firing a jet of solution at an electrode target,^{4,5} embedding an electrode in a microfluidic channel,⁶ vibrating a wire-shaped electrode,⁷ subjecting the solution to ultrasonication,⁸ and rotating the electrode.^{2,9-14}

By far the most popular and widely used hydrodynamic methods are those that involve a rotating electrode. The rotating electrode geometries most amenable to mathematical modeling are the rotating disk electrode (RDE),⁹⁻¹⁴ the rotating ring-disk electrode (RRDE),¹⁵⁻²⁶ and the rotating cylinder electrode (RCE).²⁷⁻³² Researchers take advantage of the steady-state laminar flow conditions adjacent to an RDE or RRDE to carefully gather information about electrode reaction kinetics.^{13,14,21,26,33-43} In contrast, the relatively chaotic and turbulent conditions adjacent to an RCE are exploited by corrosion scientists⁴⁴⁻⁶⁹ wishing to mimic flow-induced pipeline corrosion conditions in the laboratory. Development of the RDE and RRDE as routine analytical tools has largely been carried out by the community of academic electroanalytical chemists, while the RCE has primarily been a tool used by the corrosion and electroplating industries.

9.2 Half Reactions

Regardless of the rotating electrode geometry being used, the common theme is that an ion or molecule is being conveyed to the electrode surface, and upon arrival, it is either oxidized or reduced depending upon the potential applied to the rotating electrode. If a sufficiently positive potential is applied to the electrode, then the molecules (or ions) tend to be oxidized, and conversely, if a sufficiently negative potential is applied to the rotating electrode, the molecules (or ions) tend to be reduced.

Reduction at a rotating electrode implies that electrons are being added to the ion or molecule by flowing out of the electrode and into the solution. A current travelling in this direction is said to be a

cathodic current. The general form of a reduction half-reaction occurring at an electrode may be written as follows:



where n is the total number of electrons added to the molecule (or ion) when it is converted from the oxidized form (O) to the reduced form (R).

Oxidation at a rotating electrode implies that electrons are being removed from an ion or molecule and are travelling out of the solution and into the electrode. A current travelling in this direction is said to be an anodic current, and the oxidation occurring at the electrode can be represented by the following redox half reaction,



Given that electrochemical half reactions can occur in either direction, they are often written using chemical equilibrium notation* as follows:



Each half reaction has an associated standard electrode potential (E°) which is a thermodynamic quantity related to the free energy associated with the equilibrium. Like many other standard thermodynamic quantities, the standard electrode potential corresponds to a given standard state. The standard state corresponds to a thermodynamic system where the activities of O and R are unity (i.e., when all solution concentrations are 1.0 mol/L, all gases are present at 1.0 atm partial pressure, and other materials are present as pure phases with unity activity).

To account for the (likely) possibility of non-unity activities, the Nernst equation (see below) can be used to express the equilibrium electrode potential ($E_{NERNSTIAN}$) in terms of the actual activities,

$$E_{NERNSTIAN} = E^{\circ} + \left(\frac{R T}{n F} \right) \ln \left(\frac{a_O}{a_R} \right) \quad (4)$$

where T is the temperature (K), F is the Faraday constant ($F = 96485 \text{ C / mol}$), and R is the ideal gas constant ($R = 8.3145 \text{ J / mol K}$). Usually, the activities of molecules or ions dissolved in solution are assumed to be the same as their molar concentrations, so the Nernst Equation is often written as

$$E_{NERNSTIAN} = E^{\circ} + \left(\frac{R T}{n F} \right) \ln \left(\frac{C_O}{C_R} \right) \quad (5)$$

* By convention, redox half reactions are generally tabulated in textbooks and other reference works as reduction reactions (with the oxidized form on the left side and the reduced form on the right side, as shown above), but it is understood that the reaction may occur in either direction depending upon the potential applied to the electrode.

where C_O and C_R are the concentrations of the dissolved molecules or ions in the oxidized and reduced forms, respectively, at the surface of the electrode. Note that any liquid or solid phase materials at the electrode surface (such as the solvent or the electrode itself) have unity activity and thus do not appear in the Nernst equation.

This half reaction at an electrode can be driven in the cathodic (reducing) direction by applying a potential to the electrode ($E_{APPLIED}$) which is more negative than the equilibrium electrode potential ($E_{APPLIED} < E_{NERNSTIAN}$). The half reaction can be driven in the oxidizing (anodic) direction by applying a potential more positive than the equilibrium electrode potential ($E_{APPLIED} > E_{NERNSTIAN}$).

9.3 Voltammetry

The term voltammetry refers broadly to any method where the electrode potential is varied while the current is measured.^{1,2} The terminology associated with voltammetry varies across different industries and academic disciplines, but the underlying principles of all voltammetric techniques are very similar.

The most common form of voltammetry involves sweeping the electrode potential from an initial value to a final value at a constant rate. When working in the context of electroanalytical chemistry with a non-rotating electrode, this technique is called linear sweep voltammetry (LSV). In the context of corrosion science, this kind of technique is usually called linear polarization resistance (LPR) or a Tafel analysis. The term cyclic voltammetry (CV) refers to a method where the electrode potential is swept repeatedly back and forth between two extremes.

When working with a rotating electrode, it is common to further specify the kind of electrode being used as part of the technique name, such as rotating disk voltammetry, rotating ring-disk voltammetry, or rotating cylinder voltammetry. In each of these techniques, the rotation rate is held constant as the electrode is swept from one potential to another potential at a constant sweep rate. In electroanalytical chemistry, the potential sweep usually spans at least 200 mV on either side of the standard electrode potential, and rotation rates are usually between 100 RPM and 2400 RPM. However, in the context of a corrosion study, the potential sweep may span a much narrower range (50 mV) using a slower sweep rate (less than 5 mV/sec) with an emphasis on higher rotation rates.

As an example, consider a solution that initially contains only the oxidized form of a molecule or ion. A rotating electrode is placed in this solution and is initially poised at a potential that is 200 mV more positive than the standard potential. At this potential, there is little or no current because there is nothing to oxidize (the molecule or ion is already oxidized), and the potential is not (yet) negative enough to cause any appreciable reduction of the molecule or ion.

Next, the electrode potential is slowly (20 mV/sec) swept in the negative (cathodic) direction (see: Figure 9-1, left). As the applied potential approaches the standard electrode potential, a cathodic current is observed (see: Figure 9-1, right). The cathodic current continues to increase as the potential moves past the standard electrode potential towards more negative potentials.

The current eventually reaches a maximum value (limiting current) once the applied potential is sufficiently negative relative to the standard electrode potential. At such a negative potential, any oxidized form of the molecule or ion (O) that reaches the surface of the electrode is immediately converted to the reduced form (R) as shown below.



The observed cathodic current is the result of electrons flowing out of the electrode and into the solution. The rate of electron flow is limited only by how fast the oxidized form (O) can arrive at the electrode surface. The maximum current observed in this circumstance is called the cathodic limiting current (i_{LC}).

Whenever an observed current is limited only by the rate at which material arrives at the electrode surface, the current is said to be mass transport limited. When working with a rotating electrode, the rate of mass transport is related to the rotation rate of the electrode. Rotating the electrode at a faster rate increases the rate at which material arrives at the electrode surface. Thus, the limiting current increases with increasing rotation rate. Experiments involving a rotating electrode are designed to purposefully exploit this fundamental relationship between the rotation rate and the limiting current.

