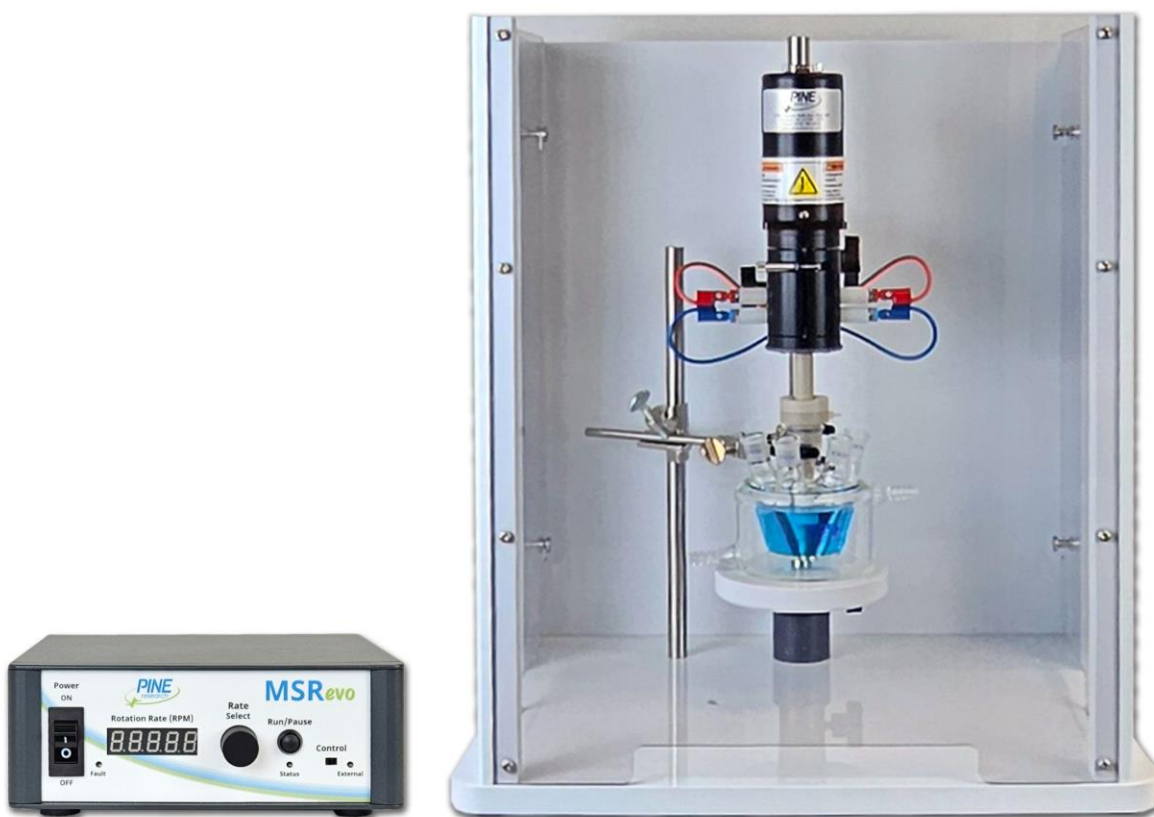


MSR evo Electrode Rotator User Guide



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1 Preface

1.1 Scope

The MSR evo Electrode Rotator is a solid-state-controlled servo-system designed to rotate an electrode in an electrochemical cell. This user guide describes the proper use of the MSR evo and covers routine operating procedures, periodic maintenance and calibration, and safety issues.

The reader of this user guide is assumed to have some basic knowledge of electronics, electrochemistry, and the modern practice of voltammetry. 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 voltammetry.

Pine Research Instrumentation provides online support for the MSR evo and other products on our website, including PDF versions of user guides and various documentation:

<https://www.pineresearch.com/>

1.2 Copyright

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1.3 Trademarks

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1.4 Use Limitation

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

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

1.5 Harmful or Corrosive Substances

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

- Eye protection (safety goggles, face shield, etc.)
- Laboratory coat (flame- 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, <i>Identifying and Evaluating Hazards in Research Laboratories: Guidelines Developed by the Hazards Identification and Evaluation Task Force of the ACS Committee on Chemical Safety</i> ; 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, <i>Prudent Practices in the Laboratory: Handling and Management of Chemical Hazards, Updated Version</i> ; National Academies Press, 2011.
3. American Chemical Society Committee on Chemical Safety. <i>Safety in Academic Chemistry Laboratories; 7th ed.</i> ; American Chemical Society: State College, PA, 2003; Vol. 2.

L'opérateur du MSR evo 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 MSR evo or other technical issues, please use the contact information below to contact Pine Research directly.

TECHNICAL SERVICE CONTACT

Pine Research Instrumentation, Inc.
<https://www.pineresearch.com>
 Phone: +1 (919) 782-8320
 Email: pinewire@pineresearch.com

If the MSR evo or one of its components or accessories must be returned to the factory 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
 Phone: +1 (724) 458-6391



Return Material Authorization Required!

Do not ship equipment to the factory without first obtaining a Return Material Authorization (RMA) form from Pine Research Instrumentation, Inc.

LIMITED WARRANTY

The MSR evo (hereafter referred to as the "INSTRUMENT") offered by Pine Research Instrumentation, Inc. (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") if used under normal conditions. The obligation under this warranty is limited to replacing or repairing parts that 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 remote support (via telephone or internet) to guide the CUSTOMER to diagnose and effect any needed repairs. In the event that remote support is unsuccessful in resolving the defect, PINE may recommend that the INSTRUMENT be returned to PINE for repair. This warranty is 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, tariffs, customs, duties, labor, parts, and freight charges.

1.7 Instrument Markings

Labels located on the MSR evo control box and motor unit bear information about the make, model, and serial number of the instrument. These labels also indicate any certifications or independent testing agency marks that pertain to the instrument (see Figure 1-1).

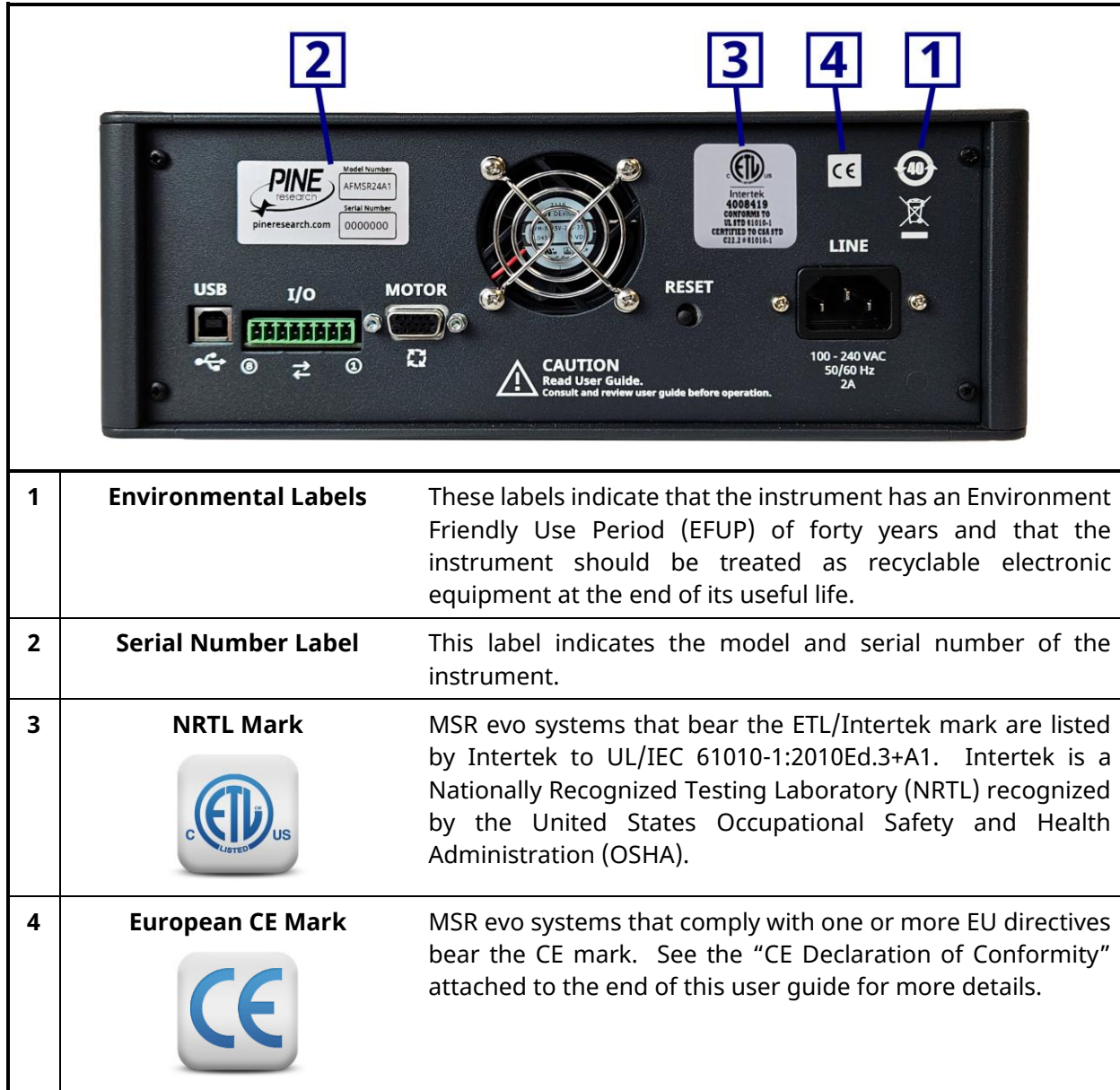


Figure 1-1. MSR evo Instrument Markings

1.7.1 Serial Number

For purposes of uniquely identifying a particular instrument, there is a label on the back panel of the MSR evo control box that indicates the model number and the serial number. A matching serial number is also found on a similar label located on the motor unit. In laboratories with multiple MSR evo rotators, it is important to keep the control unit together with the matching motor unit because the two components are calibrated together as a system (see Section 6.6).

1.7.2 Model Numbers

The MSR evo model number has the format 'AFMSR24XY' where X and Y are single characters used to indicate the particular configuration of the rotator (see Table 1-1). Part numbers for electrodes and other accessories compatible with the MSR evo are described in more detail later (see Section 5 and Section 7).




Model Number:	A	F	M	S	R	2	4	X	Y	Model Name
	A	F	M	S	R	2	4	A	1	MSR evo

Table 1-1. MSR evo Instrument Model Number and Model Name

1.8 Icons (Icônes)

Special icons are used to call attention to safety warnings and other useful information found in this document (see Table 1-2, Table 1-3, and Table 1-4).

Des icônes spéciales sont utilisées pour attirer l'attention sur des avertissements de sécurité et autres renseignements utiles disponibles dans ce document (voir Tableau 1-2, Tableau 1-3, et Tableau 1-4).

	<p>STOP: 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: <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: Important or supplemental information.</p> <p>REMARQUE: <i>Renseignements importants ou complémentaires.</i></p>
	<p>TIP: Useful hint or advice.</p> <p>CONSEIL: <i>Astuce ou conseil utile.</i></p>


	<p>WAIT TIME: Describes an operation or process that requires an unusually long time to complete.</p> <p>TEMPS D'ATTENTE: <i>Décrit une opération ou un processus prenant un temps anormalement long à s'effectuer.</i></p>
---	---

Table 1-2. Special Icons Used in this Document

(Tableau 1-2. 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: <i>Indique les informations nécessaires à la prévention de blessures corporelles ou de mort d'un individu ou à la prévention des dommages aux équipements.</i></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: <i>Indique les informations nécessaires à la prévention de blessures corporelles ou de mort d'un individu à cause de la vitesse élevée de rotation de l'arbre.</i></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: <i>Indique les informations nécessaires à la prévention des blessures ou la mort d'une personne à cause d'une décharge électrique.</i></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: <i>Indique les informations nécessaires à la prévention des dommages oculaires à cause de la lumière d'un faisceau laser.</i></p>

Table 1-3. Safety Warning Icons Used in this Document

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

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

Table 1-4. Other Safety Warning Icons Used in this Document

(Tableau 1-4. Autres icônes d'avertissement de sécurité utilisées dans ce document)

1.9 Safety Labels (*Étiquettes de sécurité*)

The following specific safety warnings are found on labels attached to the motor unit and on the inside of the control unit (see Section 6.6.1).

Les avertissements de sécurité spécifiques suivants se trouvent sur les étiquettes apposées sur le bloc moteur et sur l'intérieur de l'unité de commande (voir section 6.6.1).



1.10 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 that 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 procédures particulières relatives à le rotateur.

	<p>WARNING:</p> <p>Failure to connect the third prong of the power cord to a proper earth ground may impair the protection provided by the system.</p> <p>AVERTISSEMENT:</p> <p><i>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.</i></p>
--	--

**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.

**WARNING:**

Rotating shaft.

Do not turn on the rotator or rotate the electrode shaft unless the enclosure window is secured to all four pins as shown below.

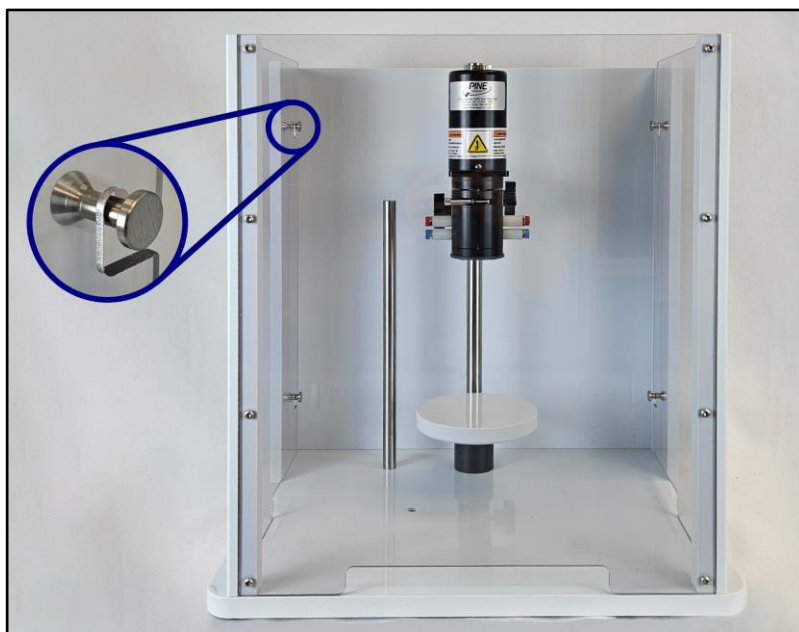
Use extreme caution when operating the rotator at rotation rates above 2000 RPM.

AVERTISSEMENT:

Arbre en rotation.

Ne mettez pas le rotateur en marche et ne marquez ni l'arbre de l'électrode en rotation si la fenêtre du boîtier n'est pas fermée à l'aide des quatre broches tel qu'indiqué ci-dessous.

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



**CAUTION:**

Do not exceed the maximum rotation rate for an electrode.

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

ATTENTION:

Ne dépassez pas la vitesse de rotation maximum pour une électrode.

Chaque type d'électrode rotative possède une vitesse de rotation maximum spécifique. Consultez la documentation pour l'électrode spécifique à utiliser pour connaître la vitesse de rotation maximum de l'électrode.

**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 the electrode shaft or 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 l'arbre de l'électrode ou 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 coupling.

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 correctement raccordé au moteur.

Inspectez l'arbre pour vous assurer qu'il est bien fixé.

**WARNING:**

Do not turn on the rotator or rotate an electrode tip if the electrode tip is not securely mounted in the electrode shaft.

Inspect the electrode tip to be certain that it is securely mounted.

AVERTISSEMENT:

Ne mettez pas le rotateur en marche ni un embout d'électrode en rotation si l'embout d'électrode n'est pas correctement fixée sur l'arbre de l'électrode.

Inspectez l'embout d'électrode pour vous assurer qu'elle est bien fixée.

**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 autre 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 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 tombée ou a été endommagée physiquement d'une autre manière ou d'une autre.

Inspectez l'embout d'électrode pour vous assurer qu'elle n'a pas été endommagée.

**WARNING:**

Do not use an electrode shaft that 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. Cet arbre est soit installé de manière incorrecte soit endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'arbre immédiatement.

**WARNING:**

Do not use an electrode tip that 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. Cet embout d'électrode est soit installée de manière incorrecte soit endommagée 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 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:

Lorsque vous montez ou descendez le bloc moteur le long de la barre principale, veillez à bien le tenir pour éviter qu'il ne chute brutalement et ne casse la cellule de verre située sous le bloc moteur.

**CAUTION:**

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

ATTENTION:

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

1.11 Electrostatic Discharge Information

Electrostatic discharge (ESD) is the rapid discharge of static electricity to ground. An ESD event occurs when two bodies of different potential approach each other closely enough such that static charge rapidly passes from one object to the next. Sensitive electronics in the path of the discharge may suffer damage. Damaging ESD events most often arise between a statically charged human body and a sensitive electronic circuit. The human body can easily accumulate static charge from simple movement from one place to another (*i.e.*, walking across a laboratory).

Instrument users must always be aware of the possibility of an ESD event and should employ good practices to minimize the chance of damaging the instrument. Some examples of good ESD prevention practices include the following:

- Self-ground your body before touching sensitive electronics or electrodes. Self-grounding may be done by touching a grounded metal surface such as a metal pipe.
- Wear a conductive wrist-strap connected to a good earth ground to prevent a charge from building up on your body.
- Wear a conductive foot/heel strap or conductive footwear in conjunction with standing on a grounded conductive floor mat.
- Increase the relative humidity in the air to minimize static generation.

The MSR evo has been tested and found to be compliant with FCC Part 15, Subpart B and European EN 61326-1:2013, ESD 61000-4-2:2009.

1.12 Hazardous Material Disclosures

Disclosure tables in both English and Mandarin Chinese are provided (see Table 1-5 and Table 1-6) that detail information pertaining to the list of hazardous substances classified under the Restriction of Hazardous Substances Directive (RoHS), as well as 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						
AFMSR24A1						
Part Name	Hazardous Substances					
	Lead (Pb)	Mercury (Hg)	Cadmium (Cd)	Hexavalent Chromium (Cr (VI))	Polybrominated biphenyls (PBB)	Polybrominated diphenyl ethers (PBDE)
Main PCB	X	O	O	O	O	O
Display PCB	O	O	O	O	O	O
Encoder PCB	X	O	O	O	O	O

This table is prepared in accordance with the provisions of SJ/T 11364

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.

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.

Note: the date of manufacture for this item AFMSR24A1 is coded in the serial number as follows:
wwyyFnn: ww indicates week; yy indicates year; and nn is the number of the item, starting with 01 each week.

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.

Table 1-5. Hazardous Materials Disclosure (English)

有害物质披露表						
AFMSR24A1						
	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)
主电路板	X	O	O	O	O	O
显示电路板	O	O	O	O	O	O
编码电路板	X	O	O	O	O	O

本表格依据 SJ/T 11364 的规定编制。

O: 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。

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。

注: 该部件 AFMSR24A1 的制造日期会按照以下格式出现在序列号码中:
wwyyFnn: 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。

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。

Table 1-6. Hazardous Materials Disclosure (Mandarin Chinese)

1.13 Software License

Purchase and operation of the MSR evo may involve the use of Pine Research's AfterMath software for various control, diagnostics, and/or analysis functions. The following software license pertains to use of AfterMath software.

PINE RESEARCH INSTRUMENTATION AFTERMATH DATA ORGANIZER/AFTERMATH BLUE SOFTWARE LICENSE

Pine Research Instrumentation, Inc. (hereafter "PINE") licenses purchasers (hereafter "LICENSEES") of Pine electrochemical products (hereafter "INSTRUMENTS") to use the AfterMath Data Organizer/AfterMath Blue software (hereafter "SOFTWARE") in conjunction with these INSTRUMENTS. This License contains the terms and conditions of use of the SOFTWARE.

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10. **Modifications, Taxes:** Any modification of this Agreement must be in writing and signed by PINE and LICENSEE. Terms and conditions set forth in any purchase order which differ from, conflict with, or are not included in this Agreement, shall not become part of this License unless specifically accepted by PINE in writing. LICENSEE shall be responsible for and shall pay, and shall reimburse PINE on request if PINE is required to pay, any sales, use, value added (VAT), consumption or other tax (excluding any tax that is based on PINE's net income), assessment, duty, tariff, or other fee or charge of any kind or nature that is levied or imposed by any governmental authority on the SOFTWARE.

LICENSEE HAS READ THIS AGREEMENT AND UNDERSTANDS AND AGREES TO ALL OF ITS TERMS AND CONDITIONS.

2 Description

2.1 Instrument Description

The MSR evo is a state-of-the-art electrode rotator that provides excellent steady-state control at constant rotation rates and also offers outstanding acceleration/deceleration control for those applications where the rotation rate must be modulated. The base rotation rate (for steady-state constant rate control) may be manually adjusted from 0 to 10,800 RPM by turning a ten-turn potentiometer knob located on the front panel of the control unit. When the rotator is running (front panel Status light is illuminated green), a built-in tachometer measures the actual rotation rate and this rate is continuously displayed on the front panel of the control unit. When the rotator is paused (front panel Status light is illuminated red), the display instead shows the set point. The MSR evo will not start rotating until the Run/Pause button is pressed. Manually turning the knob and observing the rotation rate is the most common way that rotation rate is controlled.

Automated control of the rotation rate is possible when the MSR evo is connected to a potentiostat system capable of supplying rotation rate control signal. While specific details vary from one system to another, the basic strategy is to use an analog signal output from a potentiostat that is proportional to the target rotation rate. This analog signal is carried by a cable (often supplied by the potentiostat manufacturer) to an 8-pin connector on the back of the MSR evo control unit. This connection permits the software that controls the potentiostat to also control the rotation rate using a constant voltage level (for steady-state rotation) or a more complex waveform such as a sine wave (for hydrodynamically modulated voltammetry).

The rotator is able to accurately follow complex waveforms and create the desired rotation rate response by using a 13,000 RPM, low inertia, permanent magnet DC motor in combination with a high voltage, bi-polar power supply. When adding a modulated input signal, the MSR evo can track and follow certain frequencies and amplitudes depending on the baseline rotation rate (MSR evo rotation rate limits: 0 to 10,800 RPM). For details on input signal modulation boundaries, see Section 2.2.1 or contact Pine Research directly.

The rotation rate is typically monitored by observing the front panel display on the control unit. In addition, the tachometer measurement can be monitored by connecting an oscilloscope, voltmeter, or other recording device to specific pins in the 8-pin connector on the back of the control unit. The voltage signal output from the tachometer is proportional to the rotation rate in the standard ratio of 1 RPM/mV. An optional selectable jumper inside the control unit may also be used to change the input ratio to either 2 RPM/mV or 4 RPM/mV if desired (see Section 6.7 for details). Note that while the input ratio can be adjusted to either of these three options, the output ratio is always 1 RPM/mV.

The control unit is connected to the motor unit using a 15-conductor cable with “straight thru” wiring terminated on each end with HD-15 connectors. The usual cable length is 152 cm (60 in), but longer cables may be available upon request.

The motor unit can be positioned vertically along a center post that is mounted in a sturdy and chemically-resistant enclosure base. A flat cell platform can also be positioned along the center post, making it easy to raise and lower the cell with respect to the motor unit. The electrochemical cell can be further secured by clamping it to a side post located adjacent to the center post.

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 can be removed to set up the cell, but the enclosure window must be securely mounted to the enclosure base before rotating the electrode.

The rotator may be used with rotating disk electrodes (RDEs), rotating ring-disk electrodes (RRDEs), and rotating cylinder electrodes (RCEs). Connections to the rotating electrode shaft are made by two pairs of silver-carbon brushes. For RDEs and RCEs, all four brushes make contact with the rotating shaft and may be shorted together to obtain four points of contact. For RRDEs, the upper brush pair contacts the disk electrode, and the lower pair contacts the ring electrode.

2.2 Instrument Specifications



INFO:

All specifications provided in this section are subject to change without notice.

MOTOR SPECIFICATIONS	
Motor Power	20 W
Supply Voltages	+30 VDC, -24 VDC
Motor Type	Brushed DC motor
Rate Control Method	Analog closed loop Temperature-compensated tachometer mounted on motor shaft
Maximum Continuous Torque	30.4 mN · m
Motor Protection	2 Amp thermal-type circuit breaker; current-limited power supplies
Modulation Boundaries	See Section 2.2.1 for details
ELECTRICAL	
Power Requirements	AC Mains: 100 – 240 VAC, $\pm 10\%$; 50/60 Hz; 2 A
Electrode Connections	Disk electrode: two red banana jacks on motor unit Ring electrode: two blue banana jacks on motor unit
Controls	Front panel: power switch, external/manual control switch, Run/Pause button Back panel: button to reset circuit breaker, 8-pin connector with external stop signal, external voltage in, and voltage out
Grounding Connections	Chassis connection on back panel 8-pin connector

(specifications table is continued on the next page)

ROTATION RATE	
Rate Limits	0 to 10,800 RPM
Rate Accuracy	100 to 200 RPM: accurate to within ± 2 counts of display reading 200 to 10,800 RPM: accurate to within $\pm 1\%$ of display reading
Rate Display	4 $\frac{1}{2}$ -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"
Control Switch (front panel)	Slide switch with LED indicator; light is on for external control, off for manual control
Rate Control (external)	Optional rate control via input signal on external I/O port Available input 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
Rate Output	Voltage output accessible from back panel 8-pin connector Output ratio is always 1 RPM/mV
PHYSICAL SPECIFICATIONS	
Shipping Information	Shipping weight: 60 lb (27 kg) Shipping dimensions: 24 x 24 x 24 in (61 x 61 x 61 cm)
Instrument Dimensions	Control unit: 10.8 x 9.8 x 4.3 in (27 x 25 x 11 cm) Rotator enclosure: 18.8 x 15.5 x 21.0 in (48 x 40 x 54 cm)
Materials	Enclosure base and sides: white polypropylene Enclosure window: clear polycarbonate Cell platform: white polypropylene Center post/Support rod: 303 stainless steel
Operating Temperature	10 to 40°C (50 to 104°F)

2.2.1 Modulation Boundaries

A modulated input signal can be summed with a baseline control signal to perform hydrodynamically modulated electrochemical techniques. Typically, a small sinusoidal waveform is applied to the input port on the back panel of the control box, and this sinusoidal modulation is superimposed on top of the baseline rotation rate. The MSR evo can track and follow these input signals within a specific range of maximum frequencies depending on the signal amplitude. Figure 2-1 shows the input signal modulation boundaries for amplitudes that are either 1%, 4%, or 10% of the baseline RPM.

For example, when applying a baseline rotation rate of 2500 RPM, and superimposing a sine wave modulation signal with an amplitude of 1% of the baseline (± 25 RPM peak), the maximum frequency that can be applied is 100 Hz. Similarly, with the same baseline rotation rate, if the superimposed signal amplitude is 10% of the baseline (± 250 RPM peak), the maximum possible input frequency is 70 Hz. These data are shown along the boundaries of the 1% and 10% regions of Figure 2-1. The remaining data show the combinations of baseline rotation rates and maximum frequencies that are possible for sinusoidal signal amplitudes that are either 1%, 4%, or 10% of the baseline RPM.

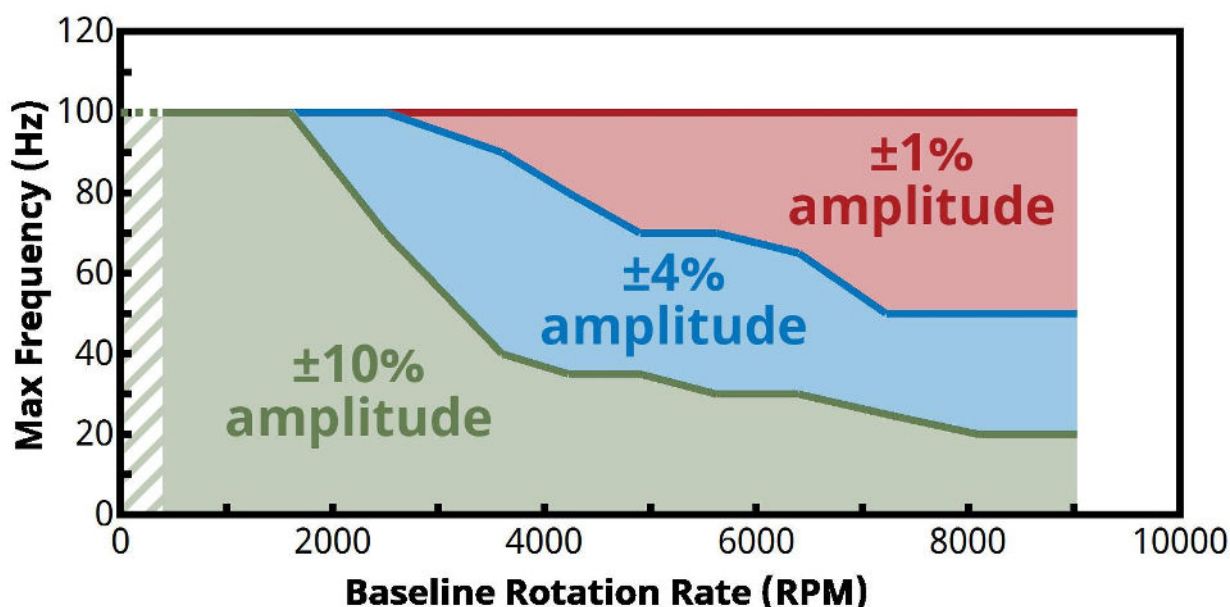


Figure 2-1. MSR evo Input Signal Modulation Boundaries

Note that the data shown in Figure 2-1 are without an added load on the rotator; that is, these data represent the fastest possible frequencies that can be applied to modulated input signals intrinsic to the MSR evo. Beyond these frequencies, circuit protection features will throttle the output signal and make modulation experiments inaccurate. Additionally, actual limits when using a shaft and a real electrochemical setup may vary slightly due to added load on the motor.