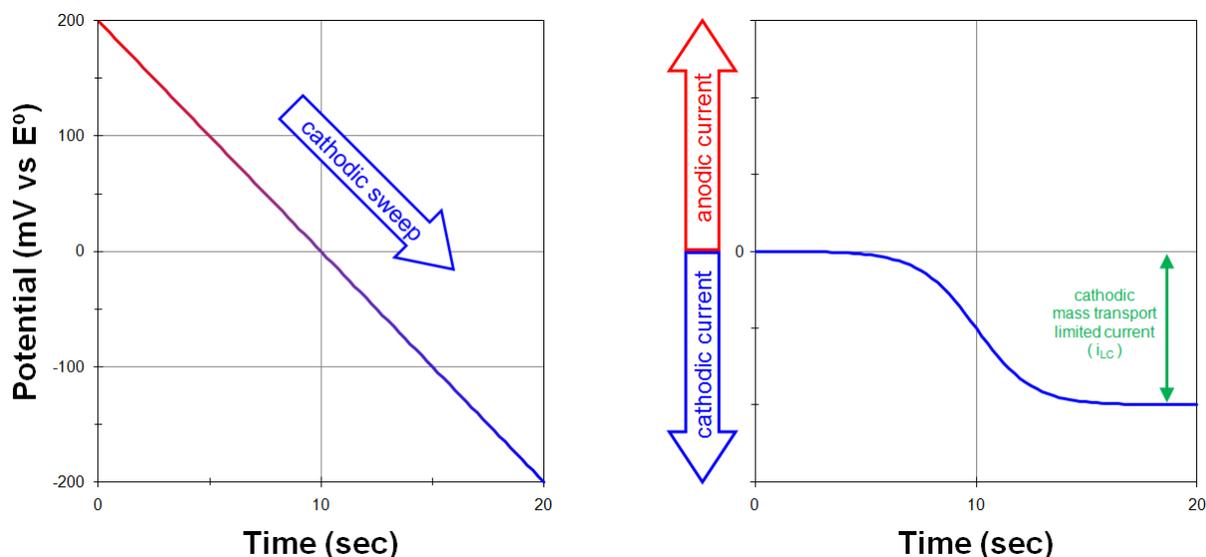


Figure 9-1. Response to a Potential Sweep (Cathodic) from a Solution Initially Containing only the Oxidized Form (O) with no Reduced Form (R)

The cathodic sweep experiment described above (see: Figure 9-1) applies to the case where the solution initially contains only the oxidized form (O) of the molecule or ion being studied. The opposite case yields similar results. Consider a solution that initially contains only the reduced form (R) of the molecule or ion being studied. The rotating electrode is initially poised at a potential that is about 200 mV more negative than the standard potential. At this potential, there is little or no current because there is nothing to reduce (the molecule or ion is already reduced), and the potential is not (yet) positive enough to cause any appreciable oxidation of the molecule or ion.

Next, the electrode potential is slowly swept in the positive (anodic) direction (see: Figure 9-2, left) and an anodic current is observed (see: Figure 9-2, right). The anodic current eventually reaches a maximum value when the potential is sufficiently positive relative to the standard electrode potential. At this point, any of the reduced form (R) that reaches the electrode surface is immediately converted to the oxidized form (O).



The observed current is the result of electrons flowing into the electrode. The maximum current observed is called the anodic limiting current (i_{LA}).

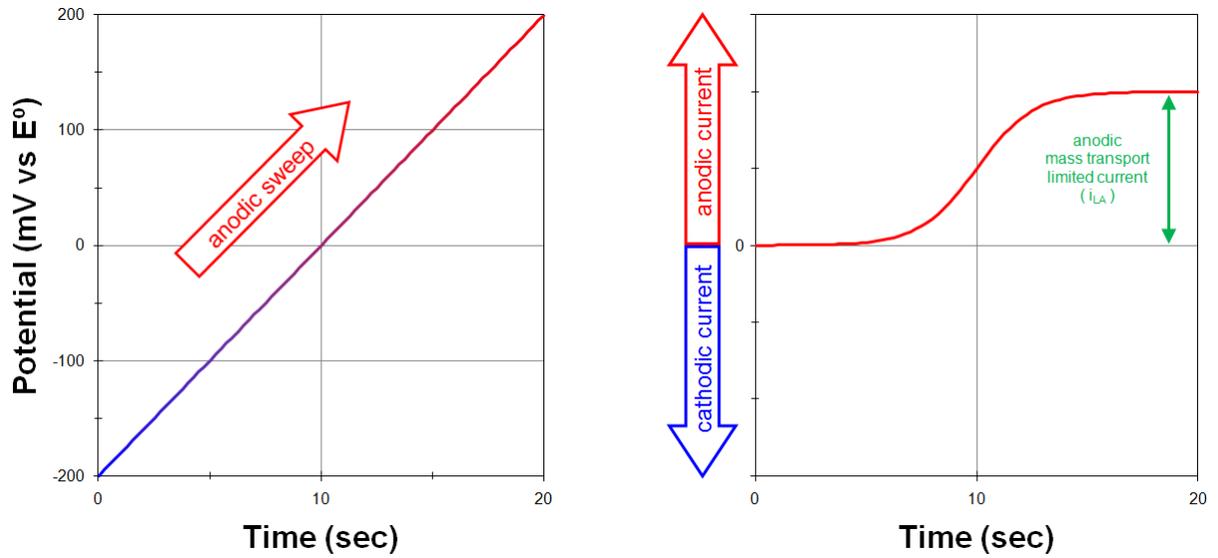


Figure 9-2. Response to a Potential Sweep (Anodic) from a Solution Initially Containing only the Reduced Form (R) with no Oxidized Form (O)

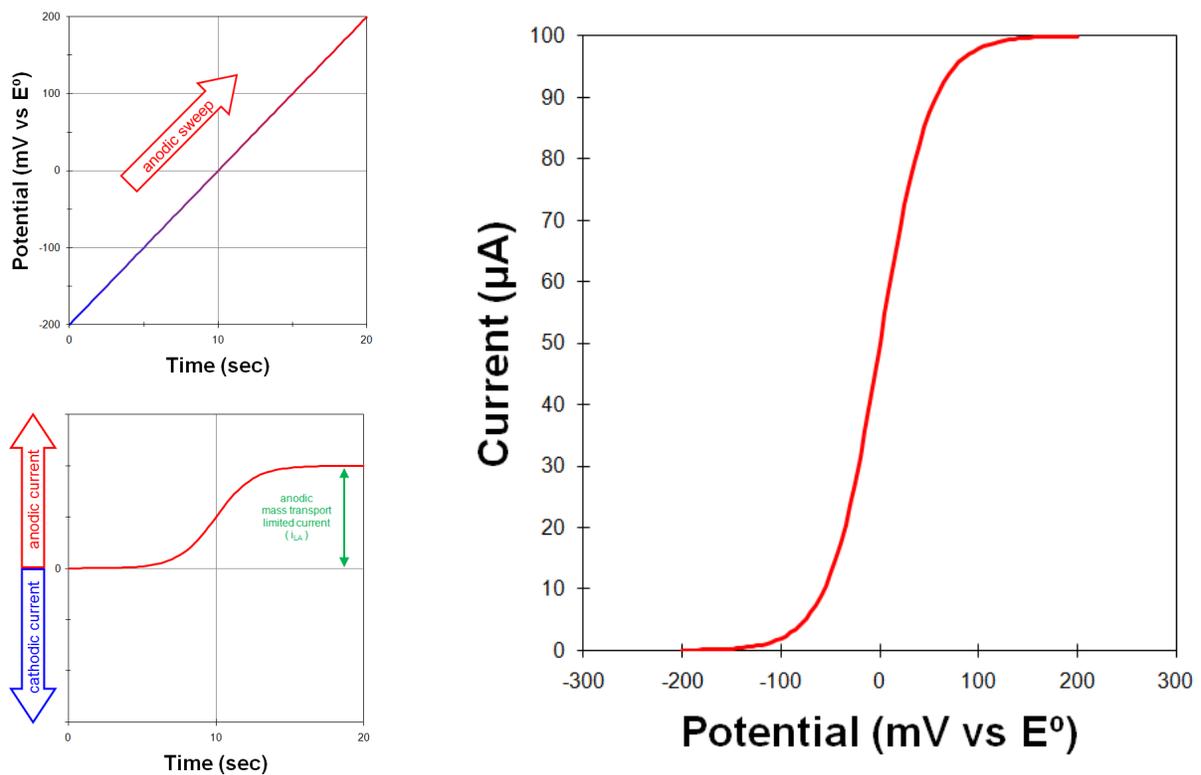


Figure 9-3. A Voltammogram is a Plot of Current versus Potential

9.3.1 Voltammogram Plotting Conventions

The two streams of data recorded during a voltammetry experiment are the potential vs. time and the current vs. time. Rather than plot these two streams separately (as shown in Figure 9-3, left), it is more common to plot current vs. potential (as shown in Figure 9-3, right). Such a plot is called a voltammogram.

Although most electroanalytical researchers agree that current should be plotted along the vertical axis and potential should be plotted along the horizontal axis, there is no widespread agreement as to the orientation (direction) for each axis. Some researchers plot positive (anodic, oxidizing) potentials toward the right while others plot negative (cathodic, reducing) potentials toward the right (as per classical polarography tradition). Furthermore, some researchers plot anodic (oxidizing) current upward along the vertical axis, while others plot cathodic (reducing) current in the upward direction.

This means there are four possible conventions for plotting a voltammogram, and one should always take a moment to ascertain the orientation of the axes before interpreting a voltammogram. Fortunately, of the four possible ways to plot a voltammogram, only two are commonly used. The older tradition (based on classical polarography) plots cathodic current upwards along the vertical axis and negative (cathodic, reducing) potentials toward the right along the horizontal axis. A complex voltammogram involving four different limiting currents (see: Figure 9-4, left) illustrates this convention, which is sometimes called the “North American” convention.

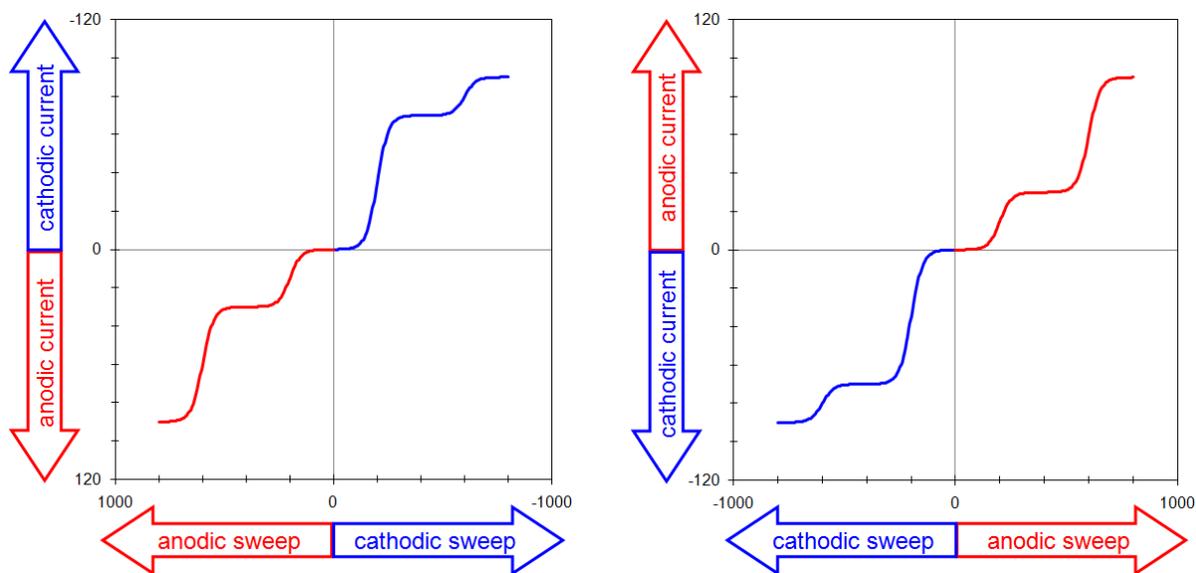


Figure 9-4. Two Popular Voltammogram Plotting Conventions

The same data may be plotted using the “European” convention (see: Figure 9-4, right). This convention plots anodic currents upward along the vertical axis and more positive (anodic, oxidizing) potentials to the right along the horizontal axis. The European convention is more readily understood by those outside the electroanalytical research community (because positive values are plotted to the right along the horizontal axis).

The European convention is used throughout the remainder of this document. Note that this choice also implies a mathematical sign convention for the current. Specifically, positive current values are considered anodic, and negative current values are considered cathodic in this document. This sign

convention is somewhat arbitrary, and electrochemical data processing software available from various manufacturers may or may not use this sign convention.

9.3.2 Measuring Limiting Currents

The theoretical voltammetric response from a rotating electrode is a symmetric sigmoid-shaped wave (like the ideal voltammograms shown in Figure 9-3 and Figure 9-4). A perfect sigmoid has a flat baseline current before the wave and a flat limiting current plateau after the wave. The height of the wave (as measured from the baseline current to the limiting current plateau) is the mass-transport limited current.

In actual “real world” experiments, the wave may be observed on top of a background current, and furthermore, the background current may be slightly sloped (see: Figure 9-5). This (undesired) background current may be due to interference from oxidation or reduction of impurities or of the solvent itself. The background current may also be due to capacitive charging and discharging of the ionic double-layer that forms next to the polarized electrode surface.

When attempting to measure the (desired) Faradaic mass-transport limited current at a rotating electrode, it is often necessary to account for the (undesired, possibly sloping) background current. If the background current has a constant slope across the entire voltammogram, then it is fairly easy to extrapolate the sloping baseline to a point underneath the limiting current plateau (see: Figure 9-5, left). The limiting current is measured as the (vertical) distance between the plateau and the extrapolated baseline. In voltammograms where there is more than one wave, the plateau for the first wave is used as the baseline for the second wave (see i_{LA2} in Figure 9-5, left).

In some cases, the slope of the background current is not constant across the entire voltammogram. That is, the slope of the baseline leading up to the wave can be different than the slope of the plateau after the wave. It can be very difficult to discern exactly where to measure the limiting current along such a voltammogram. One approach is to extrapolate the baseline forward through the wave and to also extrapolate the plateau backward through the wave. Then, the limiting current is measured as the vertical distance between the baseline and plateau at a point corresponding to the center of the voltammogram (see i_{LA} in Figure 9-5, right).

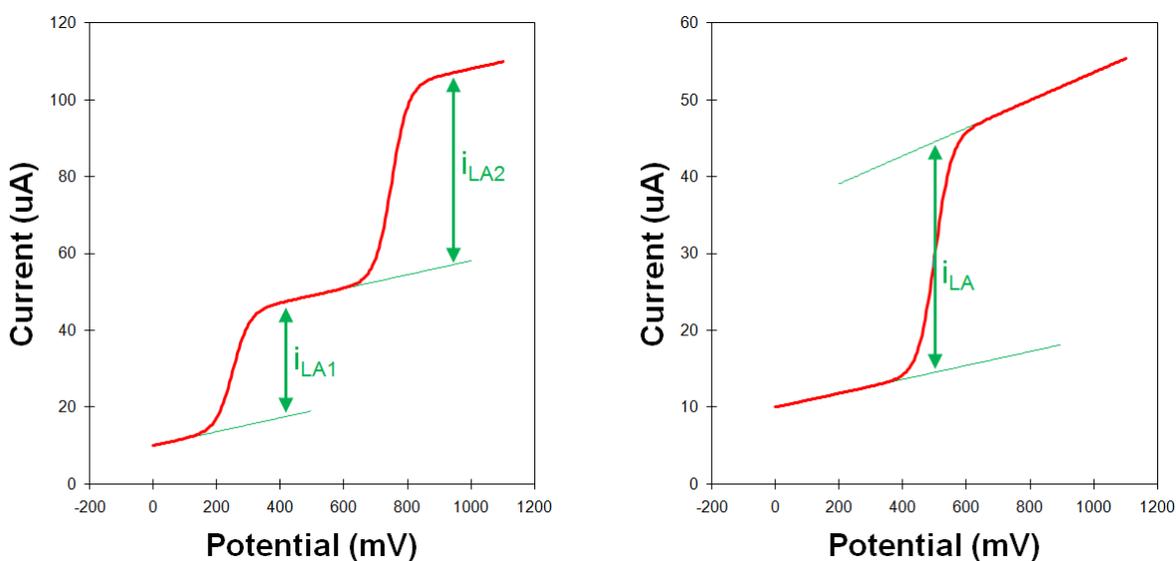


Figure 9-5. Sloping Backgrounds in Voltammograms

Finally, it should be noted that when the oxidized form (O) and the reduced form (R) of a molecule or ion are both present in a solution at the same time, the voltammogram is likely to exhibit both a cathodic and an anodic limiting current (see: Figure 9-6). It can be very difficult to measure the limiting current properly in this case, especially if there is also a sloping background current. For this reason, most experiments with rotating electrodes are conducted in solutions where only one form of the molecule or ion is initially present.