The modulation boundary limits for the MSR evo were interrogated in accordance with published standards, including the popular *Electrochemical Methods: Fundamentals and Applications* textbook by Bard and Faulkner, as well as the famous works of (among others) Albery, Bruckenstein, Deslouis, and Tribollet (select bibliography is provided below).

1. AJ Bard and LR Faulkner, *Electrochemical Methods: Fundamentals and Applications*, 3rd Edition, John Wiley & Sons, New York (2022), Section 10.5, pp 435-442.
2. K Tokuda, S Bruckenstein, and B Miller, *J. Electrochem. Soc.*, 122 (1975) 1316.
3. C Deslouis, I Epelboin, C Gabrielli, and B Tribollet, *J. Electroanal. Chem.*, 82 (1977) 251-269.
4. WJ Albery, AR Hillman, and S Bruckenstein, *J. Electroanal. Chem.*, 100 (1979) 687-709.
5. C Deslouis, C Gabrielli, PS Fanchine, and B Tribollet, *J. Electrochem. Soc.*, 129 (1982) 107.
6. C Deslouis and B Tribollet, *Electrochim. Acta*, 35 (1990) 1637-1648.

2.3 MSR evo Packing Details

The MSR evo, as shipped from the production facility, comes in a large cardboard box and is packed in the manner shown below (see Figure 2-2):



Figure 2-2. Packed MSR evo as Received from Factory

2.4 Major System Components

The major system components of the MSR evo are shown below (see Figure 2-3):

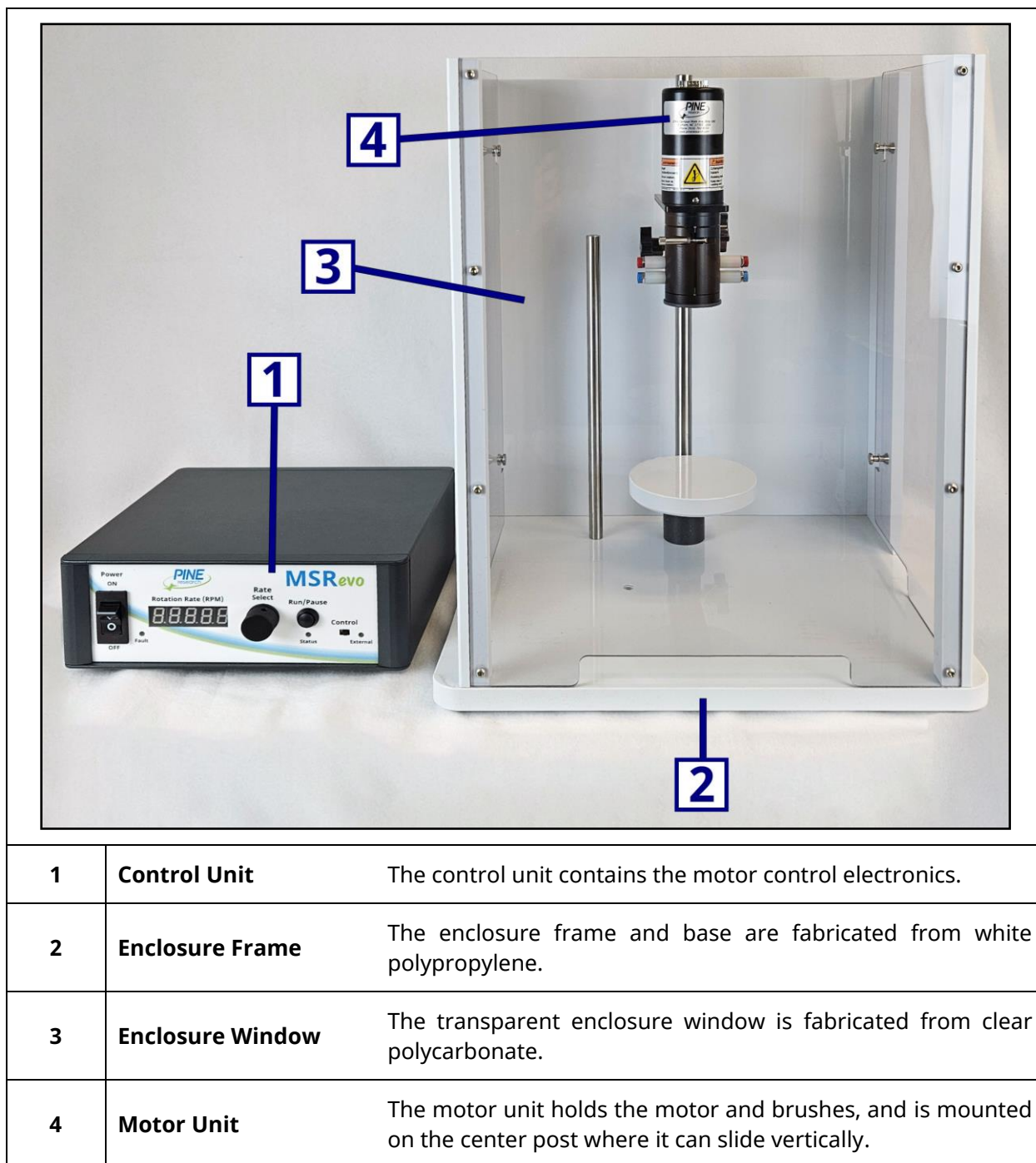


Figure 2-3. Major Components of the MSR evo Rotator System

2.5 MSR evo Enclosure Components

The enclosure included with the MSR evo (see Figure 2-4) is critical for safe operation of the instrument, and must be securely installed prior to conducting experiments (see Section 1.10 for general safety warnings).

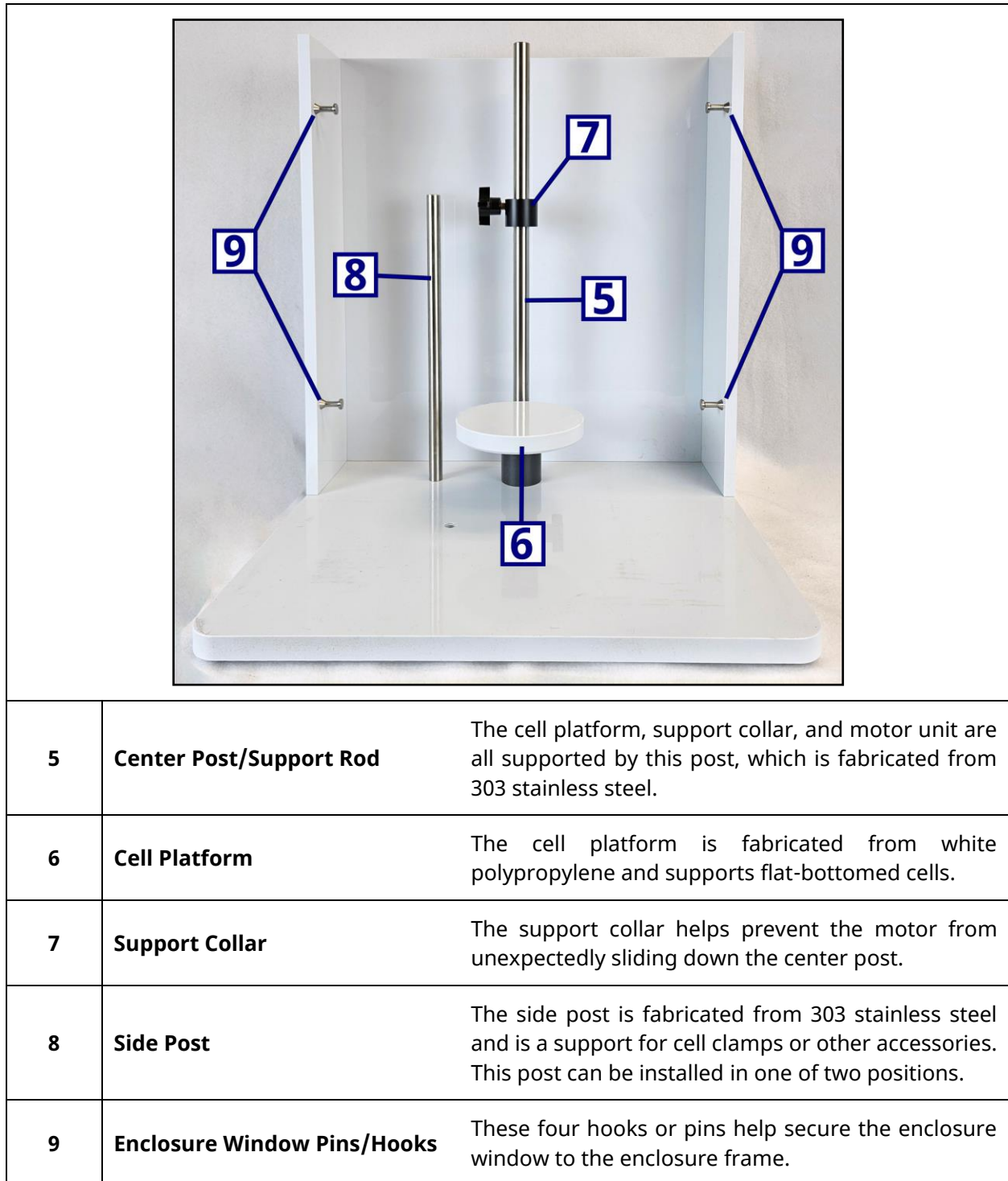


Figure 2-4. MSR evo Enclosure Components

2.6 Motor Unit Components

The MSR evo motor unit (see Figure 2-5) includes the following components:

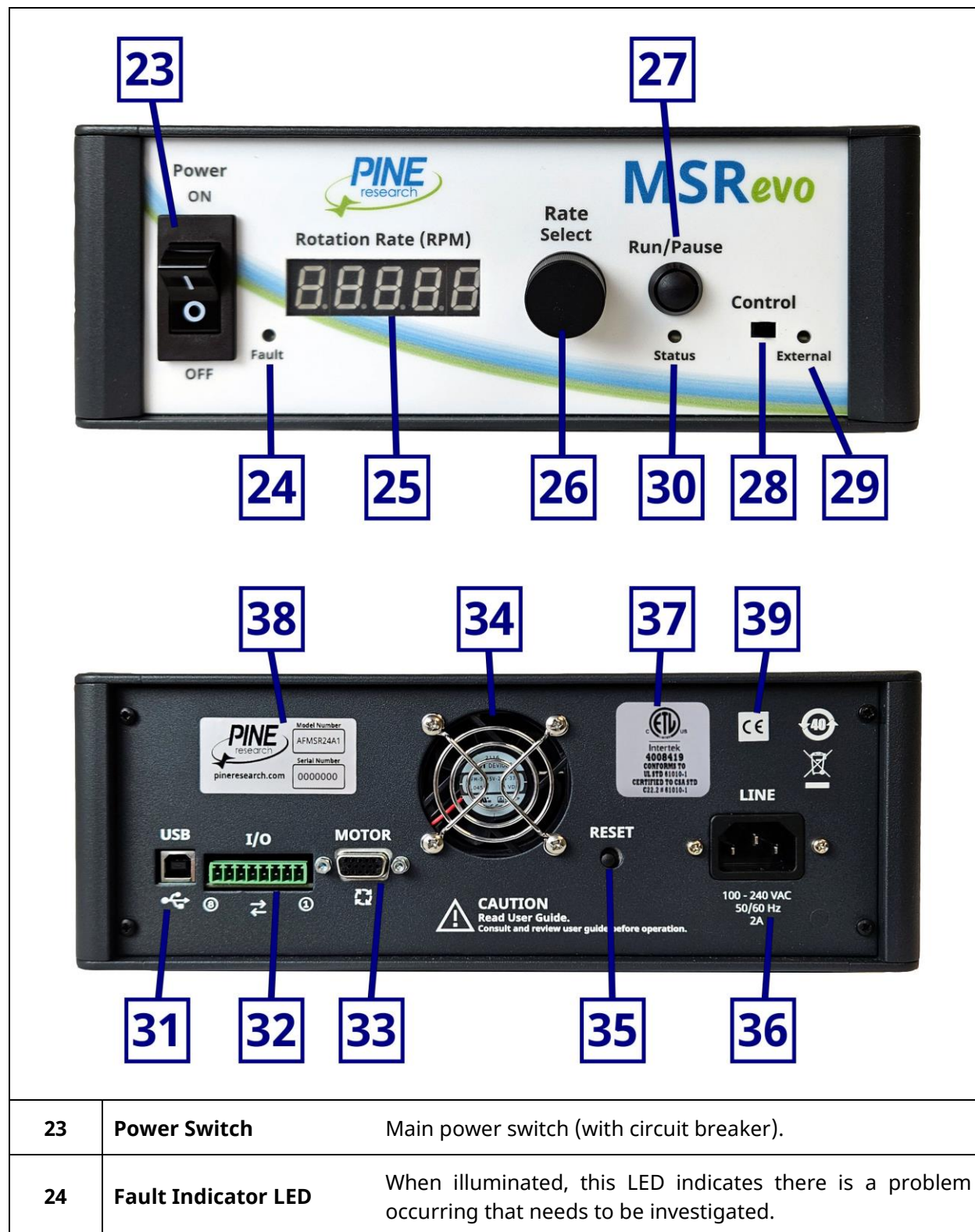


10	Motor Control Cable	This cable connects the control unit to the motor unit.
11	Motor Cable Connector	Accepts one end of the motor control cable.
12	Upper Brush Pair (red)	These upper two spring-loaded silver-carbon brushes press on opposing sides of the rotating shaft and make electrical contact with the disk electrode when using an RDE or RRDE tip, or the cylinder electrode when using an RCE.
13	Lower Brush Pair (blue)	These lower two spring-loaded silver carbon brushes press on opposing sides of the rotating shaft and make electrical contact with the ring electrode when using an RRDE tip.
14	Lift Knob	This knob should be loosened when moving the motor unit up and down the center post/support rod. Tighten the knob to secure the motor unit in place.
15	Motor Housing	This aluminum housing shields the electrochemical cell from any electromagnetic interference from the motor.
16	Clamshell Doors	These doors open to permit access to the brush chamber.
17	Door Latch	Secures clamshell doors in the closed position during operation.
18	Brush Contact	Spring-loaded silver-carbon brush that provides electrical contact with the rotating shaft and electrodes.
19	Motor Coupling	Used to attach the shaft to the motor.
20	Motor Coupling Hex Screw Pair	Hex screws located on either side of the motor coupling tighten to hold the shaft securely inside the motor coupling.
21	Rotating Shaft	The top end of the rotating shaft (sold separately) is mounted in the motor coupling and the electrode tip or cylinder (sold separately) is located at the bottom end.
22	Lower Bearing Assembly	An easily-replaceable bearing assembly stabilizes the rotating shaft at the point where the shaft exits the motor unit.

Figure 2-5. MSR evo Motor Unit Components

2.7 Control Unit Components

The MSR evo control unit (see Figure 2-6) includes the following components:



25	Rotation Rate Display	4 ½-digit display of rotation rate (RPM).
26	Rate Control Knob	10-turn knob used to manually adjust rotation rate.
27	Run/Pause Button	This button is a toggle control that alternates between pausing the rotation of the motor and allowing it to continue running.
28	Control Switch	This switch toggles the rotator between manual and external control modes.
29	External Control Indicator LED	When illuminated, this LED indicates the MSR evo rotation rate can only be controlled via external input.
30	Status Indicator LED	This LED provides information regarding the rotator status specifically related to its Run/Pause state.
31	External USB Port	This USB port may optionally allow serial communication with other devices.
32	External I/O Port	This 8-pin connector allows the MSR evo to be connected to and controlled by an auxiliary instrument (such as a potentiostat).
33	Motor Cable Connector	Accepts one end of the motor control cable.
34	Cooling Fan	This fan cools the control unit electronics during operation.
35	Circuit Reset Button	This button resets the motor protection circuitry.
36	Power Cord Connector	Connects to external electrical power cord.
37	NRTL Mark	MSR evo systems that bear the ETL/Intertek mark are listed by Intertek to UL/IEC 61010-1:2010Ed.3+A1. Intertek is a Nationally Recognized Testing Laboratory (NRTL) recognized by the United States Occupational Safety and Health Administration (OSHA).
38	Serial Number Label	This label indicates the make, model, and serial number of the rotator.
39	European CE Mark	MSR evo systems that comply with one or more EU directives bear the CE mark. See the “CE Declaration of Conformity” attached to the end of this user guide for more details.

Figure 2-6. MSR evo Control Unit Components

The External I/O Port on the back panel of the MSR evo Control Unit can be used both to monitor the rotation rate (via a voltage output signal) and/or to control the rotation rate (via a voltage input signal). There are eight pins on the External I/O Port (see Figure 2-7). The control signal for the rotation rate is typically generated by a potentiostat (see Section 4.6.3).

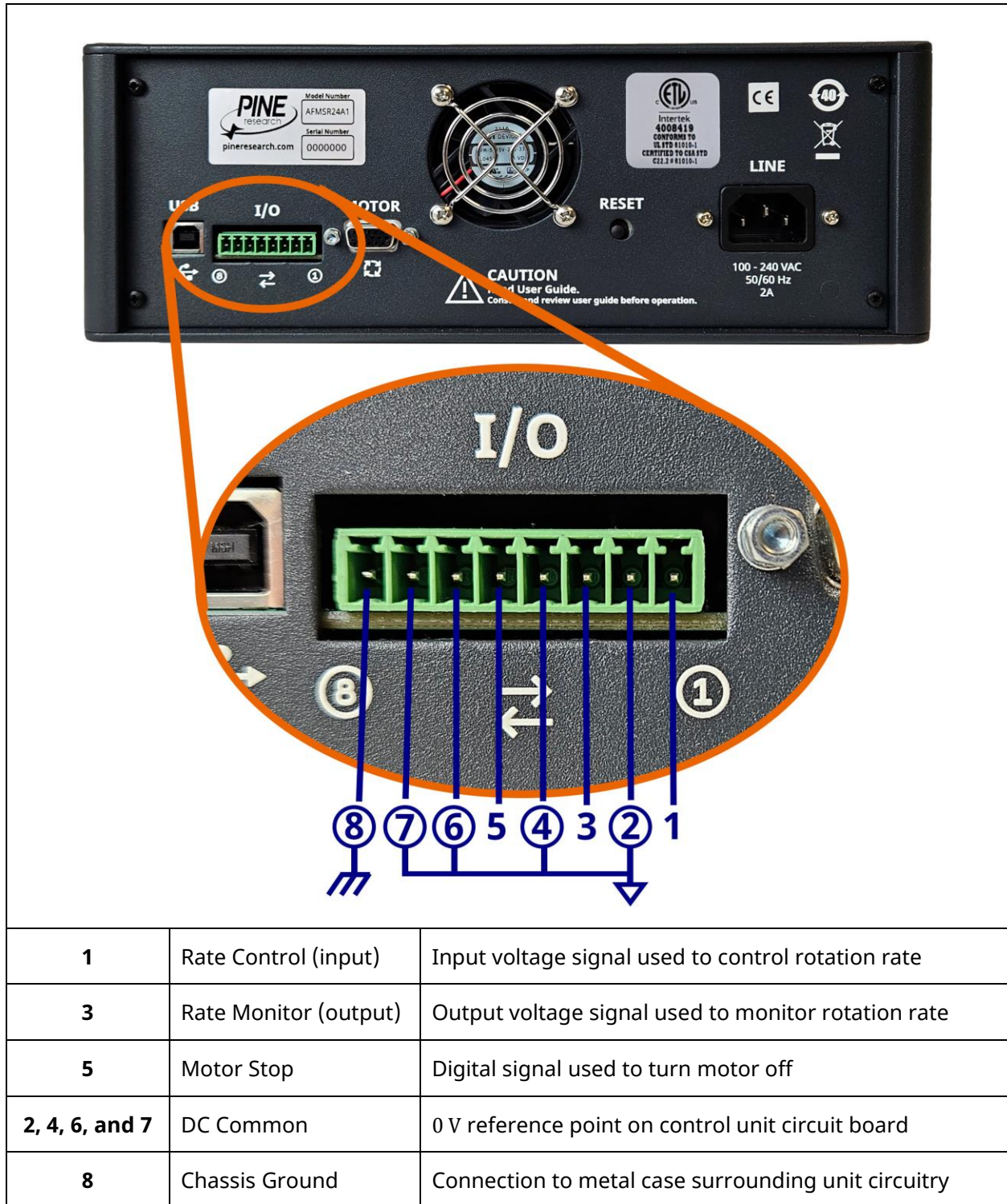


Figure 2-7. Pinout of External I/O Port on MSR evo Control Unit Back Panel

2.8 Typical Rotating Disk Electrode (RDE) Design

Most rotating disk electrodes (RDEs) consist of two parts, a shaft and a tip, but in some cases the entire electrode and shaft may be a single piece. In certain cases, the RDE tips are a single piece, Fixed-Disk design where the disk cannot be physically separated from the shroud. In other cases, the RDE tips are designated as ChangeDisk and the disk can be taken in and out of the shroud repeatedly (see Figure 2-8).

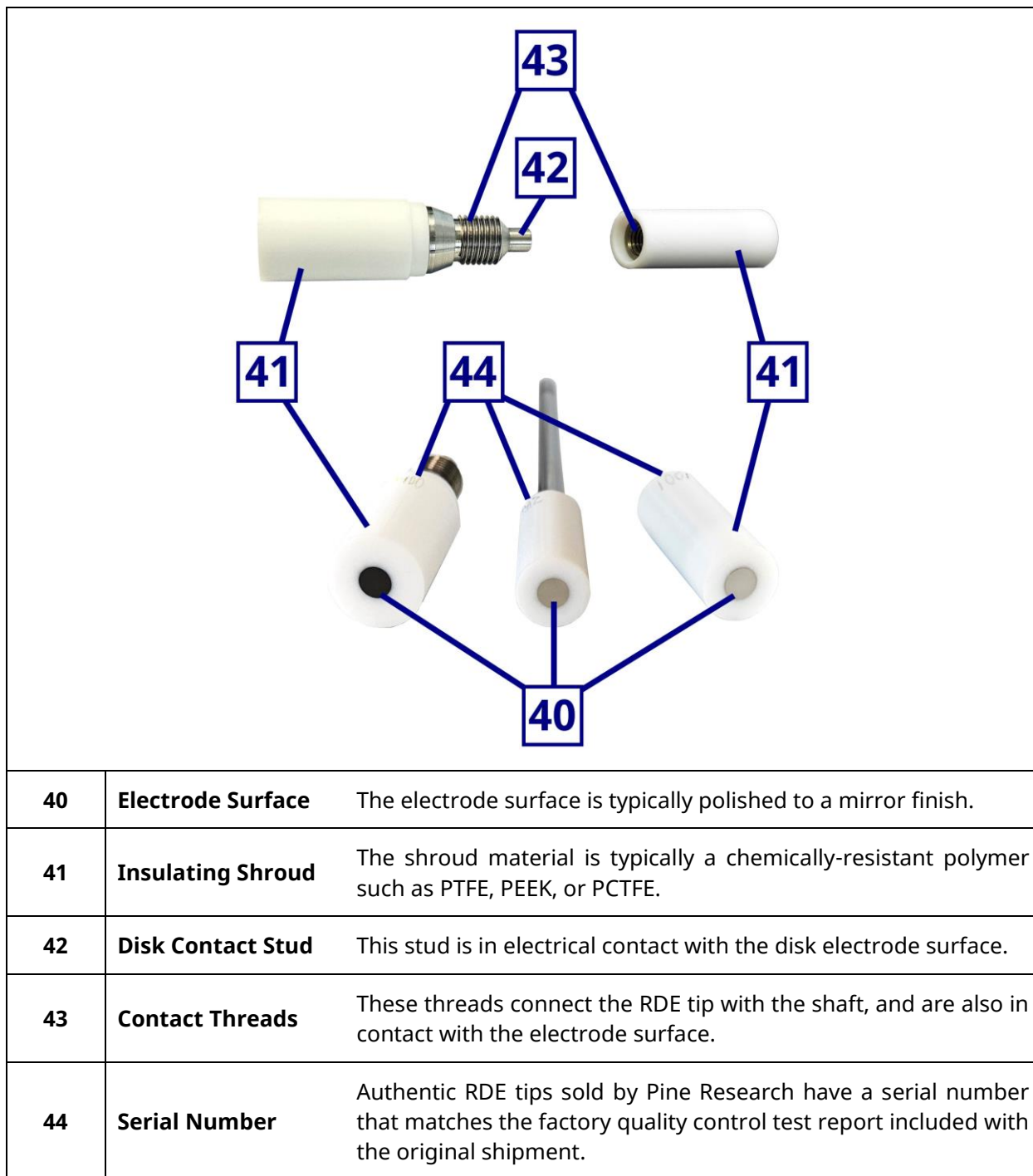


Figure 2-8. Typical Rotating Disk Electrode (RDE) Tips

2.9 Typical Rotating Ring-Disk Electrode (RRDE) Design

Rotating ring-disk electrodes (RRDEs) are designed either as a single piece, Fixed-Disk design where the disk cannot be physically separated from the shroud, or as ChangeDisk where the disk can be taken in and out of the shroud repeatedly. In both cases, the ring is permanently fixed (see Figure 2-9).

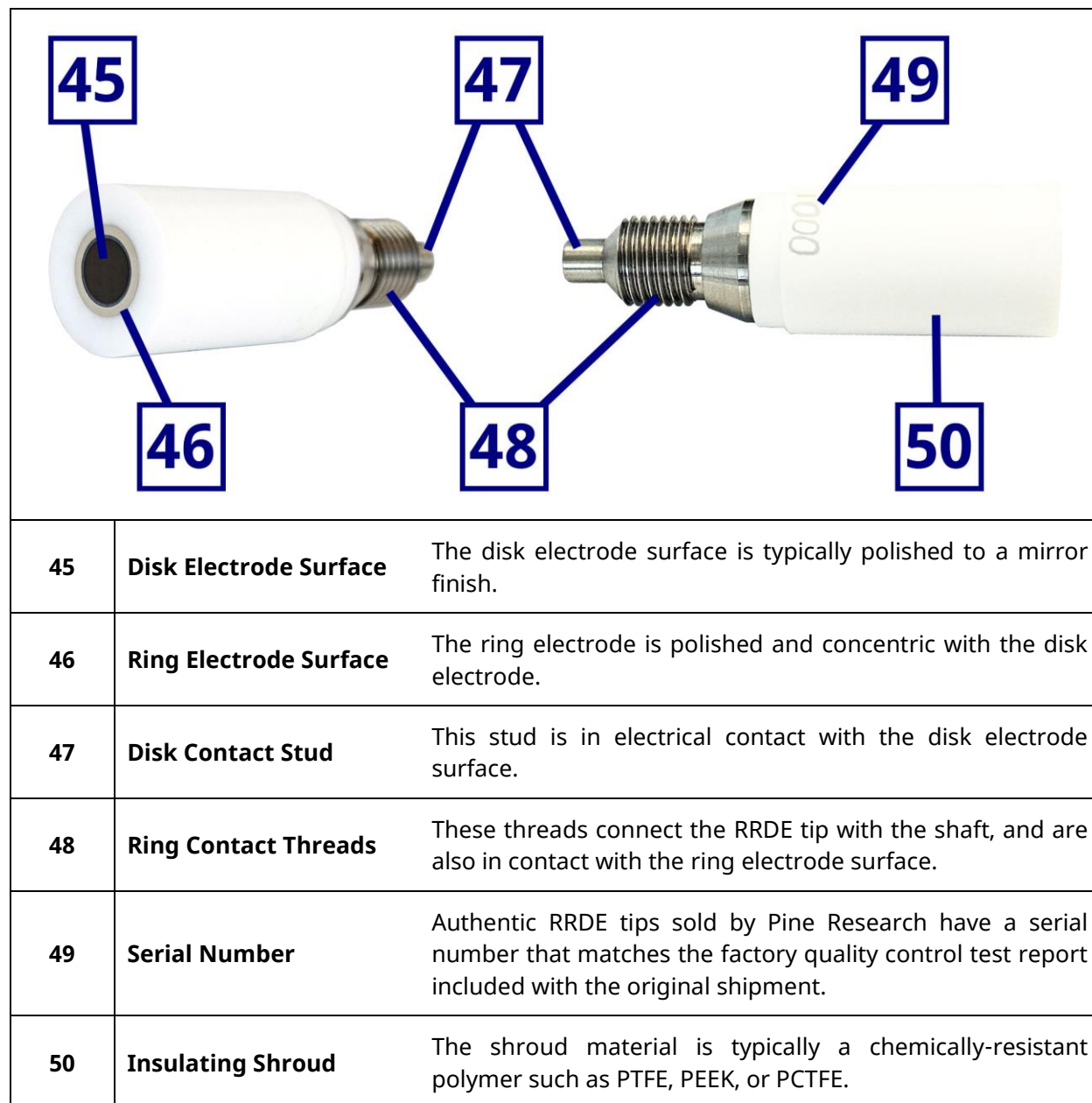


Figure 2-9. Typical Rotating Ring-Disk Electrode (RRDE) Tips

3 Installation

3.1 Site Preparation

The rotator system should be located on a sturdy and level surface (*e.g.*, a laboratory bench) with ample clearance above and around the perimeter of the rotator enclosure. The front of the rotator should be unobstructed, and there should be at least 20 cm clearance on each side and behind the rotator, for a total table space of about 80 cm x 90 cm. The location should also include enough space for the control unit (30 cm x 30 cm) and vertical clearance to easily raise and lower the motor unit.