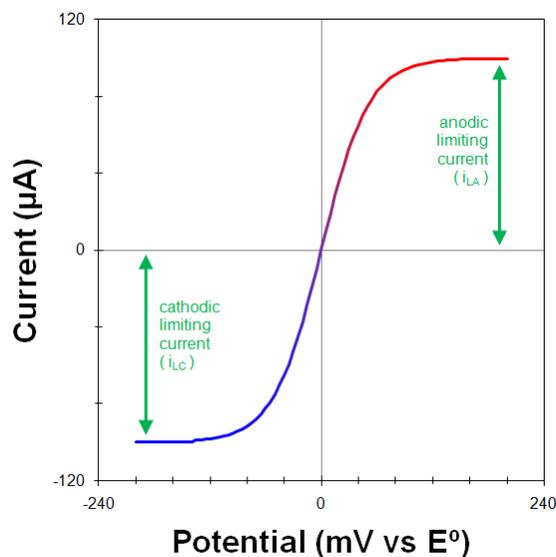


Figure 9-6. Voltammogram for a Solution Containing Both O and R

9.4 Rotating Disk Electrode (RDE) Theory

The general theory describing mass transport at a rotating disk electrode (RDE) was developed by Benjamin Levich at the Institute of Electrochemistry at the Academy of Sciences of the USSR. Levich described the theory in his landmark book, *Physicochemical Hydrodynamics*, originally published in Russian in 1952. Ten years later, Levich's book was translated⁹ from Russian to English, and the RDE became more widely known¹¹ to Western researchers. In the early 1960's, Stanley Bruckenstein¹⁰ at the University of Minnesota (and his students Dennis Johnson and Duane Napp) and Ronnie Bell¹² at Oxford University (and his student John Albery) began working with rotating electrodes. Subsequent generations of researchers expanded on this initial work, and the rotating disk electrode has since grown into a mature tool for probing electrochemical reaction kinetics.¹³

The laminar flow at a rotating disk electrode conveys a steady stream of material from the bulk solution to the electrode surface. While the bulk solution far away from the electrode remains well-stirred by the convection induced by rotation, the portion of the solution nearer to the electrode surface tends to rotate with the electrode. Thus, if the solution is viewed from the frame of reference of the rotating electrode surface, then the solution appears relatively stagnant. This relatively stagnant layer is known as the hydrodynamic boundary layer, and its thickness (δ_H) can be approximated,

$$\delta_H = 3.6 (v/\omega)^{1/2} \quad (8)$$

in terms of the kinematic viscosity of the solution (ν) and the angular rotation rate ($\omega = 2\pi f / 60$, where f is the rotation rate in revolutions per minute). In an aqueous solution at a moderate rotation rate (~ 1000 RPM), the stagnant layer is approximately 300 to 400 μm thick.

Net movement of material to the electrode surface can be described mathematically by applying general convection-diffusion concepts from fluid dynamics. Mass transport of material from the bulk solution into the stagnant layer occurs by convection (due to the stirring action of the rotating electrode). But after the material enters the stagnant layer and moves closer to the electrode surface, convection becomes less important and diffusion becomes more important. Indeed, the final movement of an ion or molecule to the electrode surface is dominated by diffusion across a very thin layer of solution immediately adjacent to the electrode known as the diffusion layer.

The diffusion layer is much thinner than the hydrodynamic layer. The diffusion layer thickness (δ_F) can be approximated as follows,

$$\delta_F = 1.61 D_F^{1/3} \nu^{1/6} \omega^{-1/2} \quad (9)$$

in terms of the diffusion coefficient (D_F) of the molecule or ion. For a molecule or ion with a typical diffusion coefficient ($D_F \approx 10^{-5} \text{ cm}^2/\text{s}$) in an aqueous solution, the diffusion layer is about twenty times thinner than the stagnant layer ($\delta_F \approx 0.05 \delta_H$).

The first mathematical treatment of convection and diffusion towards a rotating disk electrode was given by Levich. Considering the case where only the oxidized form of a molecule (or ion) of interest is initially present in the electrochemical cell, the cathodic limiting current (i_{LC}) observed at a rotating disk electrode is given by the Levich equation,^{2,9}

$$i_{LC} = 0.620 n F A D_O^{2/3} \nu^{-1/6} \omega^{1/2} C_O \quad (10)$$

in terms of the concentration of the oxidized form in the solution (C_O), the electrode area (A), the Faraday constant ($F = 96485 \text{ C/mol}$), the kinematic viscosity of the solution (ν), the diffusion coefficient (D_O) of the oxidized form, and the angular rotation rate (ω). Alternatively, when the solution initially contains only the reduced form, the equation for the anodic limiting current (i_{LA}) can be written as

$$i_{LA} = 0.620 n F A D_R^{2/3} \nu^{-1/6} \omega^{1/2} C_R \quad (11)$$

where the concentration and diffusion coefficient terms (C_R and D_R) refer to the reduced form rather than the oxidized form.

9.4.1 Levich Study

A Levich study is a common experiment performed using a rotating disk electrode in which a series of voltammograms is acquired over a range of different rotation rates. For a simple electrochemical system where the rate of the half reaction is governed only by mass transport to the electrode surface, the overall magnitude of the voltammogram should increase with the square root of the rotation rate (see: Figure 9-7, left).

The currents measured during a Levich study are usually plotted against the square root of the rotation rate on a graph called a Levich plot. As predicted by the Levich equation, the limiting current (see red circles on Figure 9-7, right) increases linearly with the square root of the rotation rate (with a slope of

$0.620 n F A D^{2/3} \nu^{-1/6} C$) and the line intercepts the vertical axis at zero. It is common to choose a set of rotation rates that are multiples of perfect squares (such as 100, 400, 900, 1600 RPM, etc.) to facilitate construction of this plot.

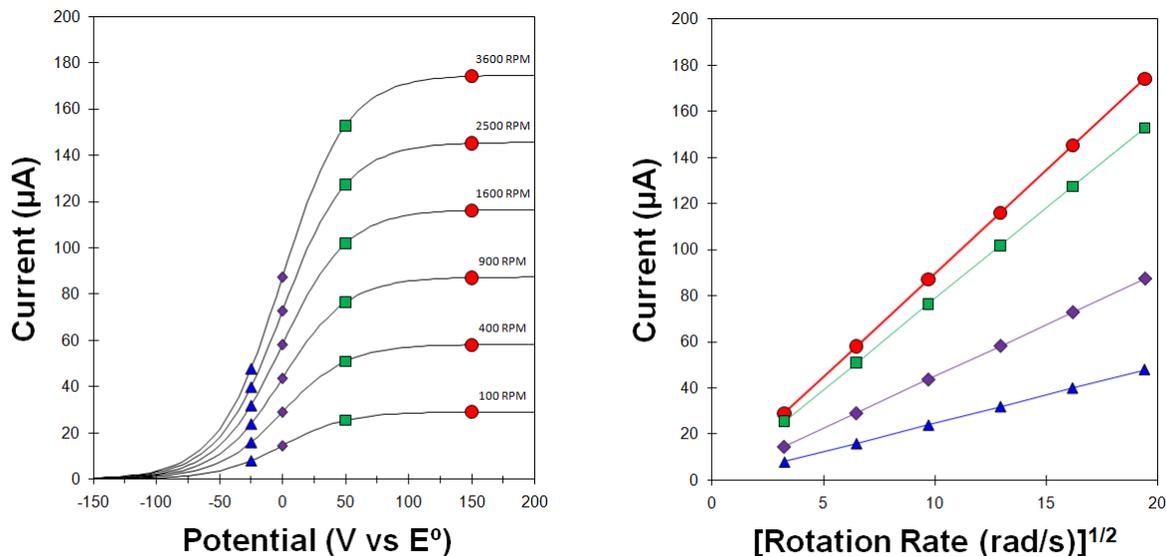


Figure 9-7. Levich Study – Voltammograms at Various Rotation Rates

If the electrochemical half-reaction observed during a Levich study is a simple and reversible half reaction (with no complications due to sluggish kinetics or coupled chemical reactions), then the shapes of the mass-transport controlled voltammograms will be sigmoidal regardless of the rotation rate. This means that the current observed at any given potential along the voltammogram will vary linearly with the square root of the rotation rate (see: Figure 9-7, right); however, it is important to remember that the Levich equation only applies to the limiting current, not to the currents along the rising portion of the sigmoid.

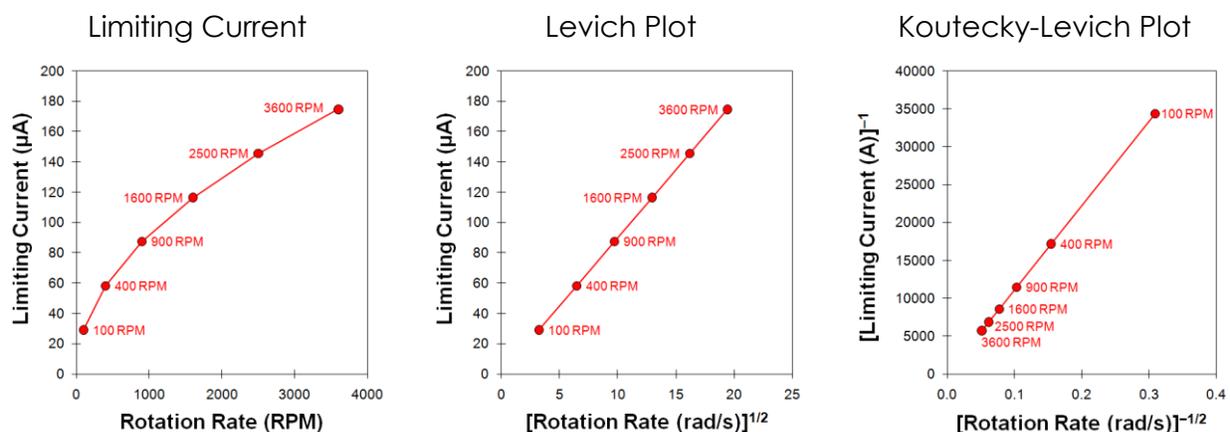


Figure 9-8. Levich Study – Limiting Current versus Rotation Rate

Because the Levich equation only applies to the limiting current, the results from a Levich experiment are typically presented as a simple plot of the limiting current versus the square root of the rotation rate

(see: Figure 9-8, center). An alternate method of presenting the data from a Levich study is based on a rearrangement of the Levich equation in terms of the reciprocal current.

$$\frac{1}{i_L} = \left(\frac{1}{0.620 n F A D^{2/3} \nu^{-1/6} C} \right) \omega^{-1/2} \quad (12)$$

A plot of reciprocal current versus the reciprocal square root of the angular rotation rate (see: Figure 9-8, right) is called a Koutecky-Levich plot^{2,14}. Again, for a simple and reversible half reaction with no complications, the data fall along a straight line that intercepts the vertical axis at zero. If the line intercepts the vertical axis above zero, however, this is a strong indication that the half-reaction is limited by sluggish kinetics rather than by mass transport.