3.2 Unpacking and Setting Up the Rotator



NOTE:

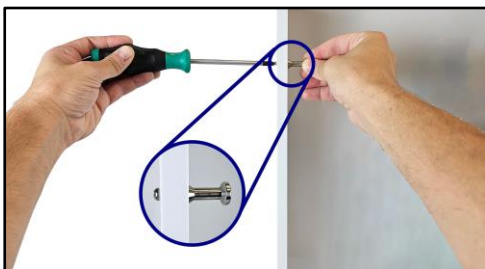
The numbers in parentheses in the installation instructions below correspond to the numbering used in the tables and figures found in Section 2 of this manual.



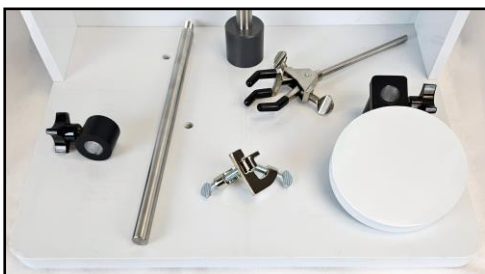
Inspect the contents of the shipping carton. Remove the top piece of cardboard to reveal the two smaller boxes in the carton. The Control Unit **(1)** is packed inside the larger box, and the smaller box holds additional components. Remove both boxes and set them aside. Next, carefully remove the Enclosure Window **(3)** and the Enclosure Frame **(2)** from the box. The Center Post/Support Rod **(5)** is pre-installed in the enclosure base.



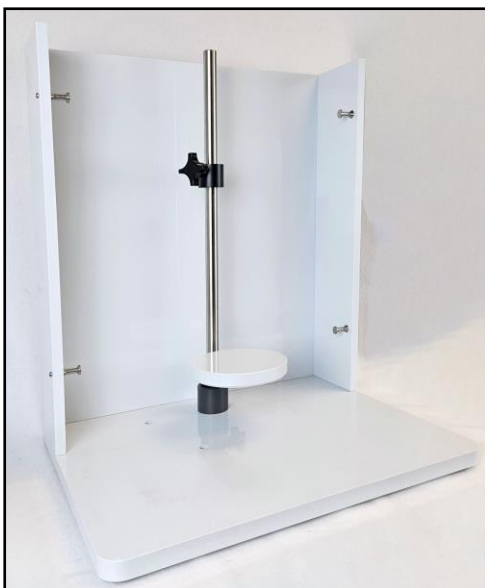
Open the smaller box. It should contain the Motor Unit **(4)**, the Support Collar **(7)**, the Cell Platform **(6)**, the Side Post **(8)**, a standard three-pronged laboratory clamp (with right-angle mount), and a small bag containing two banana cables and some additional assembly hardware.



Locate the small bag of hardware. Remove the four Enclosure Window Pins/Hooks **(9)** and four screws. Place each screw in one of the pre-drilled holes along the side walls of the enclosure, two on the left and two on the right. Install the pins onto the screws. Properly installed pins will point inwards as shown.



Locate the Support Collar **(7)**, Side Post **(8)**, three-pronged laboratory clamp (with right-angle mount), Cell Platform **(6)**, and large plastic washer (usually shipped in the hardware bag).



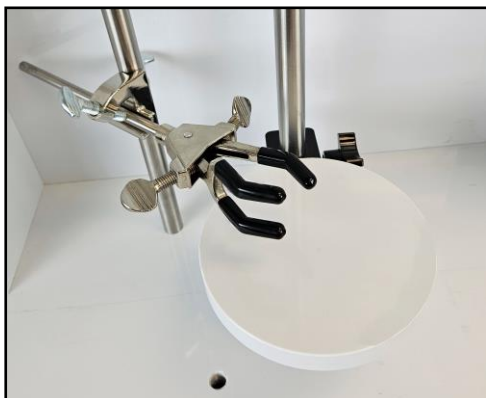
Slide the Cell Platform **(6)** onto the Center Post/Support Rod **(5)** and position it near the bottom with the platform facing up. Tighten the knob to secure the Cell Platform **(6)** to the Center Post/Support Rod **(5)**. Next, slide the Support Collar **(7)** on to the Center Post/Support Rod **(5)** and position it slightly above the midpoint with the knob on the left side. Tighten the knob to secure the Support Collar **(7)**. Slide the plastic washer on the Center Post/Support Rod **(5)** and allow it to rest on top of the Support Collar **(7)**.



Carefully slide the Motor Unit **(4)** on to the Center Post/Support Rod **(5)** until it rests on the Support Collar **(7)**. Tighten the knob to secure the Motor Unit **(4)** to the Center Post/Support Rod **(5)**.

**NOTE:**

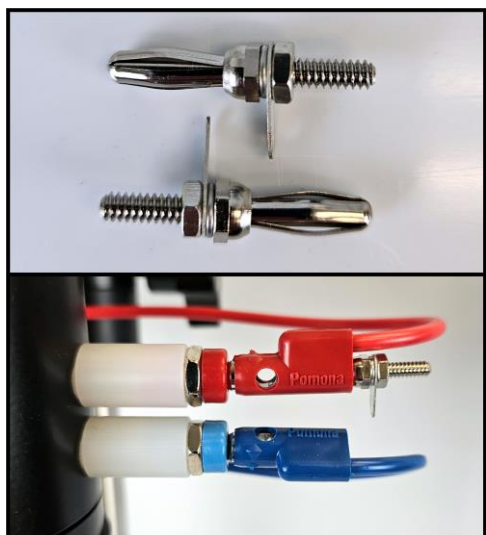
The relative vertical positions of the cell platform, support collar, and motor unit may be adjusted as needed to fit the specific size and shape of a particular electrochemical cell.



There are several holes in the floor of the Enclosure Frame (2) that are threaded to accept the Side Post (8). Choose one of these holes and install the side post in it. A laboratory clamp can then be optionally installed onto the Side Post (8).



There are two short banana cables (red and blue) that serve as optional jumpers between the left and right brush connections. If desired, use the red cable to connect the upper (red) pair of brush connections, and use the blue cable to connect the lower (blue) pair of brush connections, running the wires behind the Motor Unit (4) assembly as shown.



If needed, included banana plug studs can be inserted into the red and blue banana cables. These flat studs are an ideal place to make connections using alligator clips.

The upper (red) jacks make electrical contact with a rotating disk electrode (RDE) or a rotating cylinder electrode (RCE) tip.

When using a rotating ring-disk electrode (RRDE), the upper (red) jacks make contact with the disk, and the lower (blue) jacks make contact with the ring.



Remove the Control Unit **(1)** from the box and place it next to the Enclosure Frame **(2)**. Plug the male end of the Motor Control Cable **(10)** into the Motor Cable Connector **(33)** on the back of the Control Unit **(1)**, and plug the female end of the cable into the Motor Cable Connector **(11)** on the top of the Motor Unit **(4)**.

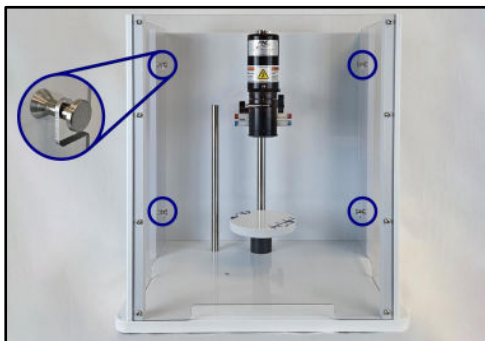
CAUTION:

The connectors on both ends of the motor control cable **MUST** be firmly secured by tightening the pair of screws on each connector. Failure to secure the connectors may result in improper control of the rotation rate, as well as illumination of the fault indicator LED on the front panel of the control unit (see Section 4.8.1 for more details)



ATTENTION:

Les connecteurs situés aux deux extrémités du câble de commande du moteur DOIVENT être fermement attachés en serrant les deux vis de chaque connecteur. Une mauvaise fixation des connecteurs entraînera un mauvais contrôle de la vitesse de rotation, ainsi que l'allumage de l'indicateur de défaut sur le panneau avant de l'unité de contrôle (voir Section 4.8.1 pour plus de détails)



Attach the Enclosure Window **(3)** by hooking it onto the four Enclosure Window Pins/Hooks **(9)**. The Enclosure Window **(3)** will rest securely on the Enclosure Frame **(2)**.



An appropriate international power cord (10 A rating) may be provided with the rotator. Use this cord to connect the Control Unit **(1)** to the local power supply (AC Mains).

The local power supply should provide an earth ground connection for the third prong on the power cord.

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.

CAUTION:



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

ATTENTION:

Un cordon d'alimentation amovible est fourni avec le rotateur. Ne remplace 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 MSR ev0. Users of the rotator should be familiar with all of the information in this section prior to operating the rotator.

4.1 The Rotating Shaft

The electrode shaft normally rotates in a clockwise direction as viewed from the top of the rotator. The upper end of a standard shaft has a 1/4" (6.35 mm) outer diameter. When properly mounted in the rotator, the upper 2.7" (68 mm) of the shaft is inside the motor unit, while the remaining length of the shaft extends down below the motor unit.

The rotator accepts shafts for use with Rotating Disk Electrodes (RDE), Rotating Cylinder Electrodes (RCE) or Rotating Ring-Disk Electrodes (RRDE). Electrical connection is accomplished using one or more silver-carbon brushes to contact metal surfaces on the upper portion of the rotating shaft. Each shaft is specially-designed to provide one or two current paths down to the end of the shaft where the electrodes are located. These current paths are electrically-isolated from the mounting area at the top of the shaft.

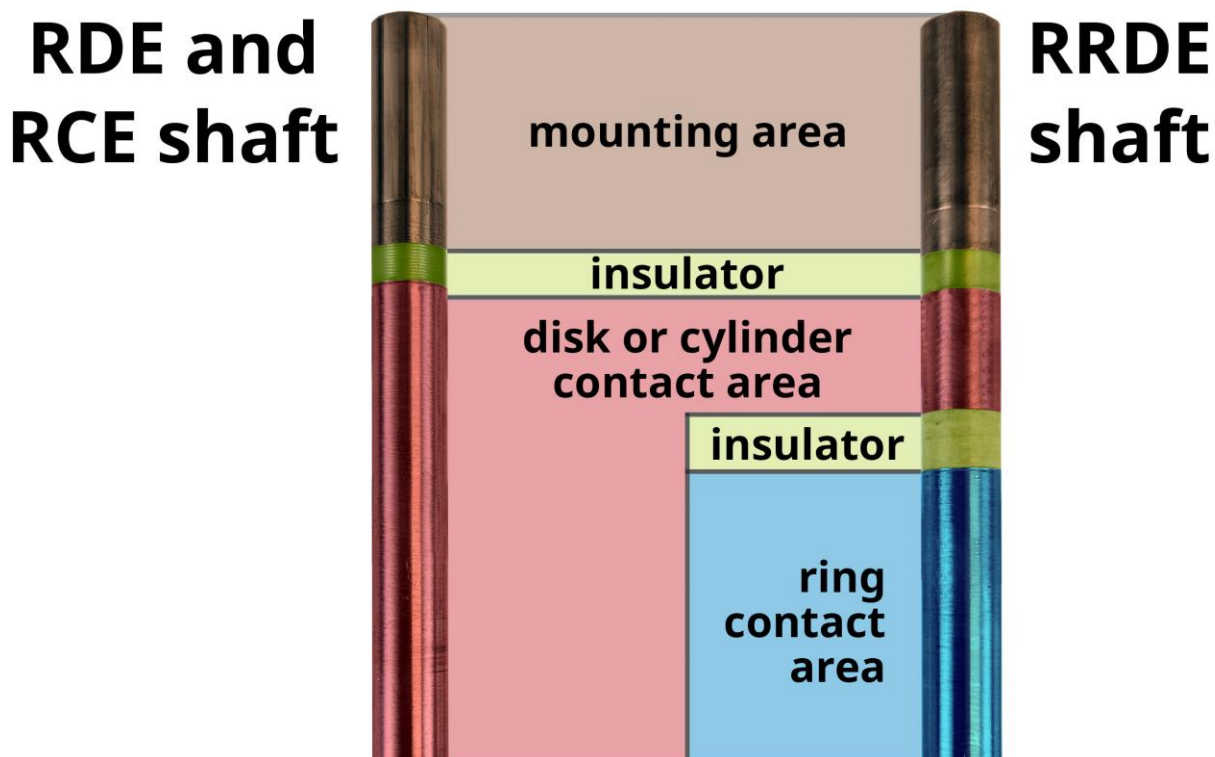


Figure 4-1. Contact Areas at the Top of Rotating Electrode Shafts

The uppermost portion of the shaft is used to mount the shaft into the rotator (see Figure 4-1). This mounting area is electrically-isolated from the remainder of the shaft so that the electrode connections remain isolated from the rotator chassis. An insulating spacer just below the mounting area isolates the mounting area from the electrode contact area.

For an RDE or RCE shaft (see Figure 4-1, left), the entire metal exterior of the shaft below the insulating spacer is in electrical contact with the disk (or cylinder) electrode. For an RRDE shaft (see Figure 4-1, right), there are two insulating spacers. The portion of the shaft between the two insulating spacers provides electrical contact with the disk electrode. The lower portion of the shaft (below the lower insulating spacer) provides electrical contact with the ring electrode.



Figure 4-2. MSR ev0 Brush Chamber (Side View)

The shaft is connected to the rotator motor via a brass motor coupling located inside the brush chamber (see Figure 4-2). Two clamshell doors surround the brush chamber. These doors are securely latched during rotator operation and push two pairs of contact brushes against the rotating shaft. The upper (red) pair of brushes makes contact with the disk (or cylinder) while the lower (blue) pair makes contact with the ring on a rotating ring-disk electrode.

4.1.1 Installing a Shaft



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 the electrode shaft or 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 l'arbre de l'électrode ou 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 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 autre manière ou d'une autre.

Inspectez l'arbre pour vous assurer qu'il n'a pas été endommagé.



Tip:

It is often easier to remove or install a shaft by disconnecting the motor control cable and inverting the entire motor unit on the center post/support rod. Several photos in this section show the MSR evo motor unit in this inverted position.



Tip:

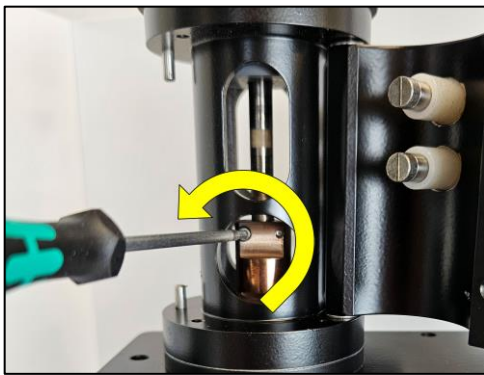
Do not lose the white plastic washer on the door latch!



Invert the orientation of the motor unit so that it is upside-down as shown.

Loosen the latch on the clamshell doors.

Open the doors to provide access to the brush chamber.



If there is a shaft already installed, use the hex driver tool (5/64", provided) to loosen the two screws on the motor coupling. Do not remove these screws entirely. Loosen them by one or two turns of the hex driver only. Usually it is necessary to hold the motor coupling in place with one hand while loosening the screws with the other hand.



Note:

A new rotator has tape around the motor coupling to protect the hex screws. Remove this tape and loosen the hex screws if needed to allow the shaft to enter the coupling.



Tip:

Apply a small amount of a silicon-based grease to the top of the shaft before installing the shaft into the motor coupling. This helps to prevent the shaft from sticking in the coupling.

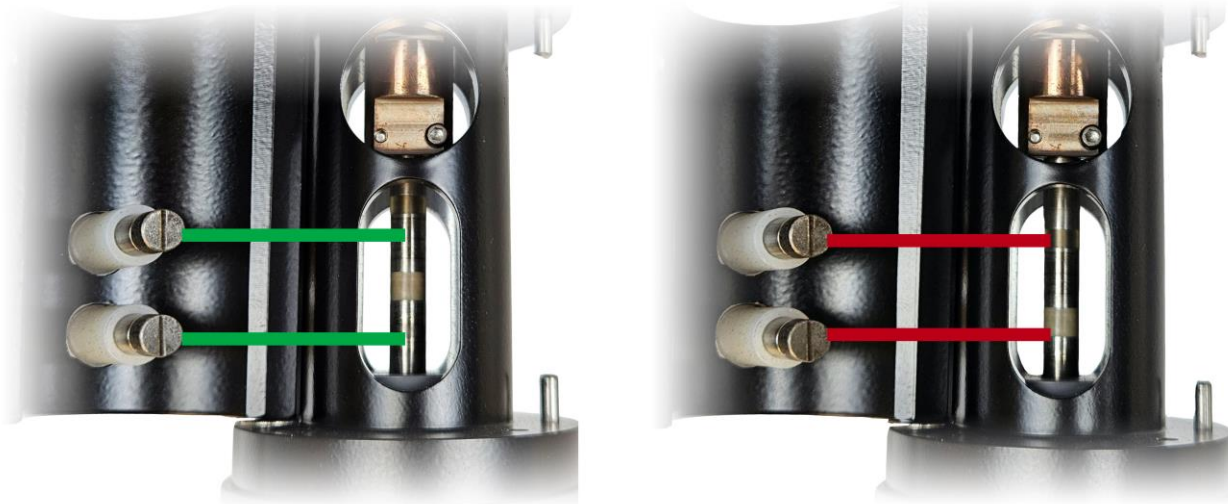
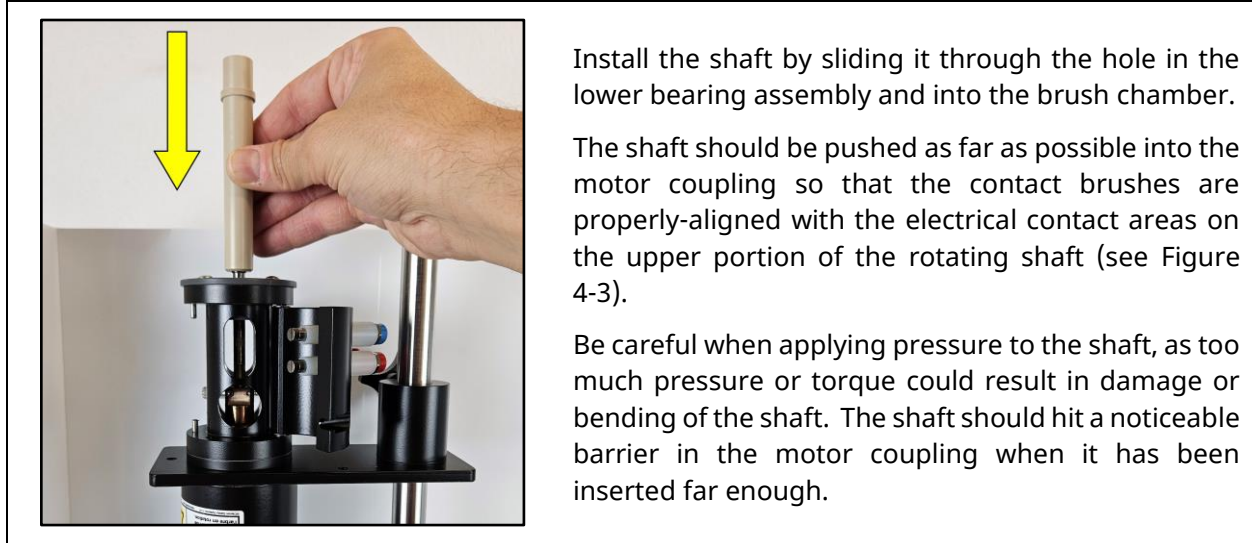
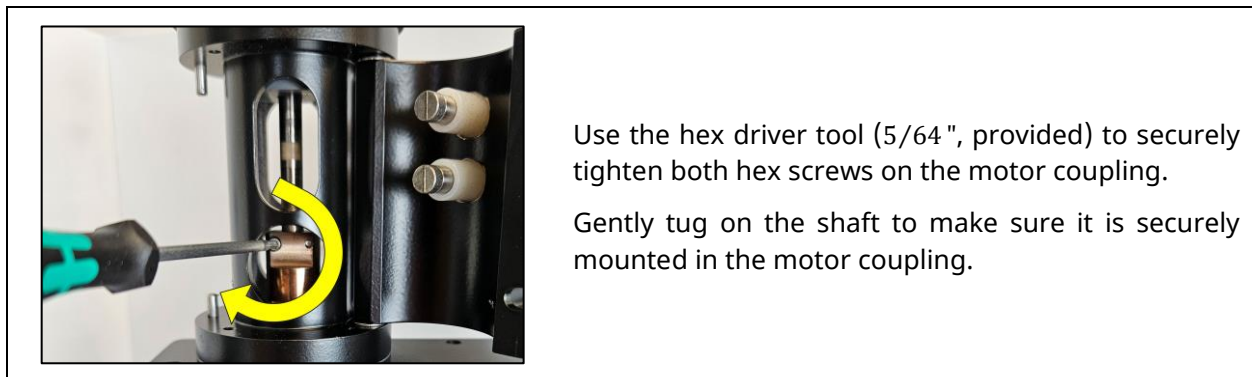
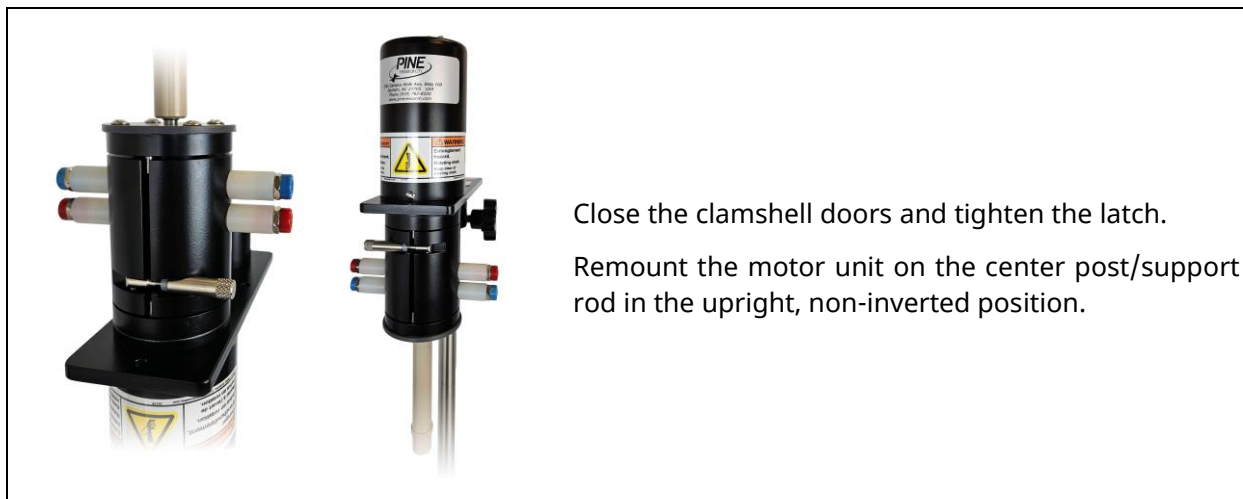


Figure 4-3. Proper (left) and Improper (right) Shaft Insertion Positions





Close the clamshell doors and tighten the latch.

Remount the motor unit on the center post/support rod in the upright, non-inverted position.

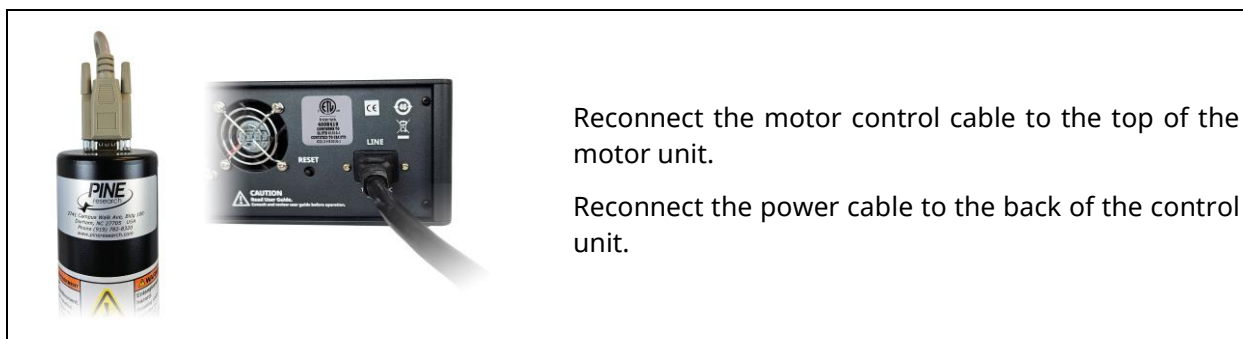


CAUTION:

Before reconnecting the rotator power cable or the motor control cable to the control unit, be sure the control unit power switch is off.

ATTENTION:

Avant de reconnecter le câble d'alimentation du rotateur ou le câble de commande du moteur à l'unité de commande, assurez-vous que l'interrupteur de l'unité de commande est en position éteinte.



Reconnect the motor control cable to the top of the motor unit.

Reconnect the power cable to the back of the control unit.



WARNING:

Do not turn on the rotator or rotate the electrode shaft if the shaft is not securely mounted in the motor coupling.

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 correctement raccordé au moteur.

Inspectez l'arbre pour vous assurer qu'il est bien fixé.



Turn on the control unit. Adjust the rotation rate knob until the set rate is between 100 and 200 RPM, and press the Run/Pause button to begin the rotation of the shaft.

While the shaft is slowly rotating, inspect the rotating shaft to assure that it is rotating properly about the axis of rotation. If the shaft is wobbling, vibrating, or tilting away from the axis of rotation, then turn off the rotator and remove the shaft.



Note:

A “Precision Shaft Alignment Kit” is available separately. This kit includes a dial indicator used to measure the “runout” at the end of the shaft (see Section 7.1 for kit part number).



WARNING:

Do not use an electrode shaft that 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. Cet arbre est soit installé de manière incorrecte soit endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'arbre immédiatement.

4.1.2 Changing the Electrode Tip on a Shaft



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 the electrode shaft or 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 l'arbre de l'électrode ou 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 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 autre 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 or otherwise physically damaged.

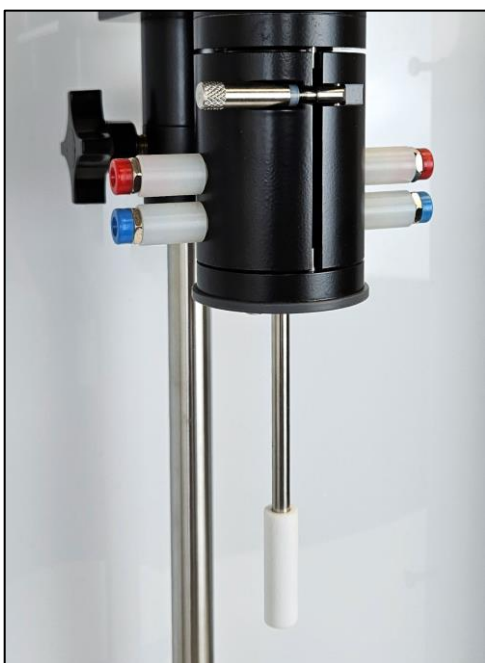


Inspect the electrode tip to be certain that it is not damaged.

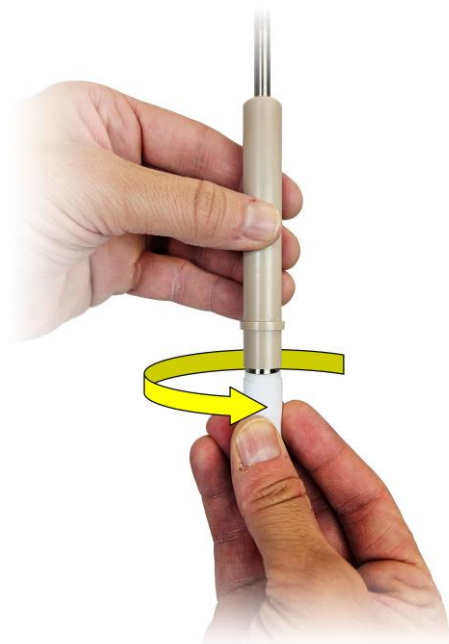
AVERTISSEMENT:

N'utilisez pas et ne tentez pas de mettre en rotation un embout d'électrode qui est tombée ou a été endommagée physiquement d'une autre manière ou d'une autre.

Inspectez l'embout d'électrode pour vous assurer qu'elle n'a pas été endommagée.



Some shafts are single-piece designs where the electrode tip is permanently attached at the bottom. In these cases, there is no additional step to disconnect or connect the electrode tip. Installing the shaft as described in Section 4.1.1 includes a concurrent connection to the electrode tip.

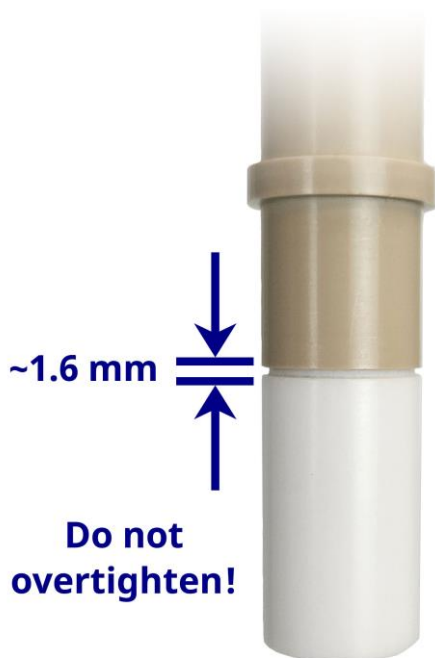


In most cases, the shaft is designed to accept a variety of different electrode tips. For these “shaft and tip” designs, the shaft may remain mounted in the rotator, and changing the tip is a simple matter of unscrewing one tip and then threading a new tip onto the shaft.

When removing or installing an electrode tip, use one hand to prevent the shaft from rotating while using the other hand to gently turn the electrode tip.

Remove the old electrode tip from the shaft by gently unscrewing it by hand. No tools are required to remove an electrode tip from a shaft.

Thread the new electrode tip onto the shaft and gently tighten it by hand. Never use a tool to tighten an electrode tip onto a shaft.



For RDE or RRDE electrode tips with 15 mm OD shrouds, there will be a small but visible gap between the tip and shaft. This visible gap is approximately 1.6 mm thick and is part of the design. Do not overtighten the electrode tip in an effort to close this gap.

Note, again, that this gap is specific to 15 mm OD shrouded electrode tips and shafts only.

**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 the factory for further instructions.

ATTENTION:

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

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

Si un embout d'électrode ne peut être retirée d'un arbre manuellement, communiquez avec l'usine pour obtenir des instructions supplémentaires.

**CAUTION:**

Before reconnecting the rotator power cable or the motor control cable to the control unit, be sure the control unit power switch is off.

ATTENTION:

Avant de reconnecter le câble d'alimentation du rotateur ou le câble de commande du moteur à l'unité de commande, assurez-vous que l'interrupteur de l'unité de commande est en position éteinte.



Reconnect the motor control cable to the top of the motor unit.

Reconnect the power cable to the back of the control unit.

**WARNING:**

Do not turn on the rotator or rotate the electrode shaft if the shaft is not securely mounted in the motor coupling.

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 correctement raccordé au moteur.

Inspectez l'arbre pour vous assurer qu'il est bien fixé.



Turn on the control unit. Adjust the rotation rate knob until the set rate is between 100 and 200 RPM, and press the Run/Pause button to begin the rotation of the shaft and installed electrode tip.

While the shaft is slowly rotating, inspect the rotating shaft and electrode tip to assure that they are rotating properly about the axis of rotation. If the shaft or electrode tip are wobbling, vibrating, or tilting away from the axis of rotation, then turn off the rotator and remove the shaft and electrode tip from the rotator.



Note:

A “Precision Shaft Alignment Kit” is available separately. This kit includes a dial indicator used to measure the “runout” at the end of the shaft (see Section 7.1 for kit part number).



WARNING:

Do not use an electrode tip that 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 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. Ce embout d'électrode est soit installée de manière incorrecte soit endommagée physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'embout d'électrode immédiatement.

4.2 Installing the Electrochemical Cell

All cells should be supported from below using the cell platform, and may be optionally clamped to the side post for extra security. For a cell with multiple side ports, carefully orient the cell so that any accessories mounted in the side ports have enough clearance. Smaller cells may be clamped using a traditional laboratory clamp secured to the center port (see Figure 4-4A). Larger cells may be clamped using a large diameter column clamp (see Figure 4-4B).

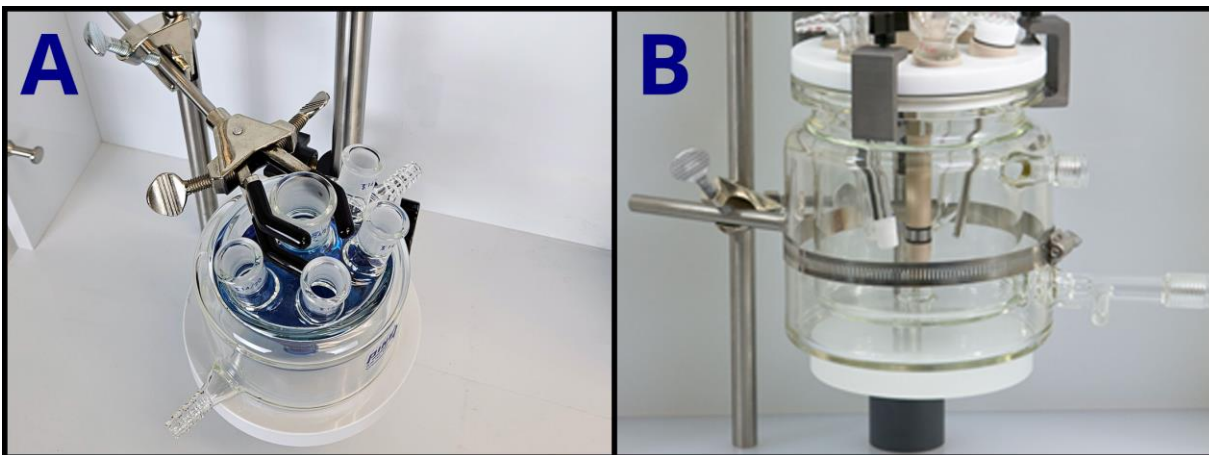


Figure 4-4. Properly Supported and Clamped Electrochemical Cells

The cell platform and clamp positions allow adjustment of the vertical position of the cell with respect to the motor unit. In addition, the vertical position of the motor unit is easily adjusted. Usually, it is easier to mount and clamp the cell in a fixed vertical position. Then, the rotating electrode can be moved vertically down into the cell or up out of the cell as needed.

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:

Lorsque vous montez ou descendez le bloc moteur le long de la barre principale, veillez à bien le tenir pour éviter qu'il ne chute brutalement et ne casse la cellule de verre située sous le bloc moteur.

When lowering an RDE or RRDE electrode tip down into the electrochemical cell, the electrode tip should be positioned so that the electrode surface is approximately 5 to 10 mm below the solution surface (see Figure 4-5). Most RDE and RRDE tips are at least 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.

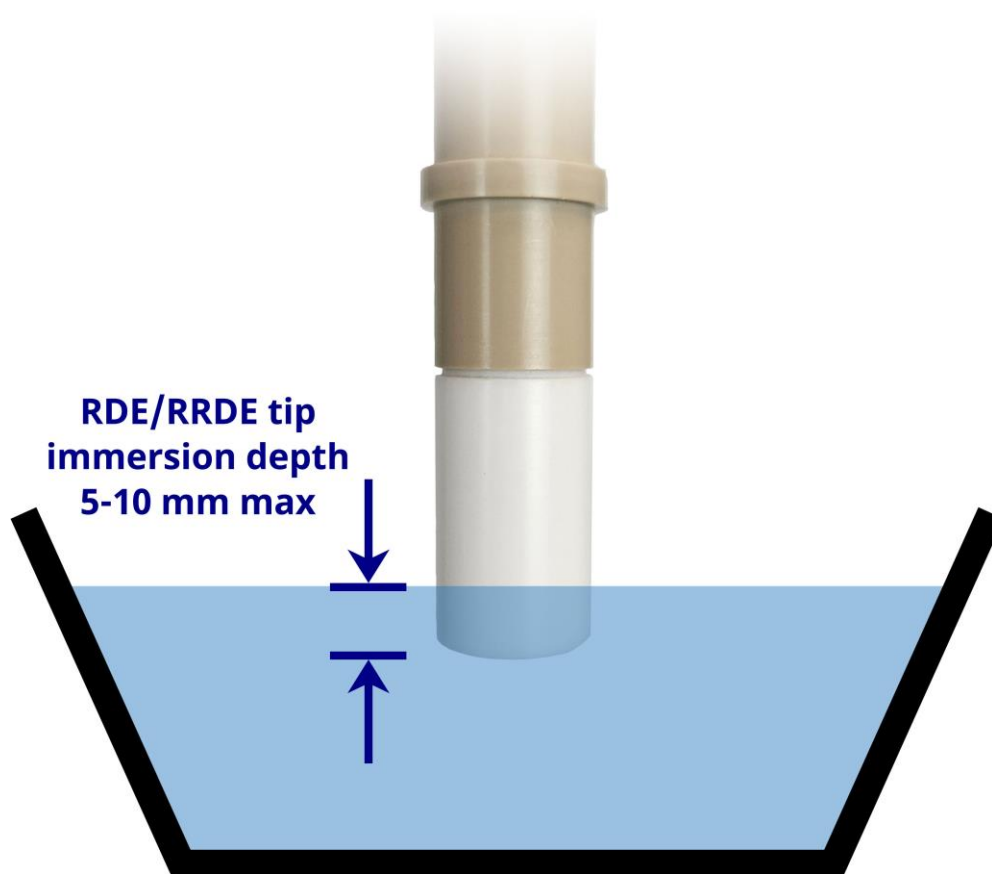


Figure 4-5. Proper Immersion Depth for RDE and RRDE Electrode Tips

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:**

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 rotative dans l'ouverture de la cellule pour qu'elle ne frotte pas les bords de l'ouverture.

Le frottement des bords de la cellule par l'arbre ou par l'embout d'électrode entraînera des dommages.

4.3 The Enclosure

After the cell has been mounted and the electrode has been lowered into the cell, securely mount the enclosure window by hooking it to the four pins on the enclosure frame (see Figure 4-6).

Note that the enclosure window has small openings near the bottom that permit cell connections, purge gas tubing, and/or circulator fluid and tubing to be carefully routed to the electrochemical cell from locations outside the enclosure.

**WARNING:****Rotating shaft.**

Do not turn on the rotator or rotate the electrode shaft unless the enclosure window is secured to all four pins.

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

AVERTISSEMENT:**Arbre en rotation.**

Ne mettez pas le rotateur en marche et ne marche ni l'arbre de l'électrode en rotation si la fenêtre du boîtier n'est pas fermée à l'aide des quatre broches.

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

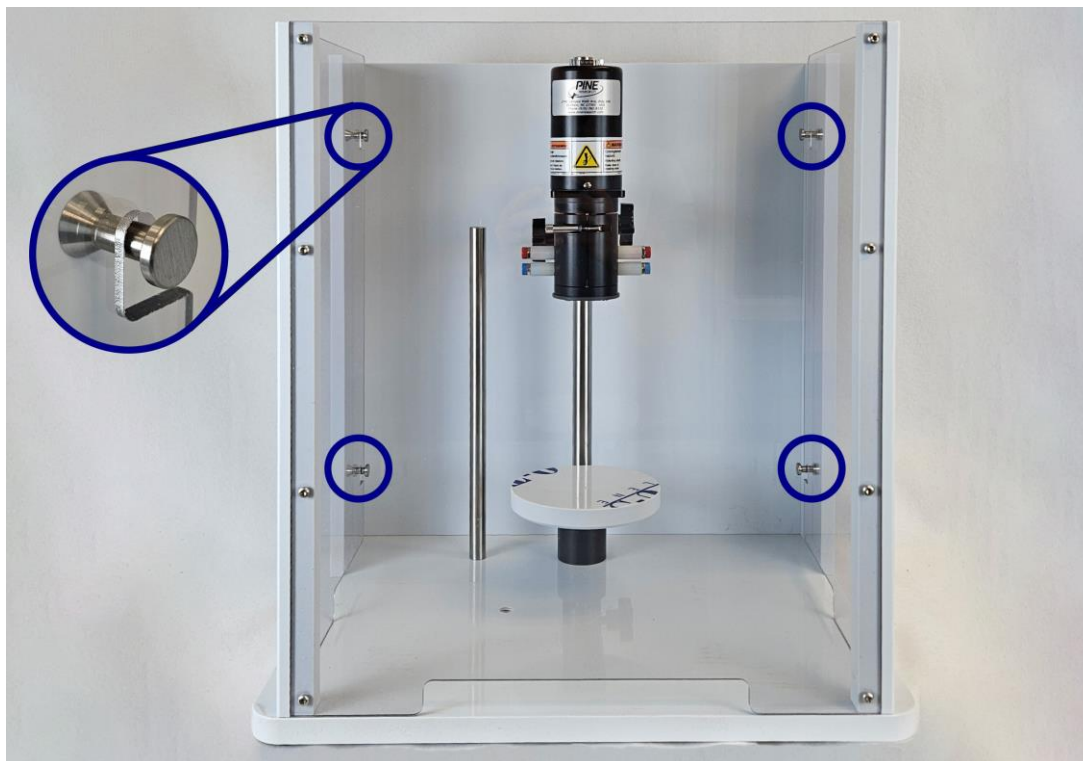


Figure 4-6. Properly Mounted Enclosure Window

Figure 4-6. Boîtier Correctement Monté

4.4 Cell Connections

The counter electrode and the reference electrode are usually mounted in appropriate side ports on the electrochemical cell (see Figure 4-7). The counter electrode is often a simple platinum wire or graphite rod to which an alligator clip is easily affixed. The reference electrode can vary depending on the type of electrolyte being used (*e.g.*, aqueous vs. non-aqueous, acidic vs. alkaline), and is often Ag/AgCl, Hg/Hg₂Cl₂ (calomel), Hg/HgO, or an Ag wire pseudo-reference.



Figure 4-7. 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. For most Pine Research potentiostats, the reference electrode cable is color-coded as white, and the counter electrode cable is color-coded as green. Many commercially-available reference electrodes have a sturdy pin connector on the top end that can accept an alligator clip. The cable that connects the reference electrode to the potentiostat should be of the shielded (coaxial) type, 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 most Pine Research potentiostat models use **GREEN** to mark the counter electrode connection and **WHITE** to mark the reference electrode connection.



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.

4.4.1 RDE and RCE Connections

There are two pairs of brushes that provide electrical contact with the rotating shaft (see Figure 4-8). The upper pair of brush contacts (red) is used to make electrical contact with a rotating disk electrode (RDE) or a rotating cylinder electrode (RCE).

There are two options for connecting the potentiostat working electrode leads for an RDE or RCE experiment. The working drive and working sense leads (shown in Figure 4-8 for a typical Pine Research potentiostat as red and orange, respectively) can be directly connected to either red brush on the MSR evo motor unit. Alternatively, a banana jumper cable can be added connecting both red brushes and the potentiostat leads connected to the banana jumper cable. This can sometimes improve the overall electrical connection, but in other cases it may introduce noise to the experiment. Ultimately, trial-and-error is required to determine the optimal configuration.

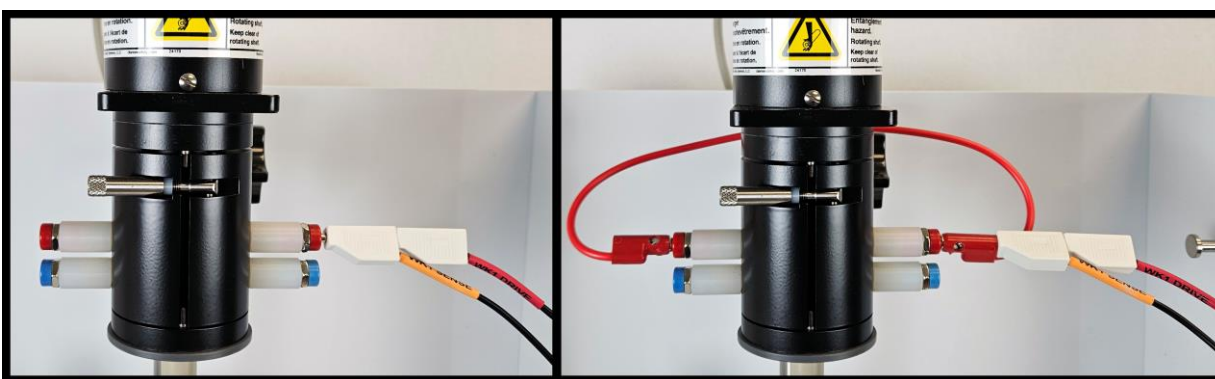


Figure 4-8. Brush Connections for a Rotating Disk (RDE) or Rotating Cylinder Electrode (RCE)



Tip:

Most modern potentiostats provide separate cable connections for the working electrode “drive” and “sense” lines. The drive line carries current while the sense line measures the potential. Both of these lines must be connected to the rotator brushes. (Note that many older potentiostats use only one cable to carry both the drive and sense signals for the working electrode.)



Note:

Cell cables on most Pine Research potentiostat models use RED to mark the working electrode drive line and ORANGE to mark the working electrode sense line. Both of these should be connected to the rotator brushes.

**Note:**

Older model potentiostats tend to use only one cable connection for both the working electrode drive and sense lines. In these cases, there is only one potentiostat cable required to be connected to the rotator brushes.

**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.

The banana jumper cables used to short the opposing brushes feature stackable banana plugs. If the potentiostat cell cables also terminate with 4 mm banana plugs, then these plugs can simply be inserted directly into either the brush contacts or the banana jumper cable. If the cell cables from the potentiostat terminate with alligator clips, then the easiest way to connect such alligator clips is to first insert a banana stud connector into the jumper cable (see Figure 4-9). The small tab on the banana stud provides a good place to attach the alligator clip.

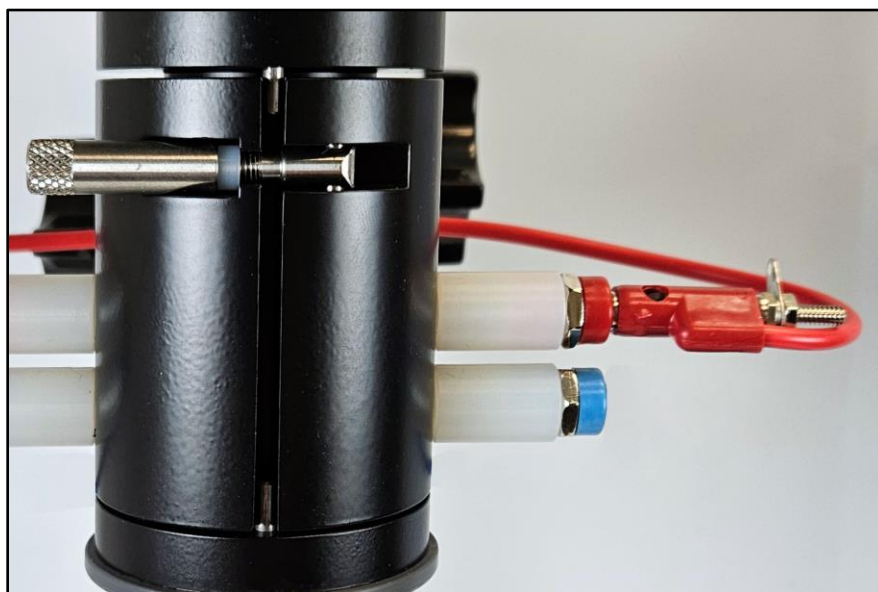


Figure 4-9. Stackable Banana Stud Connector

4.4.2 RRDE Connections

The lower pair of brush contacts are only used with a rotating ring-disk electrode (RRDE) (see Figure 4-10). The lower pair of (blue) brushes make electrical connection to the ring electrode while the upper pair of (red) brushes make electrical connection to the disk electrode. Banana jumper cables can also be optionally used to short together the opposing brushes in each pair, similarly to the procedure described in Section 4.4.1. As with RDE and RCE experiments, trial-and-error is necessary to determine the optimal electrical configuration.

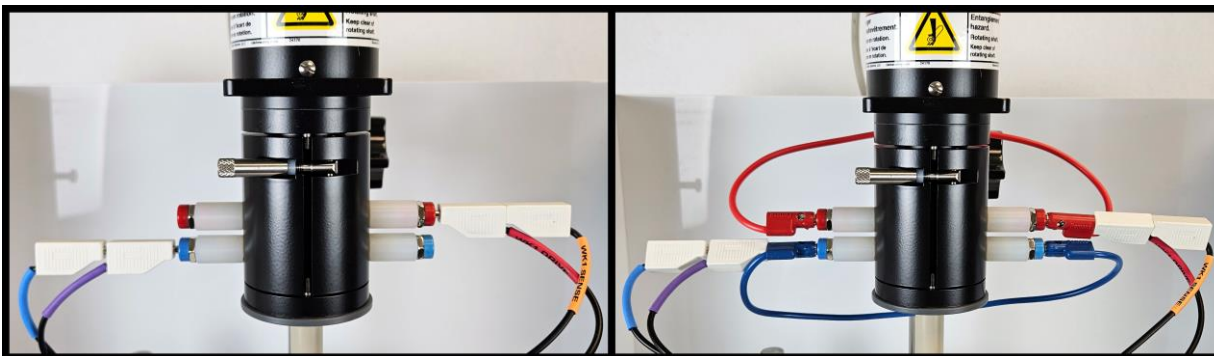


Figure 4-10. Brush Connections for a Rotating Ring-Disk Electrode (RRDE)

Tip:



It is possible that the brush assemblies on a rotator that has been in use for some time may have been replaced or swapped, and thus, the colors of the brushes may not be as described in this section.

The important concept to remember is that the **UPPER** pair of brushes contacts the disk electrode, and the **LOWER** pair of brushes contacts the ring electrode.

Tip:



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

Note:



Cell cables on most Pine Research bipotentiostat models use **RED** to mark the first (disk) working electrode drive line and **ORANGE** to mark the first (disk) working electrode sense line. Both of these should be connected to the **UPPER** (red) rotator brushes.

Cell cables on most Pine Research bipotentiostat models use **BLUE** to mark the second (ring) working electrode drive line and **VIOLET** to mark the second (ring) working electrode sense line. Both of these should be connected to the **LOWER** (blue) rotator brushes.

Note:



Older model bipotentiostats tend to use only one cable connection each for both disk and ring working electrode drive and sense lines. In these cases, there is only one bipotentiostat cable required to be connected to each pair of rotator brushes.

4.4.3 Routing Cables and Tubing

The motor control cable may be routed out of the top of the enclosure to connect the motor unit to the control unit (see Figure 4-11A). The enclosure window has slots along the bottom that provide clearance for routing cell cables and any tubing out of the enclosure (see Figure 4-11B). If required, cables and tubing may be routed through the back panel by drilling small holes in the panel. Any such drilled holes should have a diameter no greater than 13 mm (0.5 in).

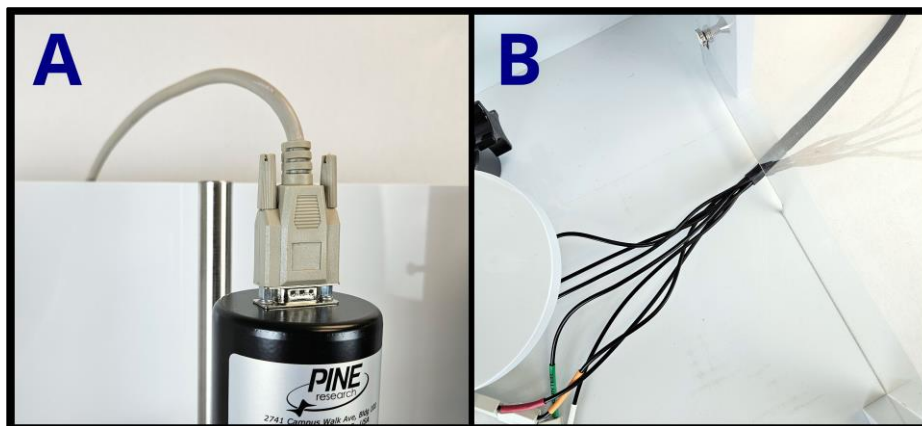


Figure 4-11. Routing Cables out of the Enclosure

4.5 Using the Rotator in a Glove Box

The rotator may be placed in a glove box when working with air- or moisture-sensitive compounds. A smaller base (sold separately) is available for purchase that can be useful for fitting inside a space-limiting glove box.

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 that contact the rotating shaft. To address the issue of brush contacts in a glove box, 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 the internal motor brushes.

See Sections 6.4 and 6.5 for more information about how to replace a worn motor.

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.

Consultez la sections 6.4 et 6.5 pour en savoir plus sur la manière de remplacer un moteur usé.

4.6 Rotation Rate Control

The primary mechanism for turning the motor on and off is the Run/Pause button on the front panel of the control unit (see Figure 4-12). This button is located above an LED Status light that indicates whether the rotator is currently in the “run” state (light is green, Figure 4-12A) or the “pause” state (light is red, Figure 4-12B).



Figure 4-12. MSR evo Control Unit Front Panel Display

A ten-turn potentiometer knob (also on the front panel of the control unit, Figure 4-12) is used to adjust the rotation rate setpoint in units of RPM. A 4 ½-digit blue LED display shows the RPM setpoint when the rotator is in the “pause” state (LED Status light is red), even though the motor is not rotating. When the rotator is toggled to the “run” state (LED Status light is green), the display shows the actual rotation rate measured by the tachometer inside the motor unit.

4.6.1 Initial Power Sequence

When the MSR evo control unit is first powered on, the rotator always reverts to the “pause” state. This means that the motor will never automatically begin rotating the moment it is turned on regardless of the rate control knob position or displayed RPM.



Note:

The MSR evo always begins in the “pause” state when it is first turned on, regardless of the rate control knob position. The motor will not automatically start rotating. The initial rate shown on the 4 ½-digit blue LED display represents the setpoint.

After the MSR evo has been powered on and 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. Toggle the Run/Pause button to begin rotation. Then, after verifying that the electrochemical cell, signal cables, motor connection cable, tubing, and other accessories are securely positioned, measurements can be made at higher rotation rates as needed.

4.6.2 Rotating the Electrode

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

If the shaft and electrode tip are properly rotating about the axis of rotation, the rotation rate may be slowly increased to the desired value using the rate control knob on the control unit. Note that while the rotator is in the “run” state, the display shows the actual rotation rate in RPM as measured by the built-in tachometer inside the motor unit.

4.6.3 External Control of the Rotation Rate

It is often convenient for the rotation rate to be controlled via an externally supplied signal. Many potentiostats are capable of providing such a signal to control the rotation rate while simultaneously performing electrochemical measurements. An externally-supplied signal is also required when performing hydrodynamically modulated voltammetry, where the rotation rate is varied sinusoidally as electrochemical measurements are made with the potentiostat (see Section 2.2.1 for more details).

Special cables are available to connect Pine Research potentiostats to the MSR evo (see Figure 4-13). These cables carry both an analog rate control signal as well as a digital control signal from the potentiostat to the external I/O port located on the back panel of the control unit. Contact Pine Research to obtain the proper cable for use with a particular potentiostat model.



Figure 4-13. Connecting Rotation Rate Control Cable to MSR evo Control Unit

The analog rate control signal from the potentiostat is a voltage that is proportional to the desired rotation rate. The MSR evo rotator is factory-configured to use a 1 RPM/mV ratio, which is the default ratio compatible with Pine Research potentiostats. Other ratios are available for use with other potentiostats (see Section 6.7).

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, causing 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).

When controlling the MSR evo rotation rate with an external source, the control switch on the front panel of the control unit must be toggled to the right position, which will cause the external control indicator LED on the front panel to become illuminated and yellow (see Figure 4-14). When the

control unit is in this state, the RPM is only controlled by an external source regardless of the rate control knob position. In other words, the rate control knob has no impact on the RPM when the external control indicator LED is illuminated. However, note that even while the control unit is in external control mode, the Run/Pause button is still functional and can be used to toggle the rotation between “run” and “pause” states.



Figure 4-14. External Control Switch and LED Indicator Set for External Control

On the other hand, when the MSR evo control unit is not being controlled by an external source (*i.e.*, when the external control indicator LED is not illuminated, and the control switch is in the left position), external rate control signals applied to the external I/O port have no effect on the rotation rate. Additionally, toggling the control switch either from left-to-right or right-to-left will place the rotator back into the “pause” state and stop rotation.

Note:



The front panel of early MSR rotators (prior to the introduction of the MSR evo in 2025) had two ways to control the rotation rate: a rate control knob (for manual control) and input jacks (for external control). These early MSR rotator models would use the summation of the knob setting and the external signal as the rotation rate setpoint.

The MSR evo uses a different approach. The MSR evo is either in manual mode or in external control mode. When in manual mode, the rate control knob adjusts the setpoint. When under external control, the externally-applied signal controls the rotation rate. Neither mode uses a summation of knob setting and external signal as the rotation rate setpoint as with early MSR rotator models.

**Note:**

Toggleing the control switch on the front panel of the control unit from left-to-right, or from right-to-left, stops rotation and places the MSR evo back into the “pause” state.

When the control switch is in the left position, the external control indicator LED is off. In this state, the RPM is set only by the rate control knob. Any added external signal will be disregarded and will not impact the RPM.

When the control switch is in the right position, the external control indicator LED is illuminated yellow. In this state, the RPM is only set by the external signal. Any position of the rate control knob will be disregarded and will not impact the RPM.

4.6.4 External Motor Stop Control

When operating in external control mode (external control switch on the front panel of the control unit in the right position), an external digital signal can be applied across the Motor Stop and DC Common pins on the external I/O port on the MSR evo control unit (see Figure 2-7 for pinout of external I/O port). This digital signal can be used by a potentiostat or other external instrument to assure that the rotation rate is exactly zero. The logic for this digital signal may be either “active HIGH” or “active LOW.”