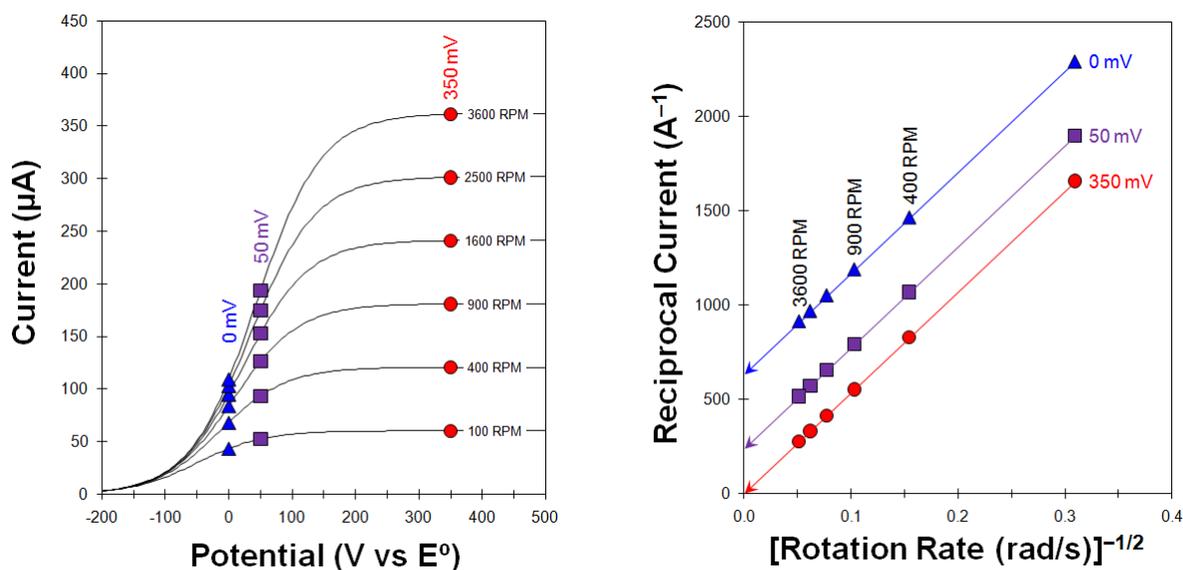


Figure 9-9. Koutecky-Levich Study – Voltammograms with Sluggish Kinetics

9.4.2 Koutecky-Levich Analysis

When the rate of a half reaction occurring at an electrode surface is limited by a combination of mass transport and sluggish kinetics, it is often possible to use a rotating disk electrode to elucidate both the mass transport parameters (such as the diffusion coefficient) and the kinetic parameters (such as the standard rate constant, k^0) from a properly designed Levich study. A full treatment of this kind of analysis¹⁴ is beyond the scope of this document, but the following is a general description of how to extract kinetic information from a set of rotating disk voltammograms.

When the electron transfer process at an electrode surface exhibits sluggish kinetics, the voltammogram appears stretched out along the potential axis, and the shape of the sigmoidal wave is slightly distorted. Comparing a set of voltammograms with facile kinetics (see: Figure 9-7) with a set of voltammograms with sluggish kinetics (see: Figure 9-9), the mass transport limited current plateau (marked by red circles in each figure) is shifted further away from the standard electrode potential (E^0) when there are slow kinetics. Stated another way, when a sluggish redox half reaction is studied with a rotating disk electrode, a larger overpotential must be applied to the electrode to overcome the sluggish kinetics and reach the mass transport limited current.

This distortion of the ideal sigmoidal shape of the voltammogram can be exploited as a way to measure the standard rate constant (k^0). The general approach is to acquire a set of voltammograms at different rotation rates (*i.e.*, perform a Levich study) and then plot the reciprocal current (sampled at particular locations along the rising portion of each voltammogram) on a Koutecky-Levich Plot. In the example provided (see: Figure 9-9, left), the current was sampled at two locations along the rising portion of the voltammograms (*at 0 and 50 mV vs. E^0* , marked with blue triangles and purple squares) and at one location on the limiting current plateau (*at 350 mV vs. E^0* , marked with red circles). A linear relationship is evident (see: Figure 9-9, right) when these sampled currents are plotted on a Koutecky-Levich Plot.

For the set of currents sampled on the limiting current plateau (see: Figure 9-9, red circles), an extrapolation back to the vertical axis (*i.e.*, to infinite rotation rate) yields a zero intercept. This is the identical result obtained for a facile half-reaction (see: Figure 9-8, right) because these currents are sampled at a high enough overpotential that there are no kinetic limitations. Only mass transport limits the current, and the usual Levich behavior applies.

However, for the two sets of currents sampled on the rising portion of the voltammogram (see: Figure 9-9, blue triangles and purple squares), the extrapolation back to the vertical axis yields non-zero intercepts. This non-zero intercept indicates a kinetic limitation, meaning that even if mass transport were infinite (*i.e.*, infinite rotation rate), the rate of the half-reaction would still be limited by the slow kinetics at the electrode surface.

The linear portion of the data on a Koutecky-Levich plot is described by the Koutecky-Levich equation.

$$\frac{1}{i_L} = \frac{1}{i_K} + \left(\frac{1}{0.620 n F A D^{2/3} \nu^{-1/6} C} \right) \omega^{-1/2} \quad (13)$$

Plotting the reciprocal current ($1/i$) against the reciprocal root of the angular rotation rate ($\omega^{-1/2}$) yields a straight line with an intercept equal to the reciprocal kinetic current ($1/i_K$). The kinetic current is the current that would be observed in the absence of any mass transport limitations. By measuring the kinetic current at a variety of different overpotentials along the voltammogram, it is possible to determine the standard rate constant for the electrochemical half reaction.

Further details regarding Koutecky-Levich theory, including various forms of the Koutecky-Levich equation which pertain to different electrochemical processes, can be found in the literature.¹⁴

9.5 Rotating Ring-Disk Electrode (RRDE) Theory

In 1958, Russian electrochemist Alexander Frumkin suggested the idea of placing a concentric ring electrode around the rotating disk electrode.¹⁵ His colleague, Lev Nekrasov, supervised construction of the world's first rotating ring-disk electrode (RRDE) apparatus.¹⁶⁻¹⁹ At the same time, Benjamin Levich and Yuri Ivanov began working on a theoretical description of solution flow at the RRDE. The four Russian researchers published their initial findings in 1959, and their work caught the attention of both Stanley Bruckenstein at the University of Minnesota and John Albery at Oxford University. Bruckenstein travelled to Moscow to learn more about the RRDE,²⁰ and after he returned home in 1965, Albery joined Bruckenstein's research group (along with Dennis Johnson and Duane Napp). The experimental and mathematical work performed by these four researchers at Minnesota generated a significant series of papers about the RRDE²¹⁻²⁶ and placed the new technique on a firm theoretical foundation. Albery returned to Oxford and (working with his student Michael Hitchman) drew these theoretical papers together in a seminal volume titled *Ring-Disk Electrodes*.²¹

The overall flow pattern at the RRDE initially brings molecules and ions to the disk electrode. After encountering the disk electrode, the subsequent outward radial flow carries a fraction of these molecules or ions past the surface of the ring electrode. This flow pattern allows products generated (upstream) by the half reaction at the disk electrode to be detected as they are swept (downstream) past the ring electrode.