For the MSR evo, the motor stop is initially configured at the factory to use “active HIGH” logic. If desired, a jumper setting inside the control unit can be configured to use the opposite logic (see Section 6.8 for details).

If the motor stop logic is configured to be “active HIGH,” then the motor is allowed to rotate if a signal greater than 2.0 V is applied across the Motor Stop and DC Common pins. If the two pins are shorted together (*i.e.*, if the motor stop stop signal is driven to ground), then the motor stops rotating.

If the motor stop logic is configured to be “active LOW,” then the motor will stop if a signal greater than 2.0 V is applied across the Motor Stop and DC Common pins. If the two pins are shorted together (*i.e.*, if the motor stop signal is driven to ground), 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 Motor Stop and DC Common pins, the motor is allowed to rotate. An internal “pull up” circuit assures that the motor stop signal remains “high” in this case.

4.7 Run/Pause Button and Control Unit Indicators

The Run/Pause button on the front panel of the control unit functions in a relatively predictable manner. That is, if the rotator is running (status indicator LED is green), whether in manual or external control mode (see Section 4.6.3), pressing the Run/Pause button will stop the rotation immediately (status indicator LED will turn red). Conversely, if the rotator is already stopped (or in the “pause” state, status indicator LED is red), pressing the Run/Pause button starts rotation based on the setpoint of the rotation rate knob (if in manual mode) or the external input (if in external control mode), also resulting in the status indicator LED to turn green again.

When in manual control mode, and while in the “pause” state (status indicator LED is red, control switch toggled to the left position, external control indicator LED is off), the rotation rate display shows the rotation setpoint but the rotator is not rotating.

When in external control mode, and while in the “pause” state (status indicator LED is red, control switch toggled to the right position, external control indicator LED is yellow), the rotation rate display will show the external RPM setpoint. Adjusting the rotation rate knob will have no effect whatsoever, because this knob setting is ignored by the MSR evo while in external control mode.

When the rotator is running, whether in manual or external control mode (status indicator LED is green, control switch in either position), the rotation rate display shows the actual rotation rate based on the built-in tachometer reading.

See Table 4-1 for set of modes, states, and indicators possible for the MSR evo control unit.

Mode, State	Rotation Rate Display	Status Indicator LED	Control Switch Position	External Control Indicator LED
Manual, pause	RPM setpoint	red	left	off
Manual, run	Tachometer reading	green	left	off
External, pause	External RPM setpoint	red	right	yellow
External, run	Tachometer reading	green	right	yellow

Table 4-1. Set of Possible Control Unit Conditions, Modes, and Indicators

4.8 Fault Conditions

The user should be aware of several fault conditions that may prevent the motor from rotating the electrode properly. In some cases, the user may be able to rectify the fault and continue using the rotator, but in other cases, it may be necessary to return the rotator to the factory for repair. Further details can be found in Section 8 and Section 1.6.

4.8.1 Motor Cable Fault

The fault indicator LED on the front panel illuminates (red color) if the motor control cable is improperly or partially connected after approximately four seconds (> 4 s). The user should ensure

that both ends of the cable are correctly installed, and then press the Run/Pause button to clear the fault and return to the “pause” state. Pressing the Run/Pause button a second time should start rotation.

4.8.2 Motor Overcurrent Fault

The fault indicator LED on the front panel illuminates (red color) if the circuit reset button (on the back panel) trips due to an overcurrent condition. An overcurrent fault can occur when attempting to rotate an electrode in a highly viscous solution or when the rotation rate is being modulated at a high frequency (see Section 2.2.1). The user should choose a less viscous solution and/or a lower modulation frequency, and then press the Run/Pause button to clear the fault and return to the “pause” state. Pressing the Run/Pause button a second time should start rotation.

4.8.3 Mechanical Obstruction

If the shaft is mechanically locked or stuck (unable to freely rotate), the control unit will stop trying to rotate the shaft. The rotation rate display will go blank and the control unit will reset. After this reset occurs, the fault indicator LED is **not** illuminated. The user must turn off the rotator, disconnect the motor cable, and then check the shaft to ensure that it is able to freely rotate.

4.9 Circuit Protection

The power switch on the front panel of the control unit also acts as a circuit breaker to help protect the control unit circuitry. If the circuit breaker trips, then it can be reset by turning the power switch to the full “off” position and then turning the switch back “on” again.

A secondary circuit breaker on the back panel protects the windings in the motor. If this circuit breaker trips, then the circuit breaker can be reset by pressing the “RESET” button on the back panel.

4.10 Proper Grounding

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 items that may electromagnetically interfere with a delicate electrochemical measurement.

To avoid issues with signal noise when making electrochemical measurements, it is important to properly ground all metal objects near an electrochemical cell to the earth ground. This generally includes the metal chassis of the instrumentation (potentiostat and rotator), the clamps and supports used to physically secure the electrochemical cell, and any peripheral equipment (heaters, stirrers, etc.) used in conjunction with the measurement.

4.10.1 Grounding Terminology

There are generally three types of grounding connections that are often confused with one another: the **earth ground**, the **chassis terminal**, and the **DC Common**. These are discussed in more detail below.

4.10.2 Earth Ground



EARTH GROUND:

An earth ground connection is a direct physical and electrical connection to the Earth. It is sometimes referred to as Protective 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 that brings the earth ground connection to the instrument's power supply. Depending on the design of the instrument, the earth ground connection may or may not pass through the power supply to the circuitry inside the instrument.

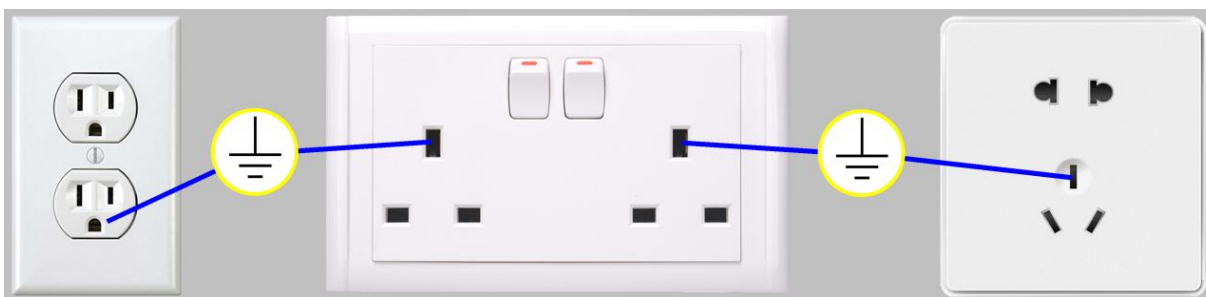


Figure 4-15. Location of Earth Ground on Common Electrical Receptacles

It is very important to note that the chassis of the rotator control unit is in direct contact with the earth ground connector. Thus, it is not possible to isolate the control unit chassis from the “third prong” earth ground on the power cord connector.

The chassis of the motor unit is also normally connected to the chassis of the control unit, and thus, to earth ground. This connection is usually made in an indirect fashion. Because the motor control cable (which connects the motor unit to the control unit) is a shielded cable, the shield assures that the chassis of the motor unit and the chassis of the control unit are electrically connected. And because the control unit chassis is connected to earth ground, the motor unit chassis is also therefore in contact with earth ground. The MSR evo uses earth ground to ensure the electrical safety of the user.



Note:

A shielded motor control cable that has HD-15 connectors on each end of the cable is used to connect motor unit to control unit. The shield line in this cable assures that the chassis of the motor unit is in contact with the chassis of the control unit. The chassis of the control unit is, in turn, in contact with earth ground via the “third prong” on the power cord; thus, the motor unit chassis is also earth grounded.

4.10.3 Chassis Terminal

**CHASSIS TERMINAL:**

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

The metal case that contains the instrument circuitry is called the instrument chassis. The chassis helps to protect the circuitry from environmental noise sources and ESD events. There is a convenient access point to the instrument chassis on the external I/O port on the back panel of the MSR evo control unit (see Figure 2-7 for pinout of external I/O port).

While scientific instruments sometimes have a chassis that “floats” with respect to the earth ground, this is not the case with the MSR evo. The chassis of the MSR evo is directly connected to earth ground via the third prong on the power entry module. There is no mechanism to separate the chassis from earth ground when operating the MSR evo.

When multiple measurement devices are used together in an experiment, it is common practice to connect the instrument chassis terminals for all of the instruments together. It is also common practice to place the electrochemical cell in a Faraday cage and connect the Faraday cage to the instrument chassis. These connections assure that the sensitive measurement circuitry in the various instruments and the electrochemical cell are all effectively sharing the same outer shield against environmental noise.

4.10.4 DC Common

**DC COMMON:**

In an analog circuit, 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 is the zero volt (0.0 V) reference point used by the motor control circuit. There is a convenient access point to the DC Common on the external I/O port on the back panel of the MSR evo control unit (see Figure 2-7 for pinout of external I/O port).

The MSR evo may send or receive analog signals to and from other electronic instruments, such as a potentiostat, waveform generator, an x-y recorder, a digital oscilloscope, a spectrometer, or a quartz crystal microbalance. These other instruments also have a DC Common line that represents the common “zero volt” analog signal level. In general, the act of connecting a signal cable from the MSR evo to another instrument connects the DC Common lines for both instruments.

The distinction between the DC Common and the instrument chassis is important to maintain and preserve whenever possible. Just like the MSR evo 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 (and therefore also earth ground) reduces the amount of interference from environmental noise.

Finally, it is important to be aware of cases where a hidden connection indirectly compromises the floating DC Common. These cases can occur when multiple instruments and/or computers are interconnected with the MSR evo as part of a larger experimental configuration. One of the other instruments may make an internal connection between DC Common and the chassis or earth ground. Finding and eliminating such hidden connections often requires some detective work using an ohmmeter.

4.10.5 Faraday Cages

When making sensitive electrochemical measurements (*e.g.*, electroanalytical methods employing DC currents less than one microampere ($< 1 \mu\text{A}$) or small amplitude AC methods such as electrochemical impedance spectroscopy), it is very important to place the entire electrochemical cell inside a metal Faraday cage to shield the experiment from environmental noise. In addition, the portion of the potentiostat cell cable (near the electrochemical cell) where the individual signal lines emerge from the protective shielding or mesh (if the cell cable has such a shielding) should also be placed inside the Faraday cage.

After placing the ends of the cell cable and the electrochemical cell inside of the Faraday cage, a secure electrical connection should be made between the metal Faraday cage and the MSR evo chassis terminal. This combination of the instrument chassis, the mesh around the cell cable, and the Faraday cage essentially puts the entire system (circuitry and cell) inside of an overall outer protective shield (*i.e.*, the cell cable mesh and the Faraday cage act as an extension of the instrument chassis).

A Faraday cage can either be purchased directly from a supplier or fabricated using inexpensive and commonly-found materials. Anything from a commercial electrical enclosure to a cardboard box lined with aluminum foil can serve as a functional Faraday cage (see Figure 4-16). A Faraday cage requires an internal volume large enough to contain the entire electrochemical cell and all of the banana plugs at the end of the cell cable that connect to the various electrodes. Care should be taken to ensure that the electrode connections do not accidentally come into contact with the conductive walls of the Faraday cage.

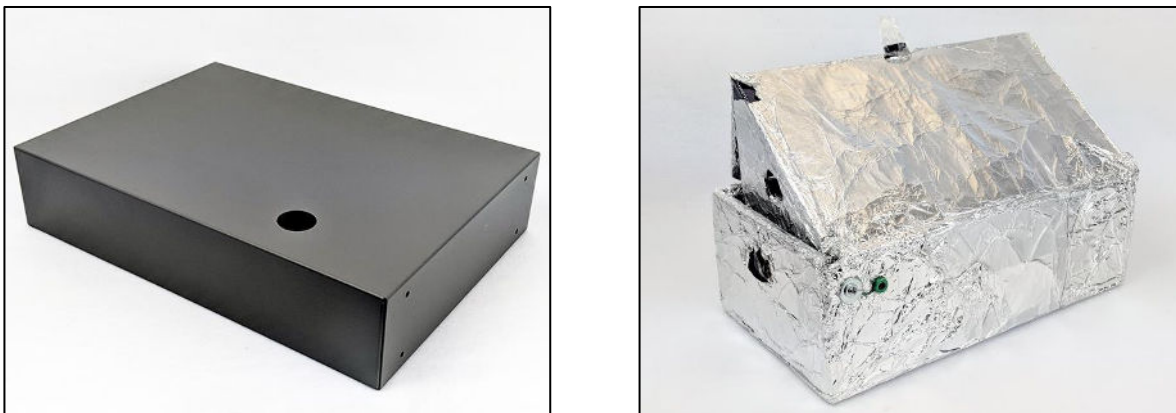


Figure 4-16. Common Examples of Faraday Cages

4.10.6 Metal Apparatus

Electrochemical cells are often mounted using various metal apparatus (such as ring stands or laboratory clamps). These mounts, along with any other metal objects located near the electrochemical setup, can interfere with sensitive electrochemical measurements, especially if they are simply allowed to “float” rather than being electrically connected to a known point in the system. Some trial-and-error may be required to determine the best way to ground such metal objects, but in many cases, an alligator clip and a banana cable (see Figure 4-17) can be used to connect the metal object to the instrument chassis, the earth ground, or to both.



Figure 4-17. Metal Objects Near the Electrochemical Cell Can be Grounded

4.10.7 Typical Grounding Strategies

While the details of proper grounding for any given electrochemical experiment may differ, a common approach when working with a rotating electrode cell is to connect the chassis of all 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 cages, 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 that injects environmental noise into sensitive signal measurements.

To prevent accidental creation of a grounding loop, bring all grounding connections together to a common point.

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 that 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 that 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 that 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.

If the potentiostat does not offer a chassis terminal connection (or if earth grounding the chassis terminal is not an option), then a less desirable, but alternative approach is to attempt to make use of the potentiostat’s signal ground (DC Common). If the DC Common (often provided as an alligator clip connection on the cell cable) is connected to the earth ground on the rotator control unit, this may (or may not) reduce or eliminate signal noise.

5 Electrodes

5.1 Electrode Handling Precautions

Rotating electrode tips and shafts are precision research tools machined to tight specifications for proper balance when rotating at high rotation rates. When not in use, an electrode tip should be cleaned, dried, and stored in its original case. When working with electrode shafts and tips, special care should be taken not to drop the shaft or tip as this will likely throw the shaft or tip off balance.



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 autre 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 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 tombée ou a été endommagée physiquement d'une autre manière ou d'une autre.

Inspectez l'embout d'électrode pour vous assurer qu'elle n'a pas été endommagée.



WARNING:

Do not use an electrode shaft that 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. Cet arbre est soit installé de manière incorrecte soit endommagé physiquement. Éteignez le rotateur, déconnectez l'alimentation électrique et retirez l'arbre immédiatement.

**WARNING:**

Do not use an electrode tip that 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. Cet embout d'électrode est soit installée de manière incorrecte soit endommagée 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.

Each type of rotating electrode 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 maximum pour une électrode.

Chaque type d'électrode rotative possède une vitesse de rotation maximum spécifique.

Consultez la documentation pour l'électrode spécifique à utiliser pour connaître la vitesse de rotation maximum de l'électrode.

**CAUTION:**

Do not apply excessive twisting force to the shroud of an electrode tip when threading it on to 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.

**CAUTION:**

Position the motor unit with respect to the glass cell so that the electrode tip is immersed approximately 1.0 cm into the test solution.

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

ATTENTION:

Positionnez le bloc moteur en fonction de la position de la cellule de verre, de telle sorte que l'embout d'électrode soit immergée sur environ 1 cm dans la solution d'essai.

Une immersion excessive peut entraîner la corrosion de l'arbre ou d'embout d'électrode en provoquant l'infiltration de liquides dans le joint situé entre l'arbre et l'embout d'électrode.

**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 rotative dans l'ouverture de la cellule pour qu'elle ne frotte pas les bords de l'ouverture.

Le frottement des bords de la cellule par l'arbre ou par l'embout d'électrode entraînera des dommages.

**TEMPERATURE LIMITATIONS:**

Electrode tips with PTFE shrouds are designed for use at room temperature (10°C to 25°C). Exposing these tips to colder or warmer temperatures is likely to compromise the seal between the PTFE shroud and the electrode surface.

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

**Note:**

After each use of rotating electrode (or electrode tip), clean and dry the electrode and then return it to the plastic storage box in which it was originally shipped.

**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 the factory for professional repolishing.

5.2 Shafts

The MSR evo rotator accepts a variety of different shaft designs (each sold separately) having a sturdy metal internal shank that is insulated with a polymeric shroud. The upper portion of the shaft is designed to mate with the motor coupling inside the brush chamber (see Figure 4-2). The lower portion of the shaft is protected with a chemically-resistant shroud material (PTFE, PEEK, or PCTFE).



Standard RDE & RCE Shaft (12 mm OD)

The lower end of this shaft features a 12 mm OD PTFE shroud and a standard 1/4-28 thread. These threads accept RDE and RCE tips with 12 mm OD shrouds.

Specifically, this shaft is compatible with E3 and E4TQ series RDE tips and with classic 12 mm OD rotating cylinder electrode tips.

A bearing assembly for mounting this shaft in a 24/25 ground glass joint is available separately.

Description

Part Number

Standard RDE & RCE Shaft (for 12 mm OD tips)	AFE3M
Bearing Assembly (24/25 taper; for Standard RDE & RCE Shaft)	AC01TPA



Precision RDE & RRDE Shaft (15 mm OD) (for use with gas-purged bearing assembly)

This shaft has a precision-machined 15 mm outer diameter that is specially-designed to mate with the 15 mm inner diameter of a gas-purged bearing assembly.

This shaft is compatible with E5, E6, E7, and E8 series tips.

Description

Part Number

Precision RDE & RRDE Shaft (15 mm OD)	AFE6MB
Gas-Purged Bearing Assembly (24/25 taper; for 15 mm OD RDE & RRDE shaft)	AC01TPA6M



Precision RCE Shaft (15 mm OD) (for use with gas-purged bearing assembly)

This shaft has a precision-machined 15 mm outer diameter that is specially-designed to mate with the 15 mm inner diameter of a gas-purged bearing assembly.

This shaft has a PEEK shroud and accepts cylinder inserts that are 15.0 mm OD x 6.3 mm tall. The cylinder inserts are sealed between a pair of washers.

Description

Part Number

Precision RCE Shaft (15 mm OD)	AFE9MBA
Gas-Purged Bearing Assembly (24/25 taper; for 15 mm OD RCE shaft)	AC01TPA6M



Precision Gas-Purged Bearing Assembly (15 mm ID)

This gas-purged bearing assembly fits into the 24/25 center port on an electrochemical cell. A small plastic hose barb on the side of the assembly allows the space within the bearing assembly to be purged with an inert gas.

The main body of the assembly is made from chemically-resistant PEEK polymer, and the bearing is ceramic.

Although the bearing is not perfectly sealed, the inner diameter of the bearing (15 mm ID) allows a precision-machined shaft (15 mm OD) to pass through the bearing assembly with a reasonably tight fit.



Description

Part Number

Gas-Purged Bearing Assembly (24/25 taper; 15 mm ID bearing)	AC01TPA6M
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Simple Taper Plug Assembly (6.35 mm ID)

This bearing assembly fits into the 24/25 center port on an electrochemical cell. The main body of the assembly is made from PTFE, and the internal diameter of the opening in the stainless steel bearing is 6.35 mm. This 6.35 mm ID opening is compatible with the standard RDE and RCE shaft (part number AFE3M) and with E2 series single-piece RDEs. This bearing assembly does not perfectly seal the electrochemical cell.

Description**Part Number**

Simple Taper Plug Assembly (24/25 taper; 6.35 mm ID bearing) AC01TPA

5.3 RDE Tips

The MSR evo rotator is compatible with a variety of RDE tips (sold separately), and each tip design is compatible with one or more shafts as described below.



E3PK Series RDE Tips (with PEEK shroud)

These RDE tips feature a 12 mm OD PEEK shroud around a 5 mm OD disk electrode. These tips fit the standard RDE shaft and may be used at rotation rates up to 3000 RPM. Standard disk materials include gold, platinum, and glassy carbon.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 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 prolonged exposure to concentrated acids.

Part numbers for these E3 series RDE tips and compatible shafts are listed below. If the part number for an item is not listed, contact Pine Research for more details.

Shafts

Standard RDE Shaft..... AFE3M

RDE Tips (carbon materials)

glassy carbon..... AFE3T050GCPK

pyrolytic graphite

edge plane AFE3T050GEPK

basal plane AFE3T050GBPK

RDE Tips (metal alloys)

1018 carbon steel AFE3T050S1PK

303 stainless steel AFE3T050T1PK

316 stainless steel AFE3T050T3PK

316L stainless steel AFE3T050T6PK

321 stainless steel AFE3T050T9PK

410 stainless steel AFE3T050T4PK

430 stainless steel AFE3T050T5PK

2205 stainless steel AFE3T050T7PK

Zeron 100 stainless steel..... AFE3T050T8PK

RDE Tips (precious metals)

gold (Au) AFE3T050AUPK

platinum (Pt) AFE3T050PTPK

palladium (Pd)..... AFE3T050PDPK

silver (Ag) AFE3T050AGPK

RDE Tips (common metals)

aluminum (Al) AFE3T050ALPK

cobalt (Co) AFE3T050COPK

copper (Cu)..... AFE3T050CUPK

iron (Fe)..... AFE3T050FEPK

niobium (Nb)..... AFE3T050NBPK

nickel (Ni)..... AFE3T050NIPK

lead (Pb)..... AFE3T050PBPK

tin (Sn)..... AFE3T050SNPK

tantalum (Ta)..... AFE3T050TAPK

titanium (Ti)..... AFE3T050TIPK

tungsten (W) AFE3T050WPK

zinc (Zn)..... AFE3T050ZNPk



E3 Series RDE Tips (with PTFE shroud)

These RDE tips feature a 12 mm OD PTFE shroud around a 5 mm OD disk electrode. These tips fit the standard RDE shaft and may be used at rotation rates up to 3000 RPM. Standard disk materials include gold, platinum, and glassy carbon.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 10°C to 25°C

Do not use this electrode outside the operating temperature range.

Part numbers for these E3 series RDE tips and compatible shafts are listed below. If the part number for an item is not listed, contact Pine Research for more details.

Shafts

Standard RDE Shaft..... AFE3M

RDE Tips (carbon materials)

glassy carbon..... AFE3T050GC

pyrolytic graphite

edge plane AFE3T050GE

basal plane AFE3T050GB

RDE Tips (metal alloys)

1018 carbon steel AFE3T050S1

303 stainless steel AFE3T050T1

316 stainless steel AFE3T050T3

316L stainless steel AFE3T050T6

321 stainless steel AFE3T050T9

410 stainless steel AFE3T050T4

430 stainless steel AFE3T050T5

2205 stainless steel AFE3T050T7

Zeron 100 stainless steel AFE3T050T8

RDE Tips (precious metals)

gold (Au) AFE3T050AU

platinum (Pt) AFE3T050PT

palladium (Pd)..... AFE3T050PD

silver (Ag)..... AFE3T050AG

RDE Tips (common metals)

aluminum (Al) AFE3T050AL

cobalt (Co) AFE3T050CO

copper (Cu)..... AFE3T050CU

iron (Fe)..... AFE3T050FE

niobium (Nb) AFE3T050NB

nickel (Ni)..... AFE3T050NI

lead (Pb)..... AFE3T050PB

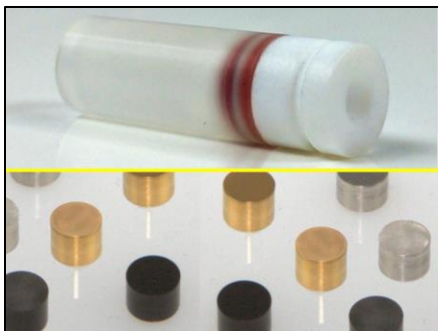
tin (Sn)..... AFE3T050SN

tantalum (Ta)..... AFE3T050TA

titanium (Ti)..... AFE3T050TI

tungsten (W) AFE3T050W

zinc (Zn)..... AFE3T050ZN



E4TQ Series ChangeDisk RDE Tips

These RDE tips feature a 12 mm OD PTFE holder that can accept a removable disk insert. These tips fit the standard RDE shaft and may be used at rotation rates up to 2000 RPM. The disk insert (5 mm OD x 4 mm thick) is typically fabricated from gold, platinum, or glassy carbon.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 10°C to 25°C

Do not use this electrode outside the operating temperature range.

Part numbers for these E4TQ series ChangeDisk RDE tips, compatible shafts, tools, and disk inserts are listed below. If the part number for a particular item is not listed, contact Pine Research for more details.

Description	Part Number
Standard RDE Shaft (for 12 mm OD RDE tips).....	AFE3M
ChangeDisk RDE tip (12 mm OD PTFE shroud, for 5 mm OD x 4 mm thick disks)	AFE4TQ050
Toolkit (for removing and polishing disk inserts).....	AFE4K050
Disk Inserts (carbon materials)	
glassy carbon.....	AFED050P040GC
pyrolytic graphite	
edge plane	AFED050P040GE
basal plane	AFED050P040GB
Disk Inserts (metal alloys)	
1018 carbon steel	AFED050P040S1
303 stainless steel	AFED050P040T1
304 stainless steel	AFED050P040T2
316 stainless steel	AFED050P040T3
316L stainless steel	AFED050P040T6
321 stainless steel	AFED050P040T9
410 stainless steel	AFED050P040T4
430 stainless steel	AFED050P040T5
2205 stainless steel	AFED050P040T7
Zeron 100 stainless steel.....	AFED050P040T8
brass	AFED050P040BR
Disk Inserts (precious metals)	
gold (Au)	AFED050P040AU
platinum (Pt)	AFED050P040PT
palladium (Pd).....	AFED050P040PD
silver (Ag).....	AFED050P040AG
Disk Inserts (common metals)	
aluminum (Al)	AFED050P040AL
cobalt (Co)	AFED050P040CO
copper (Cu).....	AFED050P040CU
iron (Fe).....	AFED050P040FE
niobium (Nb).....	AFED050P040NB
nickel (Ni).....	AFED050P040NI
lead (Pb).....	AFED050P040PB
tin (Sn).....	AFED050P040SN
tantalum (Ta).....	AFED050P040TA
titanium (Ti).....	AFED050P040TI
tungsten (W)	AFED050P040WU
zinc (Zn).....	AFED050P040ZN



E5PK Series RDE Tips (with PEEK shroud)

These RDE tips feature a 15 mm OD PEEK shroud around a 5 mm OD disk electrode. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM. Standard disk materials include gold, platinum, and glassy carbon.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 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 prolonged exposure to concentrated acids.

Part numbers for these E5 series RDE tips and compatible shafts are listed below. If the part number for a particular item is not listed, contact Pine Research for more details.

Shafts

Precision RDE & RRDE Shaft.....AFE6MB

RDE Tips (carbon materials)

glassy carbon.....AFE5T050GCPK

pyrolytic graphite

edge planeAFE5T050GEPK

basal planeAFE5T050GBPK

RDE Tips (metal alloys)

1018 carbon steelAFE5T050S1PK

303 stainless steelAFE5T050T1PK

316 stainless steelAFE5T050T3PK

316L stainless steelAFE5T050T6PK

321 stainless steelAFE5T050T9PK

410 stainless steelAFE5T050T4PK

430 stainless steelAFE5T050T5PK

2205 stainless steelAFE5T050T7PK

Zeron 100 stainless steelAFE5T050T8PK

RDE Tips (precious metals)

gold (Au) AFE5T050AUPK

platinum (Pt) AFE5T050PTPK

palladium (Pd)..... AFE5T050PDPK

silver (Ag)..... AFE5T050AGPK

RDE Tips (common metals)

aluminum (Al) AFE5T050ALPK

cobalt (Co) AFE5T050COPK

copper (Cu)..... AFE5T050CUPK

iron (Fe)..... AFE5T050FEPK

niobium (Nb) AFE5T050NBPK

nickel (Ni)..... AFE5T050NIPK

lead (Pb)..... AFE5T050PBPK

tin (Sn)..... AFE5T050SNPK

tantalum (Ta)..... AFE5T050TAPK

titanium (Ti)..... AFE3T050TIPK

tungsten (W) AFE3T050WPK

zinc (Zn)..... AFE3T050ZNPk



E5 Series RDE Tips (with PTFE shroud)

These RDE tips feature a 15 mm OD PTFE shroud around a 5 mm OD disk electrode. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM. Standard disk materials include gold, platinum, and glassy carbon.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 10°C to 25°C

Do not use this electrode outside the operating temperature range.

Part numbers for these E5 series RDE tips and compatible shafts are listed below. If a part number is not listed, contact Pine Research for more details.

Shafts

Precision RDE & RRDE Shaft.....AFE6MB

RDE Tips (carbon materials)

glassy carbon..... AFE5T050GC

pyrolytic graphite

edge plane AFE5T050GE

basal plane AFE5T050GB

RDE Tips (metal alloys)

1018 carbon steel AFE5T050S1

303 stainless steel AFE5T050T1

316 stainless steel AFE5T050T3

316L stainless steel AFE5T050T6

321 stainless steel AFE5T050T9

410 stainless steel AFE5T050T4

430 stainless steel AFE5T050T5

2205 stainless steel AFE5T050T7

Zeron 100 stainless steel AFE5T050T8

RDE Tips (precious metals)

gold (Au) AFE5T050AU

platinum (Pt) AFE5T050PT

palladium (Pd)..... AFE5T050PD

silver (Ag) AFE5T050AG

RDE Tips (common metals)

aluminum (Al) AFE5T050AL

cobalt (Co) AFE5T050CO

copper (Cu)..... AFE5T050CU

iron (Fe)..... AFE5T050FE

niobium (Nb) AFE5T050NB

nickel (Ni) AFE5T050NI

lead (Pb)..... AFE5T050PB

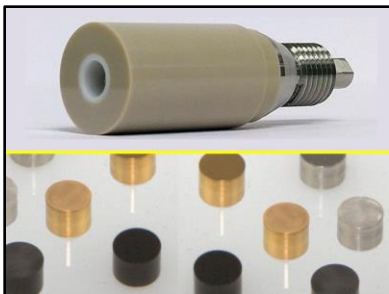
tin (Sn) AFE5T050SN

tantalum (Ta)..... AFE5T050TA

titanium (Ti) AFE3T050TI

tungsten (W) AFE3T050W

zinc (Zn)..... AFE3T050ZN



E5TQ & E5TQPK Series ChangeDisk RDE Tips

These RDE tips feature a 15 mm OD shroud that accepts a 5 mm OD removable disk insert. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM. A thin, hydrophobic PTFE seal (known as a “U-cup”) surrounds the disk insert when it is mounted in the shroud. The shroud may be PEEK or PTFE.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



CHEMICAL INCOMPATIBILITY:

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

Part numbers for these ChangeDisk RDE tips, compatible shafts, tools and disk inserts are listed below. If a part number is not listed, contact Pine Research for more details.

Description	Part Number
ChangeDisk RDE tip (15 mm OD PEEK shroud, for use from 10°C to 80°C)	AFE5TQ050PK
ChangeDisk RDE tip (15 mm OD PTFE shroud, for use from 10°C to 25°C).....	AFE5TQ050
Precision RDE & RRDE Shaft (for 15 mm OD tips).....	AFE6MB
Toolkit (for removing and polishing disk inserts).....	AFE6K050
Disk Inserts (carbon materials)	
glassy carbon.....	AFED050P040GC
pyrolytic graphite	
edge plane	AFED050P040GE
basal plane	AFED050P040GB
Disk Inserts (metal alloys)	
1018 carbon steel	AFED050P040S1
303 stainless steel	AFED050P040T1
304 stainless steel	AFED050P040T2
316 stainless steel	AFED050P040T3
316L stainless steel	AFED050P040T6
321 stainless steel	AFED050P040T9
410 stainless steel	AFED050P040T4
430 stainless steel	AFED050P040T5
2205 stainless steel	AFED050P040T7
Zeron 100 stainless steel.....	AFED050P040T8
brass	AFED050P040BR
Disk Inserts (precious metals)	
gold (Au)	AFED050P040AU
platinum (Pt)	AFED050P040PT
palladium (Pd).....	AFED050P040PD
silver (Ag)	AFED050P040AG
Disk Inserts (common metals)	
aluminum (Al)	AFED050P040AL
cobalt (Co)	AFED050P040CO
copper (Cu).....	AFED050P040CU
iron (Fe).....	AFED050P040FE
niobium (Nb).....	AFED050P040NB
nickel (Ni).....	AFED050P040NI
lead (Pb).....	AFED050P040PB
tin (Sn).....	AFED050P040SN
tantalum (Ta).....	AFED050P040TA
titanium (Ti).....	AFED050P040TI
tungsten (W)	AFED050P040WU
zinc (Zn).....	AFED050P040ZN

5.4 Single-Piece RDE Designs



E2MPK Series FastSpeed RDEs (with PEEK shroud)

These single-piece rotating disk electrodes are ideal for applications requiring a high rotation rate (up to 7000 RPM). Each electrode features a 5 mm OD disk sealed inside a 12 mm OD PEEK shroud.



CAUTION:

Maximum Rotation Rate: 7000 RPM. Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximum: 7000 TR/MIN. Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximum.



WARNING:

Rotating shaft. Use extreme caution when operating the rotator at rotation rates above 2000 RPM. Always secure the enclosure around the rotator before rotating the electrode (see Figure 4-6).

AVERTISSEMENT:

Arbre en rotation. Soyez extrêmement prudent lorsque vous utilisez le rotateur à des vitesses de rotation supérieures à 2000 tr/min. Fixez correctement le boîtier autour du rotateur du rotateur avant de mettre l'électrode en rotation (voir figure 4.6).



OPERATING TEMPERATURE RANGE: 10°C to 80°C

Do not use this electrode outside the operating temperature range.

Part numbers for single-piece RDEs are listed below. If the part number for a particular electrode material is not listed, contact Pine Research for more details.

Single-Piece RDEs (carbon materials)

glassy carbon.....	AFE2M050GCPK
pyrolytic graphite	
edge plane	AFE2M050GEPK
basal plane	AFE2M050GBPCK

Single-Piece RDEs (metal alloys)

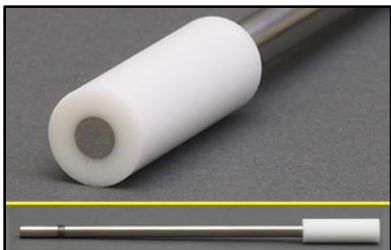
1018 carbon steel	AFE2M050S1PK
303 stainless steel	AFE2M050T1PK
316 stainless steel	AFE2M050T3PK
316L stainless steel	AFE2M050T6PK
321 stainless steel	AFE2M050T9PK
410 stainless steel	AFE2M050T4PK
430 stainless steel	AFE2M050T5PK
2205 stainless steel	AFE2M050T7PK
Zeron 100 stainless steel	AFE2M050T8PK

Single-Piece RDEs (precious metals)

gold (Au)	AFE2M050AUPK
platinum (Pt)	AFE2M050PTPK
palladium (Pd).....	AFE2M050PDPK
silver (Ag).....	AFE2M050AGPK

Single-Piece RDEs (common metals)

aluminum (Al)	AFE2M050ALPK
cobalt (Co)	AFE2M050COPK
copper (Cu).....	AFE2M050CUPK
iron (Fe).....	AFE2M050FEPK
niobium (Nb).....	AFE2M050NBPK
nickel (Ni).....	AFE2M050NIPK
lead (Pb).....	AFE2M050PBPK
tin (Sn).....	AFE2M050SNPK
tantalum (Ta).....	AFE2M050TAPK
titanium (Ti).....	AFE2M050TIPK
tungsten (W)	AFE2M050WPK
zinc (Zn).....	AFE2M050ZNPCK



E2M Series FastSpeed RDEs (with PTFE shroud)

These single-piece rotating disk electrodes are ideal for applications requiring a high rotation rate (up to 7000 RPM). Each electrode features a 5 mm OD disk sealed inside a 12 mm OD PTFE shroud.



CAUTION:

Maximum Rotation Rate: 7000 RPM. Do not rotate at rates higher than the maximum rotation rate.

ATTENTION:

Vitesse de rotation maximum: 7000 TR/MIN. Ne mettez pas l'appareil en rotation à des vitesses supérieures à la vitesse de rotation maximum.



WARNING:

Rotating shaft. Use extreme caution when operating the rotator at rotation rates above 2000 RPM. Always secure the enclosure around the rotator before rotating the electrode (see Figure 4-6).

AVERTISSEMENT:

Arbre en rotation. Soyez extrêmement prudent lorsque vous utilisez le rotateur à des vitesses de rotation supérieures à 2000 tr/min. Fixez correctement le boîtier autour du rotateur du rotateur avant de mettre l'électrode en rotation (voir figure 4.6).



OPERATING TEMPERATURE RANGE: 10°C to 25°C

Do not use this electrode outside the operating temperature range.

Part numbers for single-piece RDEs are listed below. If the part number for a particular electrode material is not listed, contact Pine Research for more details.

Single-Piece RDEs (carbon materials)

glassy carbon.....	AFE2M050GC
pyrolytic graphite	
edge plane	AFE2M050GE
basal plane	AFE2M050GB

Single-Piece RDEs (precious metals)

gold (Au)	AFE2M050AU
platinum (Pt)	AFE2M050PT
palladium (Pd).....	AFE2M050PD
silver (Ag).....	AFE2M050AG

Single-Piece RDEs (metal alloys)

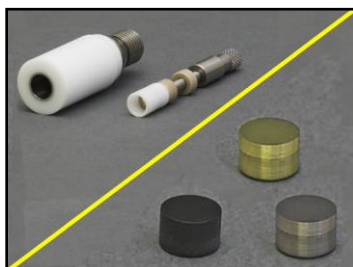
1018 carbon steel	AFE2M050S1
303 stainless steel	AFE2M050T1
316 stainless steel	AFE2M050T3
316L stainless steel	AFE2M050T6
321 stainless steel	AFE2M050T9
410 stainless steel	AFE2M050T4
430 stainless steel	AFE2M050T5
2205 stainless steel	AFE2M050T7
Zeron 100 stainless steel.....	AFE2M050T8

Single-Piece RDEs (common metals)

aluminum (Al)	AFE2M050AL
cobalt (Co)	AFE2M050CO
copper (Cu).....	AFE2M050CU
iron (Fe).....	AFE2M050FE
niobium (Nb).....	AFE2M050NB
nickel (Ni).....	AFE2M050NI
lead (Pb).....	AFE2M050PB
tin (Sn).....	AFE2M050SN
tantalum (Ta).....	AFE2M050TA
titanium (Ti).....	AFE2M050TI
tungsten (W)	AFE2M050W
zinc (Zn).....	AFE2M050ZN

5.5 RRDE Tips

All RRDE tips have 15 mm OD shrouds made from either PTFE or PEEK. The ring electrode is permanently mounted in the RRDE tip, but the disk electrode may be permanently mounted or removable.



E6R1 Series ChangeDisk RRDE Tips (PTFE shroud)

These ring-disk electrode tips feature a PTFE shroud and the option to remove and replace the disk insert. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 10°C to 25°C

Do not use this electrode outside the operating temperature range.

Part numbers for ring assemblies, disk inserts, shafts, and tools are listed below. If the part number for a particular item is not listed, contact Pine Research for more details.

Ring Assemblies

platinum (PTFE shroud).....AFE6R1PT
gold (PTFE shroud).....AFE6R1AU
glassy carbon (PTFE shroud)AFE6R1GC

Disk Inserts (carbon materials)

glassy carbon.....AFED050P040GC
pyrolytic graphite
edge planeAFED050P040GE
basal planeAFED050P040GB

Disk Inserts (metal alloys)

1018 carbon steelAFED050P040S1
303 stainless steelAFED050P040T1
304 stainless steelAFED050P040T2
316 stainless steelAFED050P040T3
316L stainless steelAFED050P040T6
321 stainless steelAFED050P040T9
410 stainless steelAFED050P040T4
430 stainless steelAFED050P040T5
2205 stainless steelAFED050P040T7
Zeron 100 stainless steelAFED050P040T8
brassAFED050P040BR

Shafts and Tools

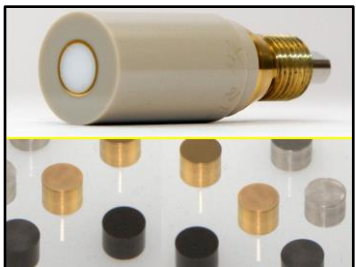
Precision RDE & RRDE shaft AFE6MB
Toolkit (disk removal & polishing).....AFE6K050

Disk Inserts (precious metals)

gold (Au)AFED050P040AU
platinum (Pt)AFED050P040PT
palladium (Pd).....AFED050P040PD
silver (Ag).....AFED050P040AG

Disk Inserts (common metals)

aluminum (Al)AFED050P040AL
cobalt (Co)AFED050P040CO
copper (Cu).....AFED050P040CU
iron (Fe).....AFED050P040FE
niobium (Nb)AFED050P040NB
nickel (Ni).....AFED050P040NI
lead (Pb).....AFED050P040PB
tin (Sn).....AFED050P040SN
tantalum (Ta).....AFED050P040TA
titanium (Ti).....AFED050P040TI
tungsten (W)AFED050P040WU
zinc (Zn).....AFED050P040ZN



E6R1PK Series ChangeDisk RRDE Tips (PEEK shroud)

These ring-disk electrode tips feature a PEEK shroud and the option to remove and replace the disk insert. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 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 prolonged exposure to concentrated acids.

Part numbers for ring assemblies, disk inserts, shafts, and tools are listed below. If a part number is not listed, contact Pine Research for more details.

Ring Assemblies

platinum (PEEK shroud) AFE6R1PTPK
gold (PEEK shroud) AFE6R1AUPK
glassy carbon (PEEK shroud) AFE6R1GCPK

Disk Inserts (carbon materials)

glassy carbon AFED050P040GC
pyrolytic graphite
edge plane AFED050P040GE
basal plane AFED050P040GB

Disk Inserts (metal alloys)

1018 carbon steel AFED050P040S1
303 stainless steel AFED050P040T1
304 stainless steel AFED050P040T2
316 stainless steel AFED050P040T3
316L stainless steel AFED050P040T6
321 stainless steel AFED050P040T9
410 stainless steel AFED050P040T4
430 stainless steel AFED050P040T5
2205 stainless steel AFED050P040T7
Zeron 100 stainless steel AFED050P040T8
brass AFED050P040BR

Shafts and Tools

Precision RDE & RRDE shaft AFE6MB
Toolkit (disk removal & polishing) AFE6K050

Disk Inserts (precious metals)

gold (Au) AFED050P040AU
platinum (Pt) AFED050P040PT
palladium (Pd) AFED050P040PD
silver (Ag) AFED050P040AG

Disk Inserts (common metals)

aluminum (Al) AFED050P040AL
cobalt (Co) AFED050P040CO
copper (Cu) AFED050P040CU
iron (Fe) AFED050P040FE
niobium (Nb) AFED050P040NB
nickel (Ni) AFED050P040NI
lead (Pb) AFED050P040PB
tin (Sn) AFED050P040SN
tantalum (Ta) AFED050P040TA
titanium (Ti) AFED050P040TI
tungsten (W) AFED050P040WU
zinc (Zn) AFED050P040ZN



E6R2 Series RRDE Tips

These ring-disk electrode tips feature a PEEK shroud and a PEEK gap between the permanently mounted disk and ring electrodes. The PEEK shroud permits these electrodes to be used at elevated temperatures. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 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 prolonged exposure to concentrated acids.

Part numbers for RRDE tips and shafts are listed below. If the part number for a particular item or electrode material is not listed, contact Pine Research for more details.

Description	Part Number
Precision RDE & RRDE Shaft (for 15 mm OD tips).....	AFE6MB
RRDE Tip (glassy carbon disk, platinum ring).....	AFE6R2GCPT
RRDE Tip (gold disk, platinum ring)	AFE6R2AUPT
RRDE Tip (platinum disk and ring)	AFE6R2PTPT
RRDE Tip (glassy carbon disk, gold ring).....	AFE6R2GCAU
RRDE Tip (gold disk and ring)	AFE6R2AUAU
RRDE Tip (platinum disk, gold ring)	AFE6R2PTAU



E7 Series ThinGap RRDE Tips

These ring-disk electrode tips feature a PTFE shroud and a thin PTFE gap (180 or 320 μm) between the permanently mounted disk and ring electrodes. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 10°C to 25°C

Do not use this electrode outside the operating temperature range.

Part numbers for RRDE tips and shafts are listed below. If the part number for a particular item or electrode material is not listed, contact Pine Research for more details.

Description	Part Number
Precision RDE & RRDE Shaft (for 15 mm OD tips).....	AFE6MB
ThinGap RRDE Tip (glassy carbon disk, platinum ring, 320 μm gap).....	AFE7R9GCPT
ThinGap RRDE Tip (glassy carbon disk and ring, 320 μm gap).....	AFE7R9GCGC
ThinGap RRDE Tip (gold disk and ring, 180 μm gap)	AFE7R8AUAU
ThinGap RRDE Tip (platinum disk and ring, 180 μm gap)	AFE7R8PTPT
ThinGap RRDE Tip (gold disk, platinum ring, 180 μm gap)	AFE7R8AUPT
ThinGap RRDE Tip (platinum disk, gold ring, 180 μm gap)	AFE7R8PTAU



E8 Series High Efficiency RRDE Tips

These ring-disk electrode tips feature a PEEK shroud and a PTFE gap between the permanently mounted disk and ring electrodes. The PEEK shroud permits these electrodes to be used at elevated temperatures. These tips fit the standard RRDE shaft and may be used at rotation rates up to 3000 RPM.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 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 prolonged exposure to concentrated acids.

Part numbers for RRDE tips and shafts are listed below. If the part number for a particular item or electrode material is not listed, contact Pine Research for more details.

Description	Part Number
Precision RDE & RRDE Shaft (for 15 mm OD tips).....	AFE6MB
RRDE Tip (glassy carbon disk, platinum ring).....	AFE8R4GCPT
RRDE Tip (glassy carbon disk, gold ring).....	AFE8R4GCAU
RRDE Tip (glassy carbon disk and ring).....	AFE8R4GCGC
RRDE Tip (gold disk and ring)	AFE8R4AUAU
RRDE Tip (platinum disk and ring)	AFE8R4PTPT
RRDE Tip (gold disk, platinum ring)	AFE8R4AUPT
RRDE Tip (platinum disk, gold ring).....	AFE8R4PTAU

5.6 15 mm OD RCE Tips

Complete rotating cylinder electrode (RCE) systems for the MSR evo rotator are available. These systems use a 15 mm diameter cylinder geometry.



15 mm OD RCE System

A typical 15-mm OD RCE system includes a 15 mm OD RCE shaft, a one liter corrosion cell equipped with a center-port gas-purged bearing assembly, a means of heating the contents of the cell, and other accessories.

The RCE shaft accepts standard cylinder samples (15 mm OD x 6.3 mm tall) fabricated from carbon steel or various stainless steels.

Contact Pine Research for more details about this system.



CAUTION:

Maximum Rotation Rate: 4000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 4000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 10°C to 80°C

Do not use this electrode outside the operating temperature range.

5.7 12 mm OD RCE Tips

Legacy users of 12 mm RCE systems are encouraged to begin working with the newer 15 mm RCE geometry; however, the 12 mm geometry remains available.



12 mm OD RCE Tips

Legacy 12 mm OD RCE tips accept cylinder inserts (12 mm OD x 7.96 mm tall) fabricated from carbon or stainless steel. Other materials are available on request. The 12 mm OD RCE tips fit on to a standard RCE/RDE shaft (part number AFE3M).

Contact Pine Research for details.



CAUTION:

Maximum Rotation Rate: 3000 RPM

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

ATTENTION:

Vitesse de rotation maximum: 3000 TR/MIN

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



OPERATING TEMPERATURE RANGE: 10°C to 25°C

Do not use this electrode outside the operating temperature range.

6 Maintenance

6.1 Routine Cleaning

Regular maintenance of the rotator primarily consists of keeping the external surfaces of the system clean by wiping them with a towel moistened with water or a mild, non-abrasive cleaner.

After about two weeks of continuous use, open the brush chamber and vacuum out any dust or debris. If necessary, remove the lower bearing assembly for better access to the brush chamber (see Section 6.3), and use a towel moistened with water or a mild, non-abrasive cleaner to clean the inner surfaces of the brush chamber.

The electrode brushes may deposit silver-carbon dust inside the brush chamber and deposit a film on the surface of the rotating shaft. A thin film on the shaft actually improves the contact between the brush and the shaft. This film does not need to be cleaned off of the shaft unless the film is rough or bumpy.

6.2 Brush Replacement

The brushes contact the rotating shaft, slowly wearing during normal use, and periodically, the brushes must be replaced. A simple brush replacement kit is available, or in the case of serious damage to the entire brush assembly, the brush and its nylon holder can be replaced.

6.2.1 Internal Brush Replacement



WARNING:

Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before continuing with this procedure.

AVERTISSEMENT:

Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant de poursuivre cette procédure.

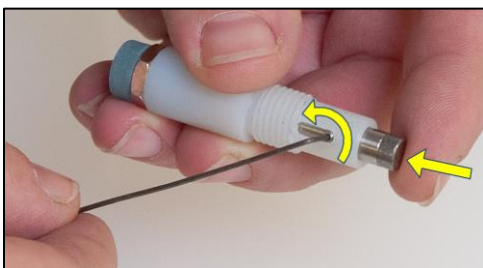


The standard brush replacement kit contains a small hex key, a new brush, and a new set screw (installed in the brush).

A special brush replacement kit should be used when the rotator is routinely operated in low humidity conditions such as inside a glove box.

Part numbers for brush replacement kits are listed in Section 7.1.

Remove the entire brush assembly from the rotator by unscrewing it as shown below. It should be possible to remove the brush assembly by hand.

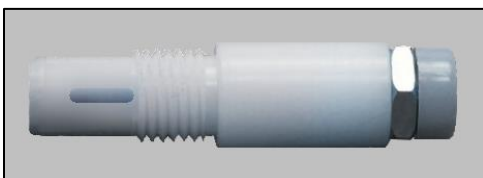


Use the small hex key to remove the set screw. Note that the required hex key (0.035") is included with the brush replacement kit.

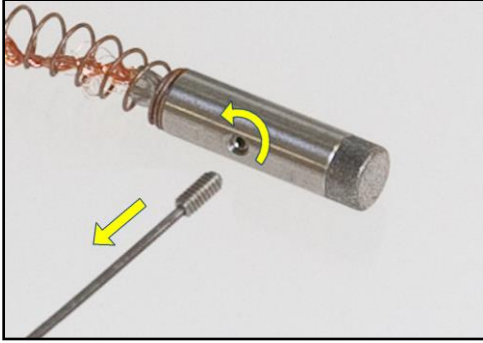


Note:

The brush is spring-loaded. When you remove the set screw, the brush will tend to fly out of the brush holder. Use a finger to hold it in place as you are removing the set screw.



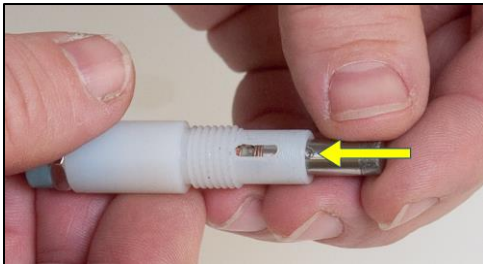
After removing the set screw, remove and discard the old brush, but do not discard the empty brush holder.



The new replacement brush includes a set screw that is already installed.

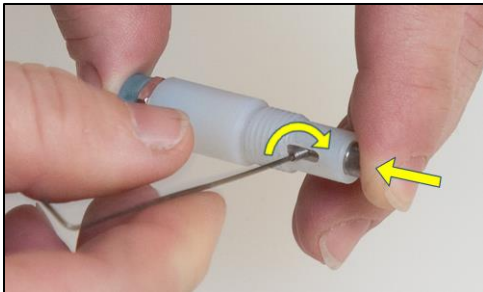
Temporarily remove this set screw.

Be careful not to misplace the set screw.



Carefully slide the new spring-loaded brush into the brush holder.

Be careful to properly align the set screw hole with the slot on the side of the brush holder.



While squeezing the new brush into the brush holder, use the hex key to reinstall the set screw.

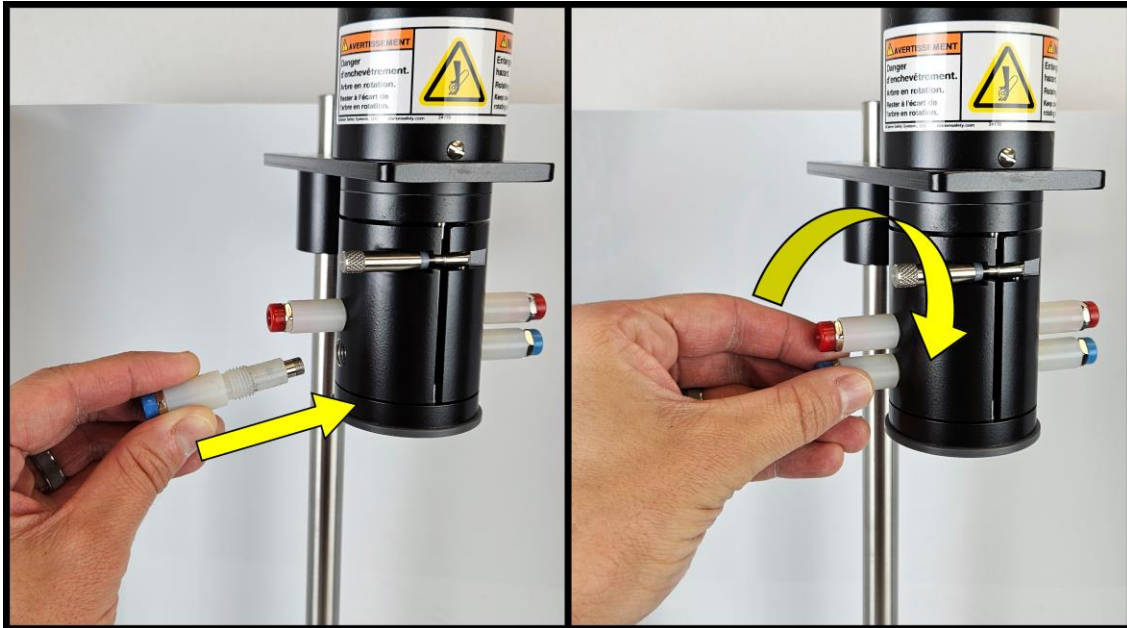
Tighten the set screw until it stops turning.



Note:

The set screw should protrude slightly into the slot, and the brush should be free to travel to the extent permitted by the width of the slot.

Reinstall the brush assembly by threading it back into the side of the rotator. Hand-tighten the brush assembly. Do not use tools to tighten the assembly.



INTENTIONAL WEAR PERIOD:

After installing a new brush, install a shaft and allow the rotator to run at 1000 RPM for at least eight (8) hours. This rotation period wears a concave groove into the new brush. This intentional wear actually improves the electrical contact between the brush and the shaft.

6.2.2 Complete Brush Assembly Replacement

In the event that the main body of the brush assembly is damaged, it may be necessary to replace the entire brush assembly.



WARNING:

Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before continuing with this procedure.

AVERTISSEMENT:

Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant de poursuivre cette procédure.

Remove the old brush assembly from the rotator by unscrewing it as shown below. Remove the old brush assembly by hand. (Use tools only if necessary!)



Install the new brush assembly by threading it by hand into the side of the rotator. Do not use tools to tighten the brush assembly.



INTENTIONAL WEAR PERIOD:



After installing a new brush, install a shaft and allow the rotator to run at 1000 RPM for at least eight (8) hours. This rotation period wears a concave groove into the new brush. This intentional wear actually improves the electrical contact between the brush and the shaft.

6.3 Lower Bearing Replacement

The lower bearing assembly is a common replacement item due to mechanical wear and also due to exposure to corrosive vapors from the cell solution. The standard lower bearing assembly contains a stainless steel bearing that is generally resistant to corrosive attack. Part numbers for the bearing assembly and other replacement parts are listed in Section 7.1.



WARNING:

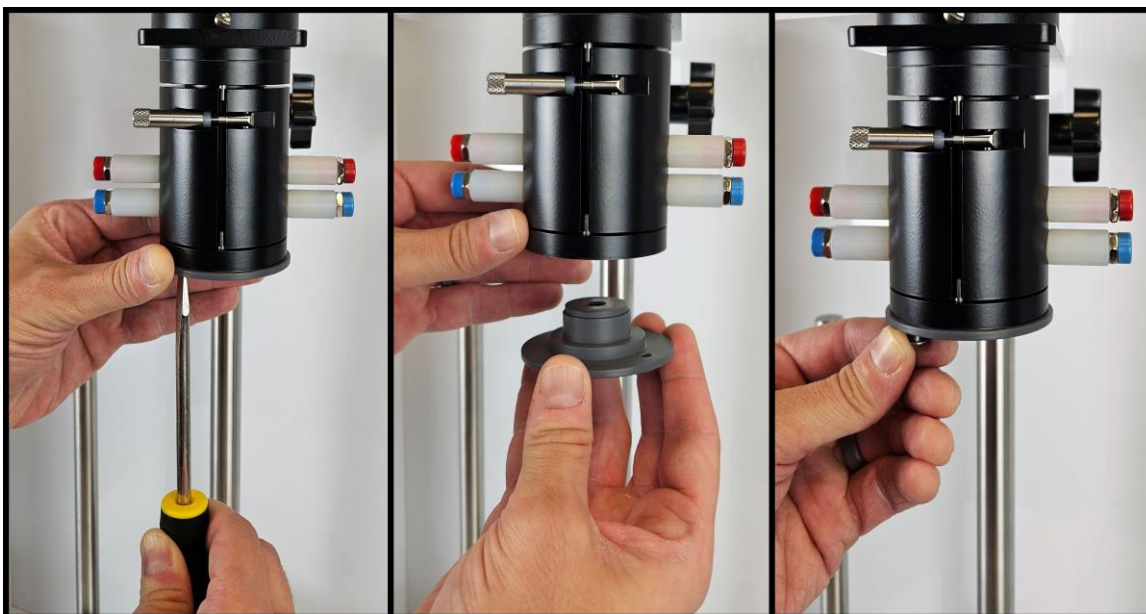
Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before continuing with this procedure.

AVERTISSEMENT:

Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant de poursuivre cette procédure.



Disconnect the motor control cable from the connector on top of the motor unit. If there is a shaft presently installed the motor unit, remove the shaft. Disconnect any signal cables from the brush banana jacks (red and blue).

Use a flathead screwdriver to loosen the four screws that secure the lower bearing assembly to the motor unit. As you are loosening the final screw with one hand, catch the bearing assembly with your other hand.