Two of the key parameters which characterize a given ring-disk geometry are the collection efficiency and the transit time.²³ The collection efficiency is the fraction of the material from the disk which subsequently flows past the ring electrode. It can be expressed as a fraction between 0.0 and 1.0 or as a percentage. Typical ring-disk geometries have collection efficiencies between 20% and 30%. The transit time is a more general concept indicating the average time required for material at the disk electrode to travel across the gap between the disk and the ring electrode. Obviously, the transit time is a function of both the gap distance and the rotation rate.

9.5.1 Theoretical Computation of the Collection Efficiency

The theoretical collection efficiency can be computed^{2,23} from the three principle diameters describing the RRDE geometry: the disk outer diameter (d_1), the ring inner diameter (d_2), and the ring outer diameter (d_3). This somewhat tedious computation is made easier by normalizing the ring diameters with respect to the disk diameter as follows:

$$\sigma_{OD} = d_3 / d_1 \quad \text{and} \quad \sigma_{ID} = d_2 / d_1$$

Three additional quantities are defined in terms of the normalized diameters as follows:

$$\sigma_A = \sigma_{ID}^3 - 1 \qquad \sigma_B = \sigma_{OD}^3 - \sigma_{ID}^3 \qquad \sigma_C = \sigma_A / \sigma_B$$

If a complex function, $G(x)$, is defined as follows,

$$G(x) = \frac{1}{4} + \left(\frac{\sqrt{3}}{4\pi} \right) \ln \left[\frac{(x^{1/3} + 1)^3}{x + 1} \right] + \left(\frac{3}{2\pi} \right) \arctan \left[\frac{2x^{1/3} - 1}{\sqrt{3}} \right]$$

then the theoretical collection efficiency ($N_{theoretical}$) for a rotating ring-disk electrode is given by the following equation first reported by Albery and Bruckenstein:²³

$$N_{theoretical} = 1 - \sigma_{OD}^2 + \sigma_B^{2/3} - G(\sigma_C) - \sigma_B^{2/3} G(\sigma_A) + \sigma_{OD}^2 G(\sigma_C \sigma_{OD}^3) \quad (14)$$

9.5.2 Empirical Measurement of the Collection Efficiency

Direct computation of the theoretical collection efficiency is possible using the above relationships if the actual machined dimensions of the disk and ring are known for a particular RRDE. In practice, the actual RRDE dimensions may not be known due to uncertainties in the machining process and changes in the dimensions induced by electrode polishing or temperature cycling. For this reason, it is common practice to empirically measure the collection efficiency using a well-behaved redox system rather than to rely upon a computed value.

The ferrocyanide/ferricyanide half reaction is a simple, single-electron, reversible half reaction that is often used as the basis for measuring collection efficiency.³⁶ The RRDE is placed in a solution containing

a small concentration ($\sim 10\text{ mM}$) of potassium ferricyanide, $K_3Fe(CN)_6$, in a suitable aqueous electrolyte solution (such as 1.0 M potassium nitrate, KNO_3) and is operated at rotation rates between 500 RPM and 2000 RPM . Initially, both the ring and the disk electrodes are held at a sufficiently positive potential that no reaction occurs. Then, the potential of the disk electrode is slowly swept ($\sim 50\text{ mV/sec}$) towards more negative potentials, and a cathodic current is observed which corresponds to the reduction of ferricyanide to ferrocyanide at the disk.



As ferricyanide is reduced at the disk electrode, the ferrocyanide generated by this process is swept outward (radially) away from the disk electrode and toward the ring electrode. The ring electrode is held constant at a positive (oxidizing) potential throughout the experiment. Some (but not all) of the ferrocyanide generated at the disk travels close enough to the ring electrode that it is oxidized back to ferricyanide. Thus, an anodic current is observed at the ring electrode due to the oxidation of ferrocyanide to ferricyanide at the ring.



The measured ratio of the ring (anodic) limiting current to the disk (cathodic) limiting current is the empirical collection efficiency. As the rotation rate increases, both the disk and the ring currents increase (see: Figure 9-10). Because both the anodic and cathodic limiting currents are proportional to the square root of the rotation rate, the empirical collection efficiency is expected to be independent of the rotation rate.

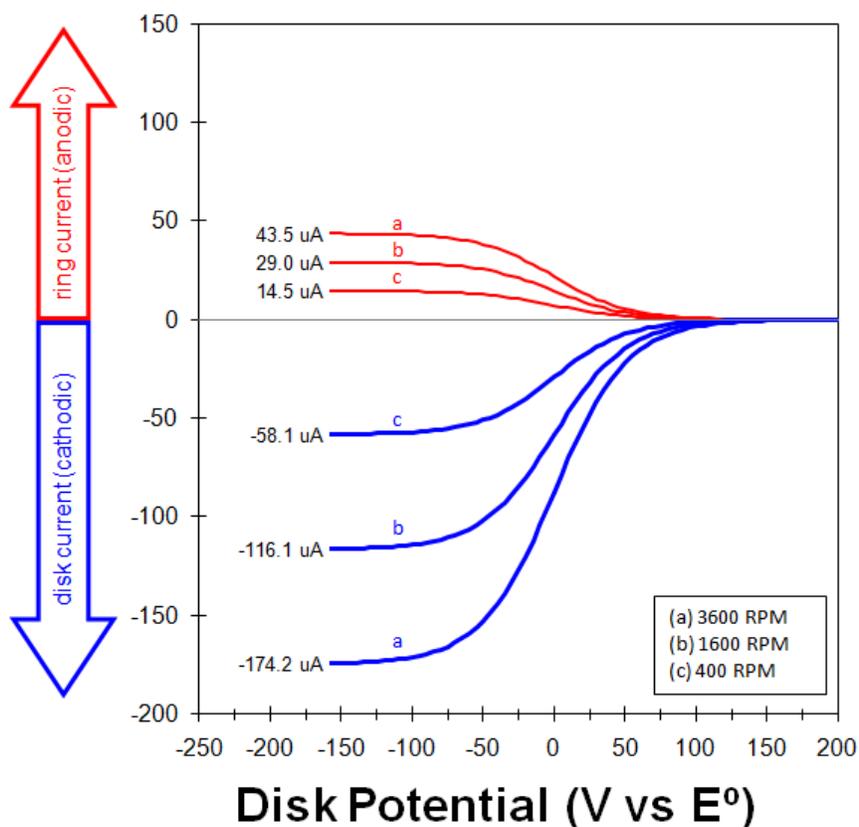


Figure 9-10. Rotating Ring-Disk Electrode Voltammograms at Various Rotation Rates

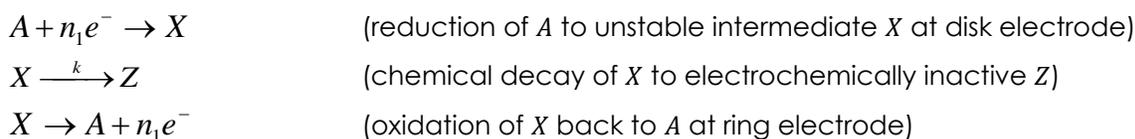
Once the collection efficiency value has been established empirically for a particular RRDE, it can be treated as a property of that particular RRDE, even if the RRDE is used to study a different half reaction in a different solution on a different day. Although the empirically measured collection efficiency ($N_{empirical}$) is a ratio of two currents which often have opposite mathematical signs (anodic and cathodic), the empirical collection efficiency is always expressed as a positive number, as follows,

$$N_{empirical} = \left| \left(\frac{i_{L,RING}}{i_{L,DISK}} \right) \left(\frac{n_D}{n_R} \right) \right| \quad (15)$$

where n_D and n_R are the number of electrons exchanged at the disk and ring (and very often, n_D and n_R are equal to each other).

9.5.3 Generator/Collector Experiments

When a molecule or ion is oxidized or reduced at an electrode, it is often transformed into an unstable intermediate chemical species which, in turn, is likely to undergo additional chemical changes. The intermediate may have a long enough lifetime that it is capable of moving to the ring electrode and being detected. Or, the intermediate may be so unstable that it decays away before it can be detected at the ring. Consider the following reaction scheme at a rotating ring-disk electrode:



In the above scheme, the disk electrode is poised at a potential where A is reduced to X , and the cathodic limiting current observed at the disk (i_{DISK}) is a measure of how much X is being “generated” at the disk electrode. At the same time, the ring electrode is poised at a more positive potential where X is oxidized back to A , and the anodic limiting current observed at the ring (i_{RING}) is a measure of how much X is being “collected” at the ring. There is also a competing chemical reaction which is capable of eliminating X before it has a chance to transit from the disk to the ring.