Note:

After the bearing assembly has been removed, it is a good idea to clean or vacuum out any debris in the brush chamber.

Align the four screw holes on the new bearing assembly with the four threaded holes in the motor unit.

Thread the four screws into the holes by hand. Then, tighten the screws with a flathead screwdriver.

6.4 Removing the Motor-Coupling Assembly

On rare occasions (such as when replacing a failed motor), it may be necessary to remove the motor-coupling assembly from the motor unit.



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.



WARNING:

Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before continuing with this procedure.

AVERTISSEMENT:

Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant de poursuivre cette procédure.

Disconnect the motor control cable from the top of the motor unit.

If there is a shaft presently installed in the motor unit, remove the shaft.

Disconnect any signal cables from the brush banana jacks (red and blue).

There are two screws that hold the cowling in place (front and back).

Use a flathead screwdriver to remove these two screws from the cowling.



Carefully begin removing the cowling from the motor unit.

The internal cable assembly will prevent the cowling from being completely removed.

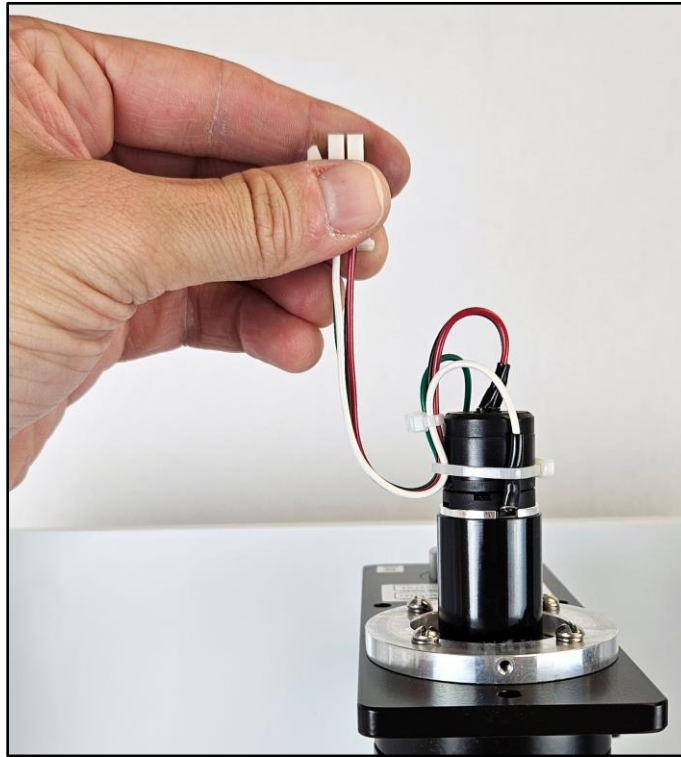
However, there is a junction in the middle of the internal cable assembly where two white connectors are joined together.

By disconnecting the cable assembly at this junction, it is possible to remove the cowling completely.

Disconnect the junction by releasing the locking mechanism that holds the connectors together.



The internal cables are secured to the motor using a plastic cable tie.
Do not remove or cut the cable tie.



CAUTION:

DO NOT REMOVE the cable tie.

The cable tie protects the fragile motor connections.

ATTENTION:

NE RETIREZ PAS l'attache de câble.

L'attache de câble protège les câbles fragiles de connexion au moteur.



Note:

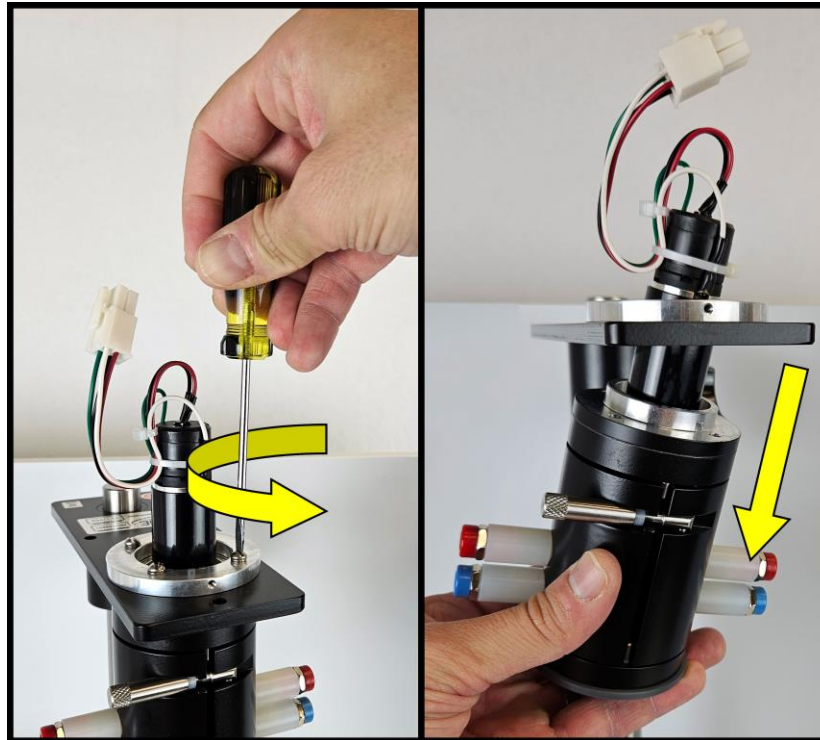
The red (positive) and black (negative) lines are connected to the tachometer, and the white (positive) and green (negative) lines are connected to the motor.

There are four screws that hold the motor in place.

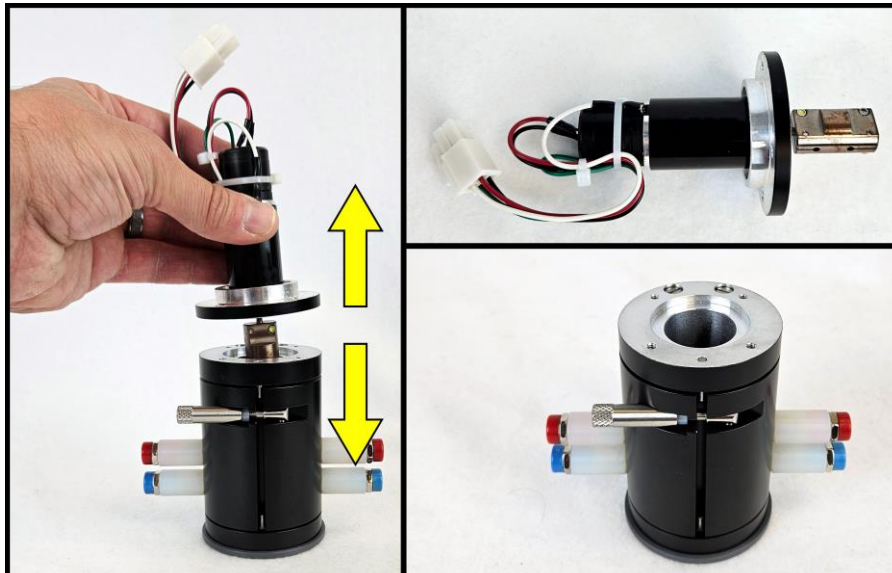
Using a flathead screwdriver, loosen and remove these four screws.

As the fourth and final screw is being removed, be sure to support the motor and brush chamber from below to prevent damage from a sudden fall.

Carefully lower the motor out of the support while guiding the fragile motor and tachometer cables through the support.



Carefully separate the motor from the brush chamber.



**Note:**

After the motor has been removed, it is a good idea to clean or vacuum out any debris in the brush chamber.

6.5 Installing a New Motor-Coupling Assembly

After removing the old motor-coupling assembly (see above), a new motor-coupling assembly may be installed.

**CAUTION:**

After installing a new motor, it is necessary to recalibrate the rotation rate using an optical tachometer (see Section 6.6).

ATTENTION:

Après l'installation d'un nouveau moteur, il est nécessaire de recalibrer la vitesse de rotation à l'aide d'un tachymètre optique (voir section 6.6).

**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.

**WARNING:**

Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before continuing with this procedure.

AVERTISSEMENT:

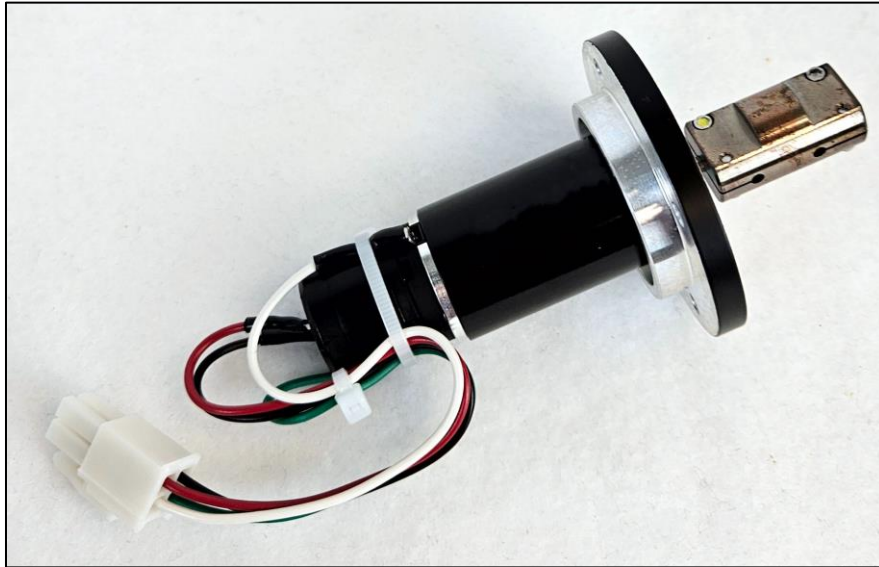
Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant de poursuivre cette procédure.

Disconnect the motor control cable from the top of the motor unit.

Disconnect any signal cables from the brush banana jacks (red and blue).

Examine the new motor coupling unit. There should be a cable tie securing the cables to the motor (black) as shown below. Do not remove this cable tie.



Remove any extra cable ties if they are present (*i.e.*, around the bottom part of the motor) so that the cable can move freely.

Align the threaded holes in the new motor with those in the brush chamber and push the motor up into the support. Carefully feed the cables through the hole as shown in the figure below.



Secure the motor and chamber to the support using four screws.

Connect the internal cable within the cowling to the motor by joining the two white connectors together.

Replace the cowling on top of the motor and secure it with two screws.

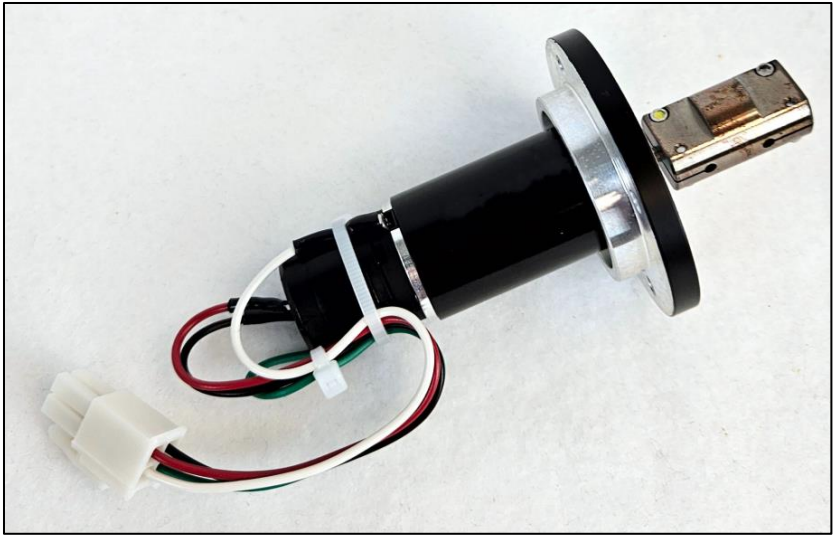


6.5.1 Motor Control Cable Wiring

The motor control cable is a 15-conductor cable that is wired “straight thru” from a male HD-15 connector on one end to a female HD-15 connector on the other end. Although there are 15 conductors, only four signals actually travel through the cable, and several conductors are grouped together (see Table 6-1). The HD-15 connector on top of the motor unit connects (internally) with the motor wiring harness that has four colored signal wires (see Table 6-2).

pins	description
1-4	Motor Supply (-)
6-9	Motor Supply (+)
11-12	Tachometer (+)
13-14	Tachometer (-)

Table 6-1. Motor Control Cable Wiring



wire color	description
green	Motor Supply (-)
white	Motor Supply (+)
red	Tachometer (+)
black	Tachometer (-)

Table 6-2. Internal Motor Cable Wiring

6.6 Rotation Rate Calibration

The rotation rate is controlled by a tunable analog feedback circuit located inside the control unit. This circuit is tuned and calibrated at the factory prior to shipment, but if the rotator needs to be recalibrated by the owner at a later date, the procedure below describes the best method for calibrating and verifying the rotation rate control circuit.

The most important tool required for calibrating the rotator is a non-contact **optical tachometer**. Pine Research offers such a simple tachometer (see Figure 6-1) as part of the MSR Calibration Kit, and this simple tachometer is suitable for routine verification or calibration of the rotation rate by the owner. The MSR Calibration Kit is sold separately (see Section 7.1 for part number).



Figure 6-1. Rotator Calibration Toolkit (including simple handheld digital tachometer)

In the event that the rotation rate must be rigorously traceable to a national or international standards organization, a more sophisticated and professional tachometer with traceable certification should be used (see Figure 6-2). When a rotator is manufactured at the factory (or when a rotator is returned to the factory for service), recalibration is performed using a traceable tachometer.



Figure 6-2. Professional Optical Tachometer with Traceable Calibration

The tachometer reads the rotation rate when it is pointed at a rotating shaft equipped with a **reflective target** (see Figure 6-3). The MSR Calibration Kit includes a shaft suitable for use as a reflective target. Alternately, a stainless steel rod (1/4 OD x 5" L; 6.35 mm OD x 100 mm L) may be mounted in the motor coupling, and a mark can be made on the rod with a marker.

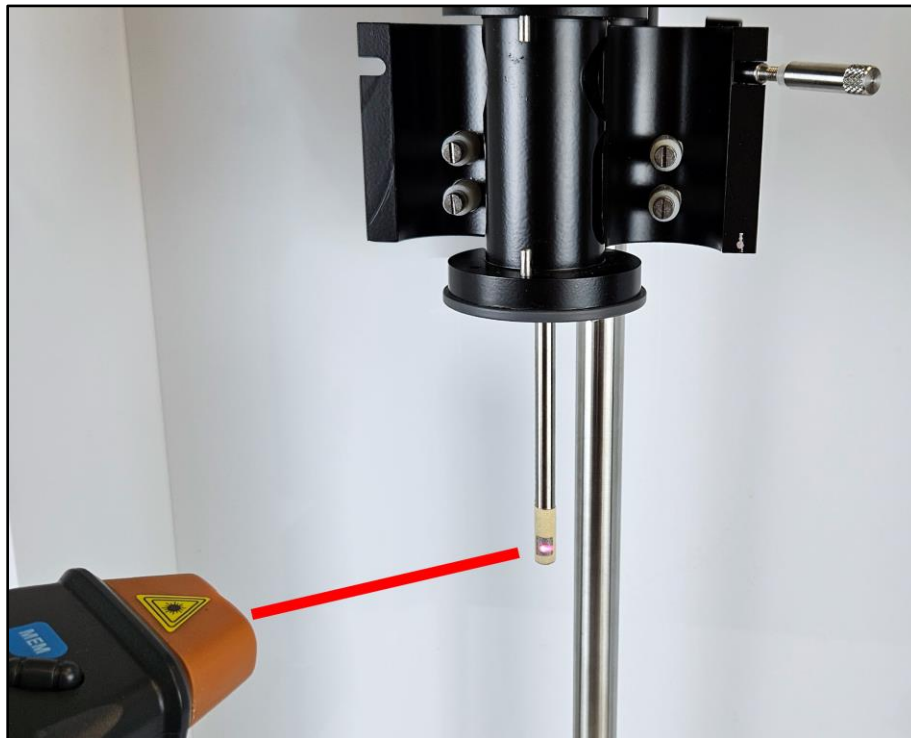


Figure 6-3. Use of Optical Tachometer with Reflective Target

The calibration process involves making adjustments to various trimmer potentiometers (trimmers) on the circuit board. The MSR Calibration Kit includes a **trimmer adjustment tool** for this purpose. Alternately, a small flathead screwdriver can be used to make these adjustments.

A calibrated **digital voltmeter** is required to confirm certain signal levels on the circuit board. It is recommended that a 4 ½-digit voltmeter be used for this purpose. The calibration process also requires a **known voltage source** (1000 mV). This known source can be a power supply or waveform generator, and the value of the known voltage (1000 mV) should be verified using the calibrated digital voltmeter.

Other tools required are a **#1 and #2 Pozidriv screwdriver** (alternatively, a medium-sized **Phillips screwdriver** may be used instead), a **3/16" (5 mm) hex driver**, **9/32" (7 – 8 mm) hex driver**, and a **5/64" (2 mm) hex driver** (to turn the hex screws on the motor coupling when installing or removing a shaft). This small hex key is included with the purchase of a new rotator and it is also available at many retail hardware supply stores.

Note that in all cases where a Pozidriv screwdriver is recommended, a Phillips screwdriver may be used as an alternative. However, be advised that the specific screws used with Pozidriv screwdrivers can become damaged or stripped over long periods of time if Phillips screwdrivers are used. Caution should be exercised when repeatedly using a Phillips screwdriver to loosen and tighten these screws.

**DANGER:**

High voltage. Risk of electric shock.

This procedure must be performed by an electrician or a qualified technician. This procedure requires working inside the control unit while the control unit is powered on and operating.

High voltages are present inside the control unit at the power entry module and on the two internal power supply modules as shown in the shaded and outlined portion of the image below.

KEEP HANDS AND TOOLS AWAY FROM THE POWER ENTRY MODULE AND THE TWO POWER SUPPLY MODULES!

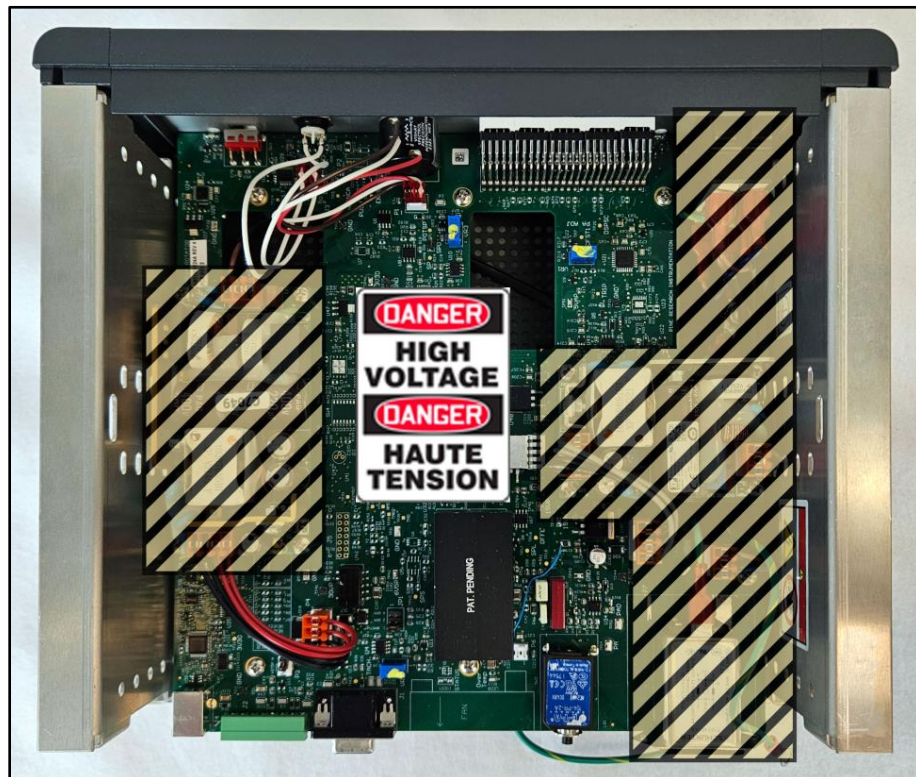
DANGER:

Haute Tension. Risque de décharge électrique.

Cette procédure doit être confiées à un électricien ou un technicien qualifié. Cette procédure requiert de travailler à l'intérieur de l'unité de commande lorsque cette dernière est alimentée et en fonctionnement.

Des tensions élevées sont présentes dans l'unité de commande au niveau du module d'entrée d'alimentation et sur les deux modules d'alimentation internes tel qu'indiqué dans la portion grisée et mise en évidence de l'image ci-dessous.

GARDEZ VOS MAINS ET VOS OUTILS ÉLOIGNÉS DU MODULE D'ENTRÉE D'ALIMENTATION ET DES DEUX MODULES D'ALIMENTATION!



**CAUTION:**

Static electricity may damage electronic components.

Ensure proper grounding when handling static sensitive components by wearing a grounding strap.

ATTENTION:

L'électricité statique est susceptible d'endommager les composants électriques.

Veillez à disposer d'une mise à la terre appropriée lorsque vous manipulez des composants sensibles en portant un ruban de mise à la terre.

**Note:**

When performing a traceable rotation rate calibration, verify the dates on the calibration certificates for the tachometer and voltmeter and confirm that the certificates have not expired.

6.6.1 Removing Top Panel of Control Unit

Turn the power switch on the front panel of the control unit off and disconnect the power cord.

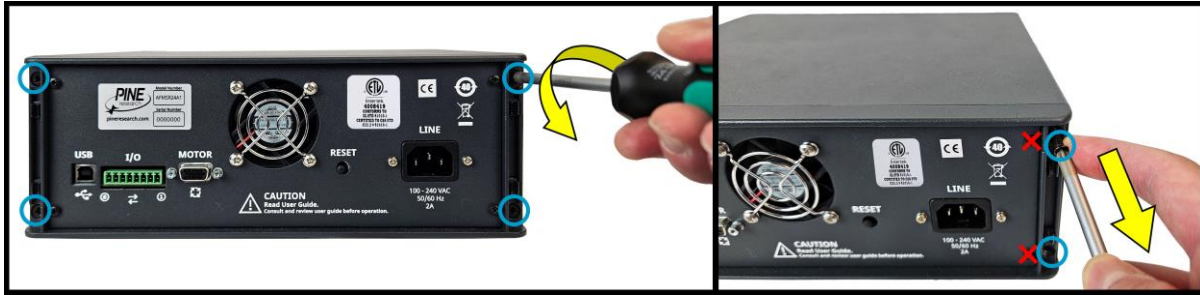
Disconnect the motor control cable from the control unit, as well as any other auxiliary cables.

With the power cord disconnected, first remove the two corner panel protectors located on the edges of the back panel of the control unit. This can be done by hand, or using a flathead screwdriver or similar tool if needed to pry the panels loose.



Using a #2 Pozidriv screwdriver (or a Phillips screwdriver), loosen and remove the four screws in the corners of the back panel.

Note: **Do not remove** the four screws near the corners that are slightly inset, as shown in the right image below with red Xs. The correct screws to remove are the ones that were underneath the previously-removed corner panel protectors.



Using a #1 Pozidriv screwdriver (or a Phillips screwdriver), loosen and remove the two screws on either side of the power cord connector.



Using a 3/16" (5 mm) hex driver, loosen and remove the two screws on either side of the motor cable connector.



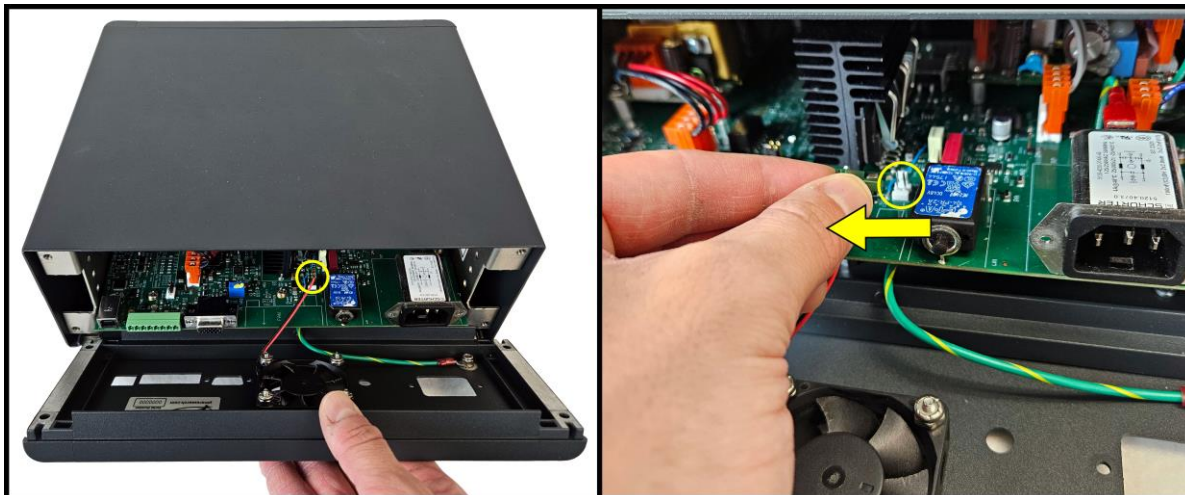
Using a #1 Pozidriv screwdriver (or a Phillips screwdriver), loosen and remove the centrally-located black screw on the bottom of the control unit near the back panel.

Do not remove the silver screw underneath the black screw (marked with a red X in the right image below).



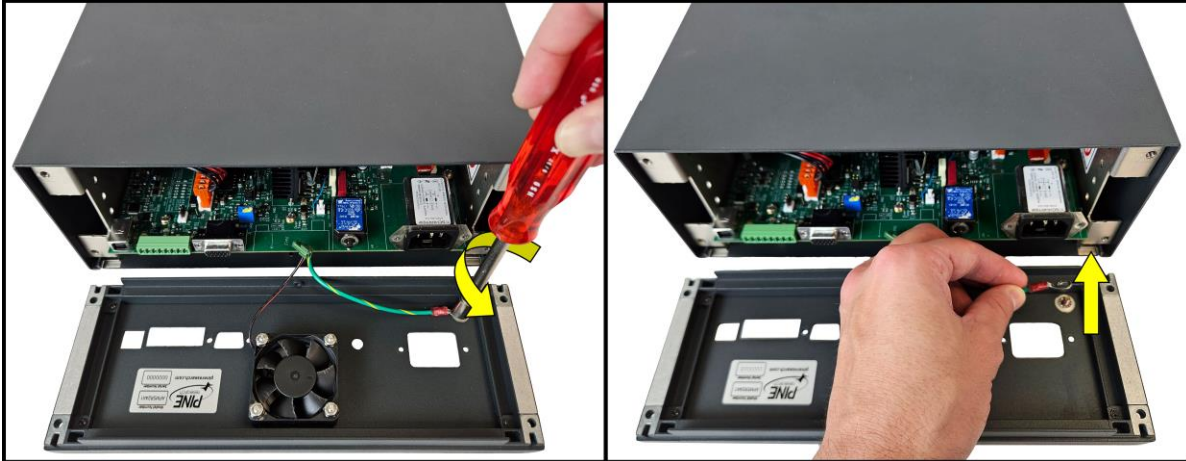
Loosen and then pivot the top of the back panel down, but **DO NOT FULLY PULL IT AWAY OR REMOVE IT FROM THE CONTROL UNIT YET**, as there are still several connections linking the back panel to the control unit.

First, with the back panel laying flat, unplug the connector to the cooling fan.



Using a 9/32" (7 – 8 mm) hex driver, loosen and remove the nut and lock washer connecting the grounding wire to the inside of the back panel.

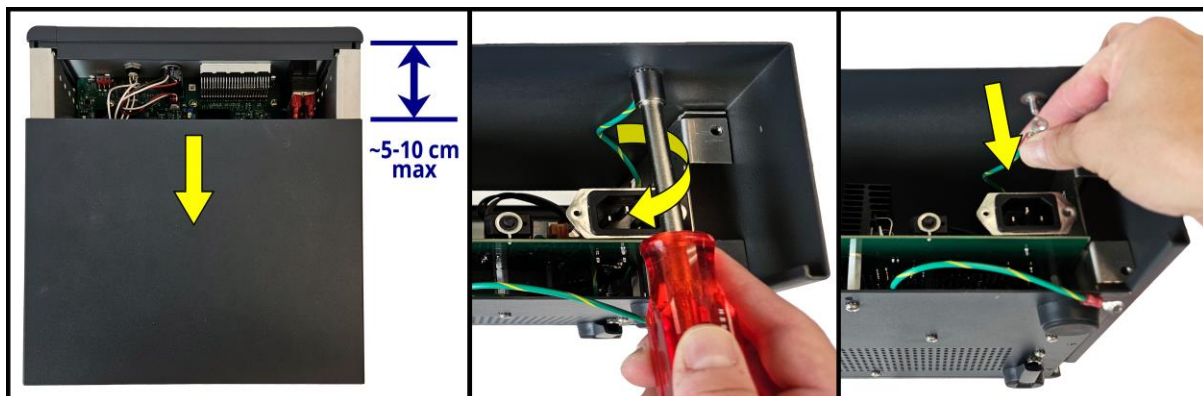
At this point, the back panel can now be completely removed from the control unit and placed carefully aside on the lab bench.