The ratio of the ring current to the disk current under these conditions is called the apparent collection efficiency ($N_{apparent}$).

$$N_{apparent} = - i_{RING} / i_{DISK} \quad (16)$$

By comparing the apparent collection efficiency ($N_{apparent}$) to the previously measured empirical collection efficiency ($N_{empirical}$) for the same RRDE, it is possible to deduce the rate at which the competing chemical pathway is converting X to Z . That is, it is possible to use an RRDE “generator/collector” experiment to measure the kinetic behavior of unstable electrochemical intermediates.

Whenever the apparent and empirical collection efficiencies are equal ($N_{apparent} = N_{empirical}$), it is an indication that the decay rate of the intermediate (via the $X \rightarrow Z$ pathway) is small with respect to the transit time required for X to travel from the disk to the ring. One way to shorten the transit time is to spin the RRDE at a faster rate. At high rotation rates, the apparent collection efficiency should approach the empirical collection efficiency. Conversely, at slower rotation rates, the apparent collection efficiency may be smaller ($N_{apparent} < N_{empirical}$) because some of the intermediate is consumed by the competing chemical pathway before X can travel to the ring.

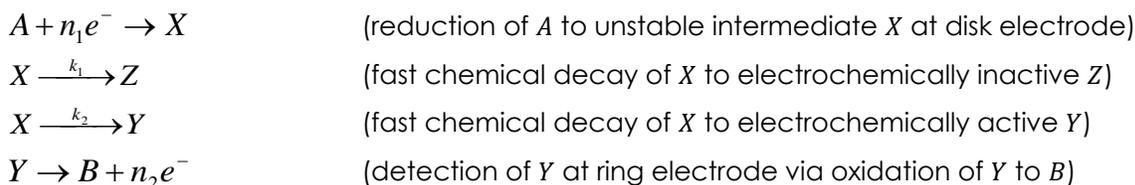
By recording a series of rotating ring-disk voltammograms at different rotation rates and analyzing the results, it is possible to estimate the rate constant (k) associated with the intermediate chemical decay pathway. Various relationships have been proposed for this kind of analysis,² and one of the simplest is shown below.

$$\frac{N_{\text{empirical}}}{N_{\text{apparent}}} = 1 + 1.28 \left(\frac{\nu}{D}\right)^{1/3} \left(\frac{k}{\omega}\right) \quad (17)$$

A plot of the ratio of the empirical to the apparent collection efficiency versus the reciprocal angular rotation rate should be linear. The slope of such a plot can yield the rate constant if the kinematic viscosity (ν) and the diffusion coefficient (D) are known.

9.5.4 Comparing Two Competing Pathways

Sometimes the intermediate generated by an electrochemical process can decay via two different pathways. As long as one of these pathways leads to an electrochemically active chemical species that can be detected at the ring, it is possible to determine which decay pathway is favored. Consider the following scheme:



In the above scheme, the disk electrode is poised at a potential where A is reduced to X , and the cathodic limiting current observed at the disk (i_{DISK}) is a measure of how much X is being “generated” at the disk electrode. The intermediate X is unstable, and as it is swept away from the disk and toward the ring, it rapidly decays to either Y or Z . By the time these species reach the ring, all of the X has decayed away, and the solution in contact with the ring contains both Y and Z . The species Z is electrochemically inactive and cannot be detected by the ring, but the species Y is active. By carefully poisoning the ring electrode at a potential appropriate for detecting Y (in this case, by oxidizing Y to B), it is possible for the ring to “collect” any Y which arrives at the surface of the ring.

The ratio of the ring current (due to Y being detected at the ring) to the disk current (due to X being generated at the disk) reveals the extent to which the $X \rightarrow Y$ pathway is favored in comparison to the $X \rightarrow Z$ pathway. The fraction of the decay by the $X \rightarrow Y$ pathway (θ_{XY}) can be computed as follows:

$$\theta_{XY} = \left(\frac{1}{N_{\text{empirical}}}\right) \left(\frac{n_1}{n_2}\right) \left|\frac{i_{\text{RING}}}{i_{\text{DISK}}}\right| \quad (18)$$

Note in the above equation that the fraction (n_1/n_2) carefully accounts for any difference in the number of electrons involved in the disk half reaction and the number of electrons involved when detecting Y at the ring electrode. Schemes involving more complex stoichiometry may require additional correction factors.

The most commonly studied reaction at the RRDE is undoubtedly the oxygen reduction reaction (ORR).³³⁻⁴³ When oxygen (O_2) is dissolved in acidic media and reduced at a platinum electrode, one pathway leads to water as the ultimate reduction product while the other pathway leads to the

formation of peroxide anions. In the context of hydrogen fuel cell research, the pathway which leads to water is preferred, and it is commonly called the four-electron pathway. The path to peroxide formation is called the two-electron pathway, and it is undesirable for a number of reasons, including the fact that peroxide can damage various polymer membrane materials found in a fuel cell. Further details on how to use an RRDE “generator/collector” experiment to distinguish between the two-electron and four-electron ORR pathways can be found in the electrochemical literature.^{33,36}

9.6 Rotating Cylinder Electrode (RCE) Theory

The rotating disk and ring-disk electrodes were developed primarily as a result of academic electroanalytical chemistry research. In contrast, the theory for the rotating cylinder electrode (RCE) was developed by industrial researchers⁴⁴⁻⁴⁶ in the corrosion and electroplating communities. While the flow of solution at a rotating disk (or ring-disk) is laminar over a wide range of rotation rates, the flow at the surface of a rotating cylinder is turbulent³² at all but the slowest rotation rates. Thus, the RCE is an excellent tool for creating and controlling turbulent flow conditions in the laboratory, and it is most commonly used to mimic turbulent corrosion conditions found in large scale industrial settings such as oilfield pipeline corrosion.⁵⁶⁻⁶⁹

The turbulent flow at a rotating cylinder electrode conveys material from the bulk solution towards the electrode surface. While the bulk solution remains well stirred by the main vortex induced by the rotating electrode, the layer of solution adjacent to the cylinder surface tends to rotate with the electrode. Thus, a high shear condition is set up at the surface of the rotating cylinder, spinning off smaller Taylor vortices adjacent to the rotating electrode.

Net movement of material to the surface of a rotating cylinder was first characterized by Morris Eisenberg^{27,28} in 1954 (about the same time that Levich was describing the rotating disk electrode). Eisenberg's work eventually led to the Eisenberg equation which gives the limiting current at a rotating cylinder electrode

$$i_L = 0.0487 n F A d_{cyl}^{+0.4} D^{+0.644} \nu^{-0.344} \omega^{+0.7} C \quad (19)$$

in terms of the concentration (C) and diffusion coefficient (D) of the molecule or ion being studied, the Faraday constant ($F = 96485$ coulombs per mole), the electrode area (A), the diameter of the cylinder (d_{cyl}), the kinematic viscosity of the solution (ν), and the angular rotation rate ($\omega = 2\pi f / 60$, where f is the rotation rate in revolutions per minute). In the years since Eisenberg's initial work with the rotating cylinder, additional work by Gabe, Kear, Walsh, and Silverman has described industrial applications of the RCE.^{29,32,46,49,62}

9.7 References

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10. Glossary

Anodic Current	Flow of charge at an electrode due to an oxidation reaction occurring at the electrode surface. For a working electrode immersed in a test solution, an anodic current corresponds to flow of electrons out of the solution and into the electrode.
Banana Cable	A banana cable is a single-wire (one conductor) signal cable often used to make connections between various electronic instruments. Each end of the cable has a banana plug. The plug consists of a cylindrical metal pin about 25 mm (one inch) long, with an outer diameter of about 4 mm, which can be inserted into a matching banana jack.
Banana Jack	Female banana connector
Banana Plug	Male banana connector
BNC Connector	The BNC (Bayonet Neill-Concelman) connector is a very common type of RF connector used for terminating coaxial cable.
Brush Contacts	Electrical contact to the rotating shaft is accomplished by means of silver-carbon brush contacts. These brushes are spring loaded to assure that they are firmly pressed against the rotating shaft at all times.
Cathodic Current	Flow of charge at an electrode due to a reduction reaction occurring at the electrode surface. For a working electrode immersed in a test solution, a cathodic current corresponds to flow of electrons out of the electrode and into the solution.
Coaxial Cable	Coaxial cable (coax) is an electrical cable with an inner conductor surrounded by a flexible, tubular insulating layer, surrounded by a tubular conducting shield. The term coaxial comes from the inner conductor and the outer shield sharing the same geometric axis. Coaxial cable is often used to carry signals from one instrument to another in situations where it is important to shield the signal from environmental noise sources.
Collection Efficiency	In the context of rotating ring-disk voltammetry, the collection efficiency is a measure of the amount of material generated at the disk electrode which ultimately makes its way to the ring electrode. It is often expressed as a percentage, and typical collection efficiencies fall between 20% and 30%.
Collection Experiment	An experiment with a rotating ring-disk electrode where the ring potential is held constant while the disk potential is swept slowly between two limits.
Convection	Convection is the movement of molecules or ions through a liquid solution as a result of bulk movement of the solution. Such bulk movement may be due to stirring the solution or due to vibrations or thermal gradients in the solution.

Counter Electrode	The counter electrode, often also called the auxiliary electrode, is one of three electrodes found in a typical three-electrode voltammetry experiment. The purpose of the counter electrode is to help carry the current across the solution by completing the circuit back to the potentiostat.
Cyclic Voltammetry	An electroanalytical method where the working electrode potential is repeatedly swept back and forth between two extremes while the working electrode current is measured.
Cylinder Insert	Most rotating cylinder electrode tips are designed to accept cylinder inserts fabricated from various alloys of interest to corrosion scientists.
Diffusion	In the context of electrochemistry in liquid solutions, diffusion is a time-dependent process consisting of random motion of ions or molecules in solution which leads to the statistical distribution of these species, gradually spreading the ions and molecules through the solution.
Diffusion Coefficient	A factor of proportionality representing the amount of substance diffusing across a unit area through a unit concentration gradient in unit time.
Diffusion Layer	Mass transport to a rotating electrode occurs via a combination of convection and diffusion. As material approaches the electrode, diffusion dominates over convection as the principle means of transport. Across the very thin layer of solution immediately adjacent to the electrode, diffusion is essentially the only means of mass transport. This thin layer is known as the diffusion layer. The diffusion layer should not be confused with the stagnant layer. The diffusion layer exists entirely within the thicker stagnant layer (see also Stagnant Layer).
Disk Insert	Some rotating disk and ring-disk electrode tips are designed to accept interchangeable disk inserts fabricated from various precious metals and advanced carbon materials.
Eisenberg Equation	The Eisenberg equation describes the mass transfer limited current at a rotating cylinder electrode.
Electroactive	An adjective used to describe a molecule or ion capable of being oxidized or reduced at an electrode surface.
Electrode	An electrode is an electrical conductor used to make contact with a nonmetallic part of a circuit.
Electrode Materials	Common electrode materials used to fabricate rotating disk and ring-disk electrodes are gold, platinum, and glassy carbon. Rotating cylinder electrodes are usually made from various alloys of steel, aluminum, or brass.
Faradaic Current	The portion of the current observed in an electroanalytical experiment that can be attributed to one or more redox processes occurring at an electrode surface.
Forced Convection	Active stirring or pumping of a liquid solution.

Half-Reaction	A balanced chemical equation showing how various molecules or ions are being reduced (or oxidized) at an electrode surface.
Hydrodynamic Layer	(see the definition of stagnant layer)
Hydrodynamic Voltammetry	A family of electroanalytical methods based upon precise control of solution flow coupled with rigorous mathematical models.
Insulating Materials	Chemically-resistant and electrically insulating polymers commonly used to fabricate rotating electrodes include PTFE, PEEK and PCTFE.
Laminar Flow	Laminar flow, sometimes known as streamline flow, occurs when a fluid flows in parallel layers, with no disruption between the layers.
Levich Equation	The Levich equation describes the mass transfer limited current at a rotating disk electrode.
Levich Study	Experiment using a rotating disk electrode in which a series of voltammograms are acquired over a range of rotation rates.
Levich Plot	A plot of limiting current vs. square root of rotation rate from a Levich study.
Linear Polarization Resistance	Term used in corrosion science for an experiment in which the electrode potential is changed from an initial value to final value at a slow and constant rate. This technique is similar to linear sweep voltammetry, but the sweep rates are much slower, and the results are plotted differently.
Linear Sweep Voltammetry	Experiment in which the working electrode potential is swept from initial value to final value at a constant rate while the current is measured.
Mass Transport Limited Current	The current corresponding to the maximum mass transfer rate of an ion or molecule to an electrode surface.
Migration	In an electroanalytical context, the term migration refers to the movement of ions across a solution under the influence of an electric field.
Non-Faradaic Current	The portion of the current observed in an electroanalytical experiment that cannot be attributed to any redox processes occurring at an electrode surface.
Overpotential	The overpotential is the difference between the formal potential of a half reaction and the potential presently being applied to the working electrode.
Oxidation	Removal of electrons from an ion or molecule.
PCTFE	A chemically inert polymer often used as an insulating shroud for an electrode. PCTFE is an abbreviation for polychlorotrifluoroethylene.

PEEK	A chemically inert polymer often used as an insulating shroud for an electrode. PEEK is an abbreviation for polyether ether ketone.
PTFE	A chemically inert polymer often used as an insulating shroud for an electrode. PTFE is an abbreviation for polytetrafluoroethylene.
Quiescent Solution	A solution in which there is little or no convection.
Redox	An adjective used to describe a molecule, ion, or process associated with an electrochemical reaction.
Reduction	Addition of electrons to an ion or molecule.
Reference Electrode	A reference electrode has a stable and well-known thermodynamic potential. The high stability of the electrode potential is usually reached by employing a redox system with constant (buffered or saturated) concentrations of the ions or molecules involved in the redox half reaction.
Reynolds Number	In fluid mechanics, the Reynolds number is a dimensionless number that gives a measure of the ratio of inertial forces to viscous forces and consequently quantifies the relative importance of these two types of forces for given flow conditions.
Rotation Rate	The rate at which a rotating electrode rotates. Experimentally, this is usually expressed in RPM, but in theoretical equations, the rotation rate is usually expressed in radians per second.
Shielding Experiment	An experiment with a rotating ring-disk electrode where the disk potential is held constant while the ring potential is swept slowly between two limits.
Stagnant Layer	At a rotating electrode, the portion of the solution near the electrode tends to rotate at nearly the same speed as the electrode surface. This layer of solution is known as the stagnant layer (or, in the context of fluid dynamics, the stagnant layer is more properly called the hydrodynamic layer). Mass transport across the stagnant layer occurs by a combination of convection and diffusion, with diffusion dominating as the material travels closer to the electrode surface (see also Diffusion Layer).
Standard Electrode Potential	A thermodynamic quantity expressing the free energy of a redox half reaction in terms of electric potential.
Sweep Rate	Rate at which the electrode potential is changed when performing a sweep voltammetry method such as cyclic voltammetry.
Three-Electrode Cell	A common electrochemical cell arrangement consisting of a working electrode, a reference electrode, and a counter electrode.

Transit Time	In the context of rotating ring-disk voltammetry, the transit time is the average amount of time required for material generated at the disk electrode to be swept over to the ring electrode.
Turbulent Flow	Chaotic (non-laminar) flow of solution.
Voltammogram	A plot of current vs. potential from an electro-analytical experiment in which the potential is swept back and forth between two limits.
Window Experiment	An experiment with a rotating ring-disk electrode where the disk potential is swept slowly between two limits, and the ring potential is swept in the same manner as the disk potential but with a constant offset between the ring and disk potentials.
Working Electrode	The electrode at which the redox process of interest occurs. While there may be many electrodes in an electrochemical cell, the focus of an experiment is typically only on a particular half reaction occurring at the working electrode.



EU Declaration of Conformity

In accordance with EN ISO 17050-1:2010

Object of the Declaration:

Product: Analytical Rotator
Model: AF01WV10
Serial numbers: 4617101 and later (wwyy1nn: where ww is the week of manufacture; yy is the year of manufacture; nn is a consecutive number, beginning with 01 each week)
Manufacturer: Pine Electronics LLC
Address: 101 Industrial Drive, Grove City, PA, 16127, USA

This declaration is issued under the sole responsibility of the manufacturer.

The object of the declaration described above is in conformity with the relevant Union harmonization legislation:

2014/35/EU	The Low Voltage Directive
2014/30/EU	The Electromagnetic Compatibility Directive
2011/65/EU	The Restriction of Hazardous Substances Directive

Conformity is shown by compliance with the applicable requirements of the following documents:

Reference and Date	Title
EN 61326-1:2013	EMC Requirements for electrical equipment for measurement, control and laboratory use
IEC 61010-1:2010, AMD1:2016	Safety Requirements for electrical equipment for measurement, control and laboratory use

Signed for and on behalf of: Pine Electronics LLC/Pine Research Instrumentation, Inc.
Place of Issue: Grove City, PA, USA
Date of Issue: 11/16/2017
Name: Edward T. Berti
Position: Principal Engineer

Signature: 

The technical documentation for the machinery is available from the above address.

The CE Mark was applied per the following reports:

Keystone Compliance: 1707-106EA
Intertek: 103238230COL-001 and 103238230COL-002