Carefully slide the top panel back around 5 – 10 cm, but **DO NOT FULLY SLIDE IT BACKWARDS OR REMOVE IT COMPLETELY YET.**

Using a 9/32" (7 – 8 mm) hex driver, loosen and remove the nut and lock washer connecting the grounding wire to the under side of the top panel.

At this point, the top panel can now slide back and be completely removed from the control unit, then placed carefully aside on the lab bench.



While the power is still switched off, the power cord unplugged, and the top panel removed, note the position of the three blue trimmers on the main circuit board. They are labeled “VR1,” “VR2,” and “VR3.” A trimmer adjustment tool (or a small flathead screwdriver) is required to adjust the small screw on the top of these trimmers.



Note that there may be a threadlock layer on the top of the trimmers blocking access to the screw. This typically yellow, paint-like film is often applied in the factory to prevent the trimmers from losing calibration during shipment or minor vibrations.

Prior to performing a calibration procedure, these films should be scraped away so the screws on each trimmer can be adjusted as needed.



6.6.2 Offset Calibration

Reconnect the power cord and motor control cable to the back of the control unit.

Connect the other end of the motor control cable to the top of the motor unit.

Turn the rate control knob fully counterclockwise. This is the position that corresponds to a nearly zero rotation rate.

Ensure the control switch on the front panel is toggled to the left position. Turn the power switch on. Ensure the motor does not rotate and that the status indicator LED is red.

Toggle the control switch to the right position. The status indicator LED should remain red. The external control indicator LED should turn yellow. The rotation rate display should read **0 RPM**.

Toggle the control switch to the left position. The status indicator LED should remain red. The external control indicator LED should turn off.

Confirm the rate control knob is still turned fully counterclockwise. Using a trimmer adjustment tool (or a small flathead screwdriver), adjust the top screw on trimmer **VR3** until the rotation rate display shows **0 RPM**.

Press the Run/Pause button. The status indicator LED should turn green.

Using a trimmer adjustment tool (or a small flathead screwdriver), adjust the top screw on trimmer **VR1** until the rotation rate display shows **0 RPM**. Ensure that the motor is at a complete stop or moving very slowly.

Press the Run/Pause button. The status indicator LED should turn red. Confirm the motor is still at a complete stop.

While in the “pause” state, measure the voltage between pins 3(+) and 6(-) of the green 8-pin external I/O port. The measured voltage should be $0.0 \pm 0.1 \text{ mV}$.



6.6.3 Manual Speed Calibration



WARNING:

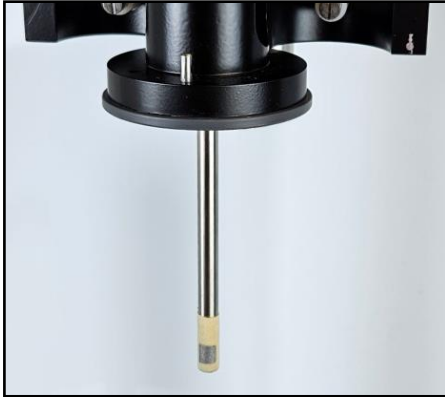
Laser radiation.

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.



Turn the power switch off. Install the tachometer target shaft into the motor coupling on the motor unit.

This target should be a metal rod with the appropriate diameter (1/4 " or 6.35 mm).

Many tachometers require that a piece of reflective tape be attached to the end of the shaft as shown.

Turn the power switch on. The status indicator LED should be red, and the rotator should be in the "pause" state.

Adjust the rate control knob on the front panel of the control unit until the rotation rate display reads **3000 RPM**. Ensure the rotator remains stationary while in the "pause" state.

Press the Run/Pause button. The status indicator LED should turn green, and the shaft will begin to rotate. The rotation rate display should continue to read **3000 ± 3 RPM**.

Point an **optical tachometer** at the reflective shaft and monitor the reading. Using a trimmer adjustment tool (or a small flathead screwdriver), adjust the top screw on trimmer **VR2** until the tachometer measures within **±1 RPM** of the control unit display.

Measure the voltage between pins 3(+) and 6(-) of the green 8-pin external I/O port. It should read **3000 ± 5 mV**, which corresponds to **±5 RPM** of the control unit display and tachometer reading.

Verify the manual speed calibration at several different rotation rates (suggested rates are **200, 500, 1000, 2000, and 5000 RPM**), following the previous steps described in this section. At each rotation rate, the rotation rate display on the front panel, the rotation rate measured by the **optical tachometer**, and the voltage measured between pins 3(+) and 6(-) of the green 8-pin external I/O port should all agree to within one percent (1%). The readings noted during this step should be recorded in a log book or on a certification sheet (see Section 6.6.5 for example certification sheet).



Note:

A convenient example certification sheet that can be used to record the verification readings can be found in Section 6.6.5.

6.6.4 External Input Speed Calibration



WARNING:

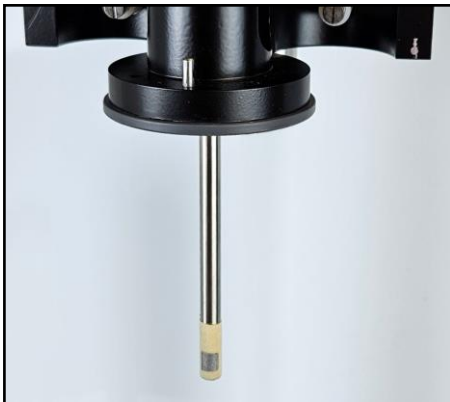
Laser radiation.

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.



Skip this step if the tachometer target shaft is already installed. If it is not already installed, continue as follows.

Turn the power switch off. Install the tachometer target shaft into the motor coupling on the motor unit.

This target should be a metal rod with the appropriate diameter (1/4" or 6.35 mm).

Many tachometers require that a piece of reflective tape be attached to the end of the shaft as shown.

Turn the power switch on. The status indicator LED should be red, and the rotator should be in the "pause" state.

Toggle the control switch to the right position. The status indicator LED should remain red. The external control indicator LED should turn yellow. The rotation rate display should read **0 RPM**.

Connect a DC voltage source to pins 1(+) and 6(-) of the green 8-pin external I/O port on the back of the control unit. Apply **3.000 V** using the DC voltage source. Press the Run/Pause button. The status indicator LED should turn green, and the shaft will begin to rotate. The rotation rate display should read **3000 ± 3 RPM**.

Point an **optical tachometer** at the reflective shaft and monitor the reading. Verify the tachometer measures **3000 ± 5 RPM**.

Verify the external input speed calibration at several different rotation rates (suggested rates are **200, 500, 1000, 2000, and 5000 RPM**, which correspond to DC voltage source inputs of **0.200, 0.500, 1.000, 2.000, and 5.000 V**, respectively), following the previous steps described in this section. At each rotation rate, the rotation rate display on the front panel and the rotation rate measured by the **optical tachometer** should agree to within one percent (1%). The readings noted during this step should be recorded in a log book or on a certification sheet (see Section 6.6.5 for example certification sheet).

**Note:**

A convenient example certification sheet that can be used to record the verification readings can be found in Section 6.6.5.

6.6.5 Example MSR evo Certification and Calibration Report

Rotation Rate Certification and Calibration Report	AFMSR24A1	_____
	Rotator Model and Serial Number	Technician (sign and date here)
	_____	_____
	Tachometer Make, Model, and SN	Tachometer Calibration Date
	_____	_____
	Voltmeter Make, Model, and SN	Voltmeter Calibration Date
	_____	_____

Control Unit Display and Output Signal Calibration

Control Unit Display Reading	Expected Rotation Rate (RPM)	Tachometer Reading (RPM)	Output Signal (V)
200	200 ± 2.0		
500	500 ± 5.0		
1000	1000 ± 10.0		
2000	2000 ± 20.0		
3000	3000 ± 30.0		
5000	5000 ± 50.0		

Control Unit Input Signal Calibration

Input Signal (V)	Expected Rotation Rate (RPM)	Tachometer Reading (RPM)	Control Unit Display Reading
0.200	200 ± 2.0		
0.500	500 ± 5.0		
1.000	1000 ± 10.0		
2.000	2000 ± 20.0		
3.000	3000 ± 30.0		
5.000	5000 ± 50.0		



Note:

Above 200 RPM, rotation rate is certified to be within $\pm 1\%$ of the value on the control unit display. From 100 to 200 RPM, the rate is certified to be within ± 2 RPM of the display reading.



Note:

The control unit and motor unit must be calibrated together as a system. This certification is valid only for the particular motor and control units with the serial number listed above.

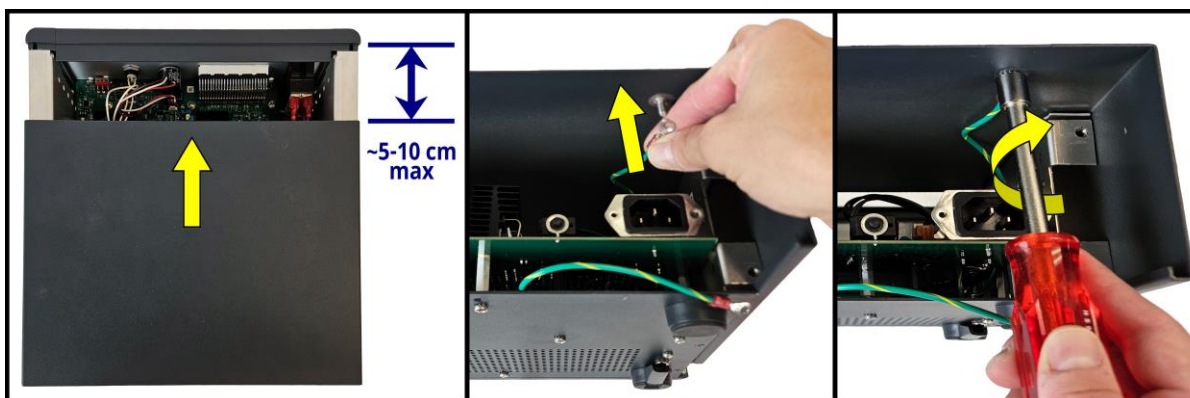
6.6.6 Replacing Top Panel of Control Unit

Turn the power switch on the front panel of the control unit off and disconnect the power cord. Disconnect the motor control cable from the control unit, as well as any other auxiliary cables.

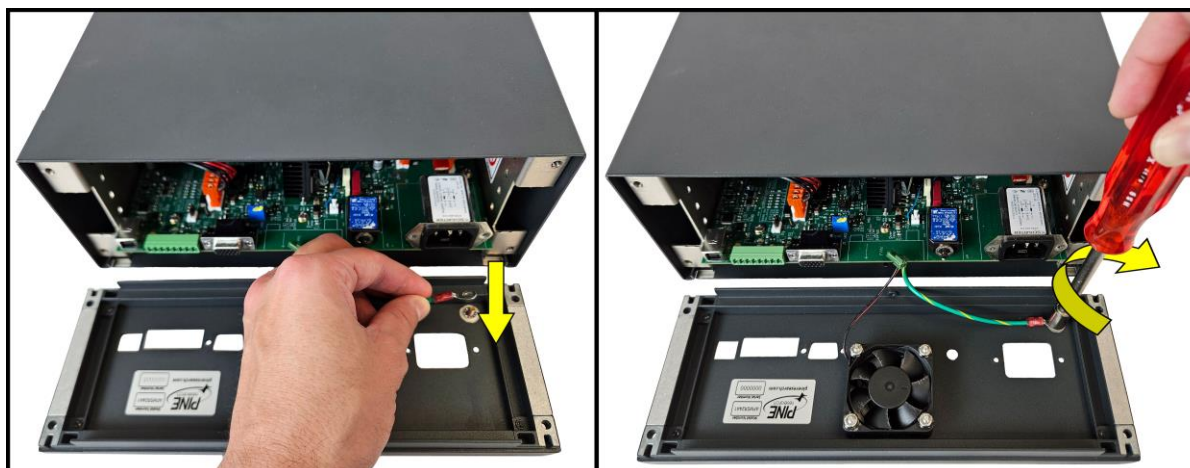
Carefully slide the top panel back onto the control unit until there is about 5 – 10 cm gap remaining, but **DO NOT FULLY SLIDE IT ALL THE WAY ON YET.**

Using a 9/32" (7 – 8 mm) hex driver, replace and tighten the nut and lock washer connecting the grounding wire to the under side of the top panel.

Once this grounding wire is secured, slide the top panel completely back onto the control unit.

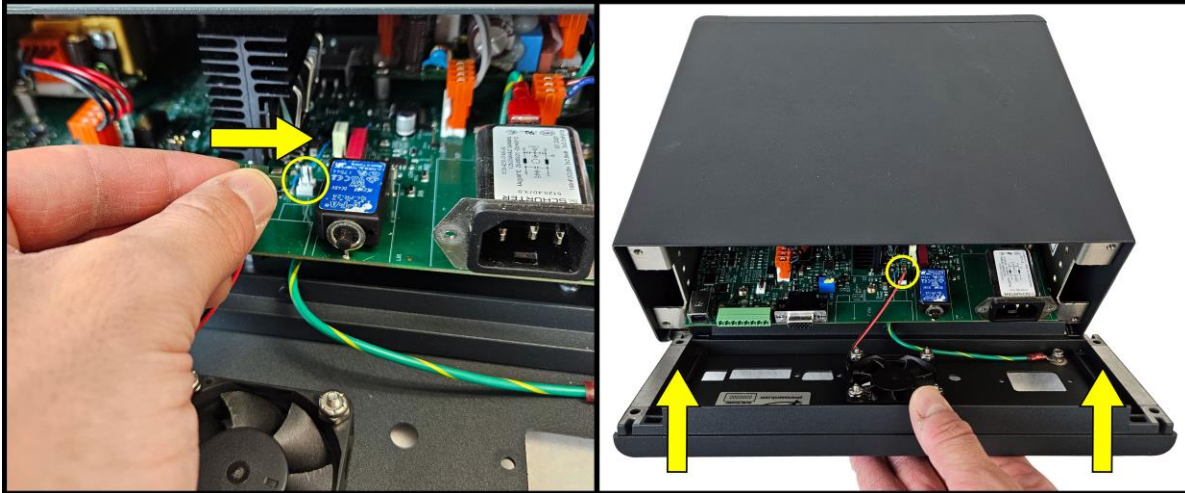


Obtain the back panel and move it until it is laying flat and near the back of the control unit. Using a 9/32" (7 – 8 mm) hex driver, replace and tighten the nut and lock washer connecting the grounding wire to the inside of the back panel.



Plug the connector to the cooling fan back into the main circuit board. Note the orientation of the plug and the pins on the circuit board. Ensure the tab on the plug faces the locking tab on the circuit board connector. The plug can mistakenly be inserted onto only a single pin instead of both pins if placed improperly. Be certain the connector is inserted properly onto both pins.

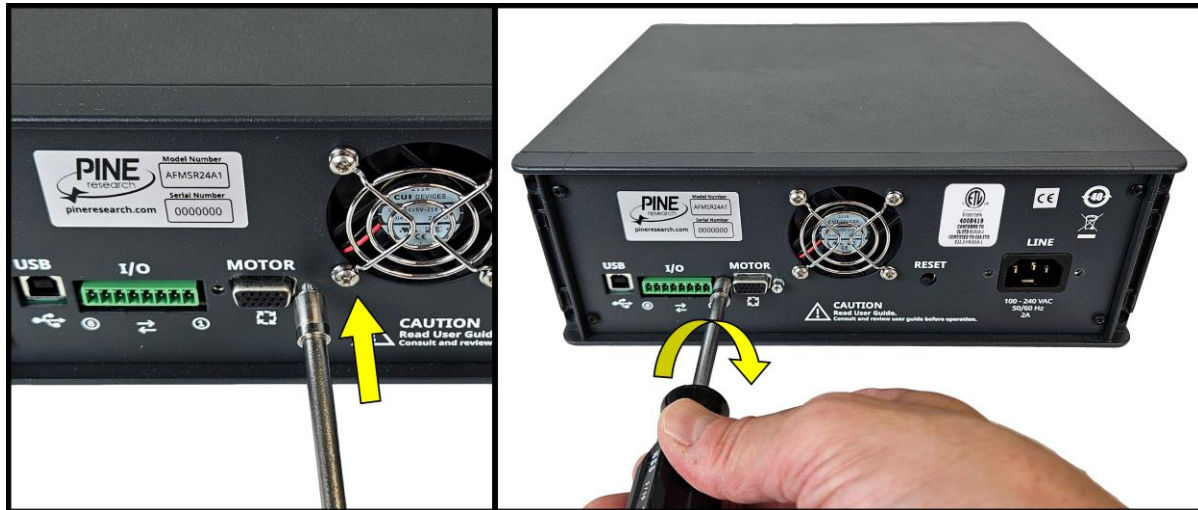
Replace the back panel of the control unit once the cooling fan connector is reattached.



Using a #1 Pozidriv screwdriver (or a Phillips screwdriver), replace and tighten the centrally-located black screw on the bottom of the control unit near the back panel.



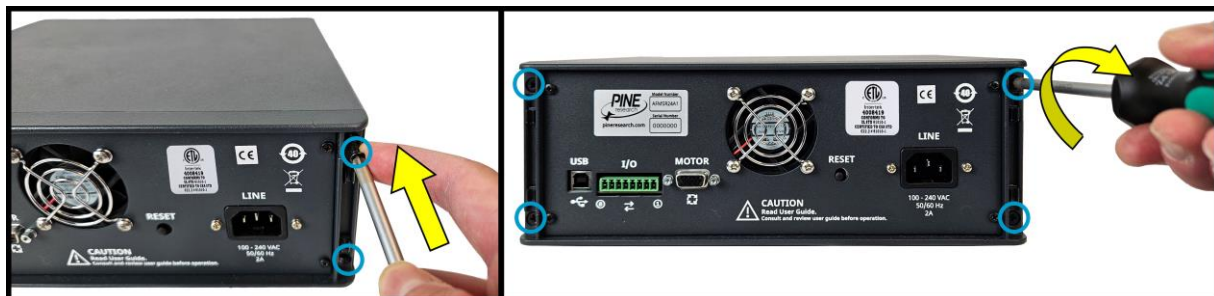
Using a 3/16" (5 mm) hex driver, replace and tighten the two screws on either side of the motor cable connector.



Using a #1 Pozidriv screwdriver (or a Phillips screwdriver), replace and tighten the two screws on either side of the power cord connector.



Using a #2 Pozidriv screwdriver (or a Phillips screwdriver), replace and tighten the four screws in the corners of the back panel.



Replace the two corner panel protectors located on the edges of the back panel of the control unit.



6.7 Changing the Input Rotation Rate Ratio

The rotation rate can be controlled by applying an external voltage signal to the external I/O port on the back panel of the control unit (see Section 4.6.3 for details). The proportionality ratio used to convert the applied voltage signal to the rotation rate can be set to one of three different values: **1, 2, or 4 RPM/mV**.

Normally, the value for the rotation rate ratio is selected to match the control signal provided by a particular potentiostat. When shipped from the factory, the MSR evo rotator is pre-configured with a ratio of **1 RPM/mV** because this ratio is compatible with Pine Research potentiostat systems.



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:

Static electricity may damage electronic components.

Ensure proper grounding when handling static sensitive components by wearing a grounding strap.

ATTENTION:

L'électricité statique est susceptible d'endommager les composants électriques.

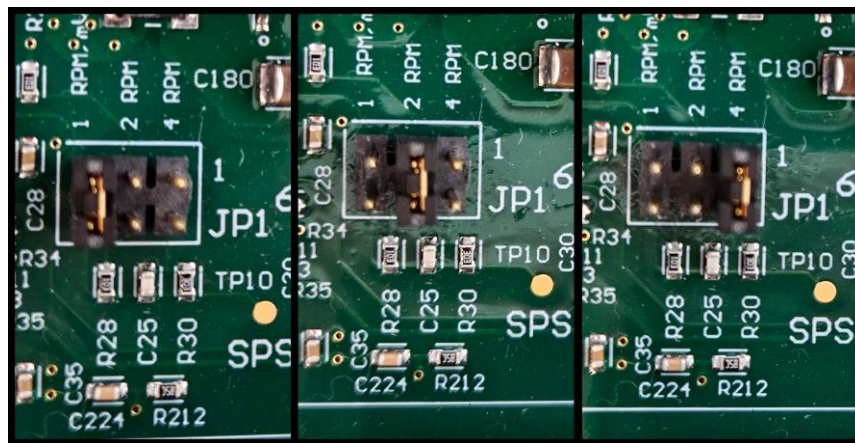
Veillez à disposer d'une mise à la terre appropriée lorsque vous manipulez des composants sensibles en portant un ruban de mise à la terre.

With the power cord removed from the control unit and the device powered off, remove the top panel of the control unit following the procedure shown in Section 6.6.1.

On the main circuit board, locate the configuration pins with the designation **JP1**. There is a small jumper that can be used to short together one of three pairs of pins.



Place the jumper across one of the three pairs of pins at **JP1**. Choose the ratio required for the particular potentiostat being used with the rotator.



1 RPM/mV

2 RPM/mV

4 RPM/mV

(Note: MSR evo rotator factory setting is **1 RPM/mV**)

Replace the cover on the control unit following the procedure shown in Section 6.6.6.

At this point the input ratio has been changed. Make a note in a log book or place a sticker on the control unit to indicate the new input ratio.

6.8 Changing the Motor Stop Signal Logic

When operating in external control mode (external control switch on the front panel of the control unit in the right position), an external digital signal can be applied across the Motor Stop and DC Common pins on the external I/O port on the MSR evo control unit (see Figure 2-7 for pinout of external I/O port). This digital signal can be used by a potentiostat or other external instrument to assure that the rotation rate is exactly zero. The logic for this digital signal may be either “active HIGH” or “active LOW.”

For the MSR evo, the motor stop is initially configured at the factory to use “active HIGH” logic. If desired, a jumper setting inside the control unit can be configured to use the opposite logic.

If the motor stop logic is configured to be “active HIGH,” then the motor is allowed to rotate if a signal greater than 2.0 V is applied across the Motor Stop and DC Common pins. If the two pins are shorted together (*i.e.*, if the motor stop stop signal is driven to ground), then the motor stops rotating.

If the motor stop logic is configured to be “active LOW,” then the motor will stop if a signal greater than 2.0 V is applied across the Motor Stop and DC Common pins. If the two pins are shorted together (*i.e.*, if the motor stop signal is driven to ground), 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 Motor Stop and DC Common pins, the motor is allowed to rotate. An internal “pull up” circuit assures that the motor stop signal remains “high” in this case.

Similarly, when the control unit is configured for “active LOW” logic and no connections are made to the Motor Stop and DC Common pins, the motor is allowed to rotate due to an internal “pull down” circuit.

Normally, the choice for the motor stop signal logic is selected to match the control signal provided by a particular potentiostat. When shipped from the factory, the MSR evo rotator is pre-configured with “active HIGH” logic because this logic is compatible with Pine Research potentiostat systems.



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:**

Static electricity may damage electronic components.

Ensure proper grounding when handling static sensitive components by wearing a grounding strap.

ATTENTION:

L'électricité statique est susceptible d'endommager les composants électriques.

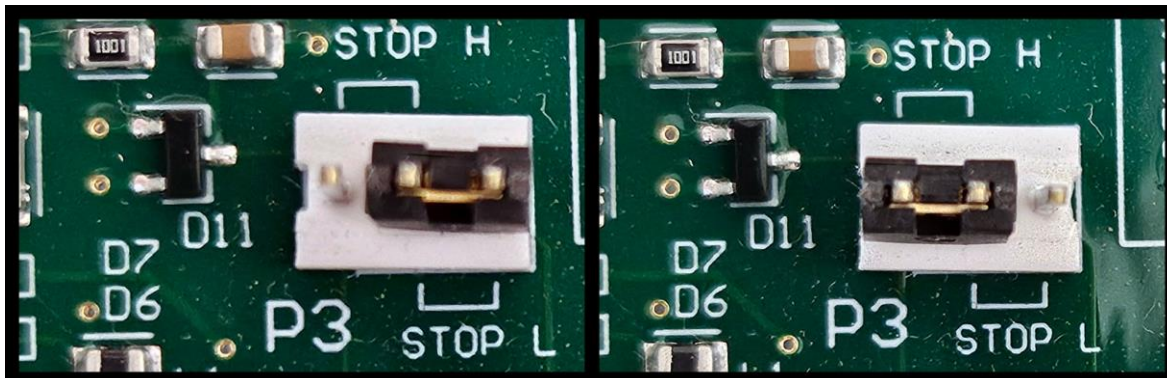
Veillez à disposer d'une mise à la terre appropriée lorsque vous manipulez des composants sensibles en portant un ruban de mise à la terre.

With the power cord removed from the control unit and the device powered off, remove the top panel of the control unit following the procedure shown in Section 6.6.1.

On the main circuit board, locate the configuration pins with the designation **P3**. There is a small jumper that can be placed in one of two positions at this location.



Place the jumper across one of the two positions shown below. Choose the position required for the particular potentiostat being used with the rotator.



Active HIGH Position

Active LOW Position

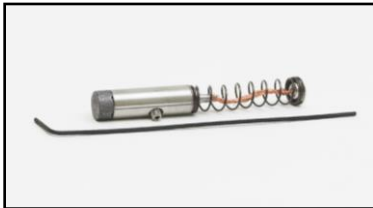
Replace the cover on the control unit following the procedure shown in Section 6.6.6.

At this point the motor stop signal logic has been changed. Make a note in a log book or place a sticker on the control unit to indicate the new logic.

7 Parts and Accessories

7.1 Mechanical Parts and Hardware

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



Brush Replacement Kit

Order this kit to replace a worn brush contact. This kit includes a spring-loaded brush and a required hex key tool. The replacement brush may be mounted in any of the four brush holders on the rotator. Special low humidity brushes are available for use in dry environments such as inside a glove box.

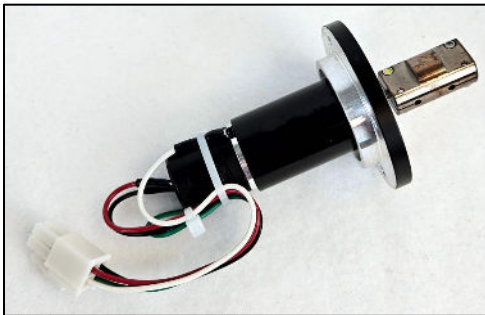
Description	Part Number
Standard Brush Kit	ACAR063RM
Low Humidity Brush Kit	ACAR063RMLHM



Complete Brush Assembly

To replace an entire brush assembly, order one of the parts below. This complete assembly includes the brush holder, a color coded banana jack, and a spring-loaded brush contact already mounted in the assembly.

Description	Part Number
Brush Assembly (blue)	ACMR3298XB-LF
Brush Assembly (red)	ACMR3298XR-LF
Brush Assembly (yellow)	ACMR3298XY-LF
Brush Assembly (green).....	ACMR3298XG-LF



Motor Coupling Assembly

The motor, motor coupling, and mounting flange are sold together as one single unit. Note that it is not possible to purchase these three items separately.

Description	Part Number
Motor Coupling Assembly	ACMR3165CE



Motor Coupling Hex Screws

This kit includes ten (10) replacement hex screws for use with the motor coupling. A pair of these screws is used to secure the rotating shaft inside the motor coupling. This kit also includes the hex key tool required to tighten these screws.

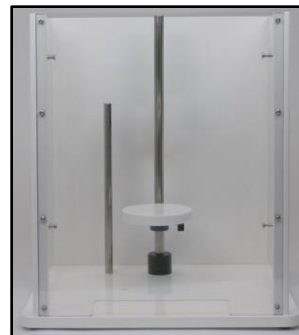
Description	Part Number
Motor Coupling Hex Screw Kit	AKMRHEX



Lower Bearing Assembly

The lower bearing assembly stabilizes the rotating shaft at the point where the shaft exits the brush chamber. The standard assembly has a stainless steel bearing.

Description	Part Number
Stainless-Steel Bearing Assembly ...	ACMR3301X



Enclosure Parts

The complete enclosure kit consists of everything in the photo above. The enclosure window and flask support post are also available separately.

Description	Part Number
Complete Enclosure Kit ...	AFMSR24-ENCLOSURE
Three-Pane Enclosure Window	ACMRN05
Flask Support Post (5/8" OD)	AC01MSRD



Three-Prong Lab Clamp

This three-pronged clamp fits a 24/25 center joint on an electrochemical cell. Standard right-angle bracket is included.

Description	Part Number
Three-Prong Clamp	AKCLAMP



Round Cell Clamp

This clamp is for use with large round cells with outer diameters between 140 and 165 mm. Standard right-angle bracket is included.

Description	Part Number
Round Cell Clamp	AKCLAMP2



Cell Platform

The cell platform is fabricated from chemically-resistant polypropylene and mounts anywhere along the center post.

Description	Part Number
Cell Platform	ACPR103



Motor Control Cable

The motor control cable has HD-15 connectors on either end and is used to connect the control unit to the motor unit. Note that two or three of these cables may be connected in series to allow for a greater distance between the control unit and the motor unit.

Description	Part Number
Motor Control Cable	EWC15DSUB



Rotation Rate Calibration Kit

This kit contains a handheld tachometer, trimmer adjustment tool, and a shaft with a reflective target (see Section 6.6 for instructions regarding the use of this kit).

Description	Part Number
Rotation Rate Calibration Kit.....	AKMSRCAL



Precision Shaft Alignment Kit

This kit contains a dial indicator (with mounting apparatus) used to measure the “runout” at the bottom end of the shaft (instructions for use are included with the kit).





Description	Part Number
Precision Shaft Alignment Kit.....	AKDIAL







7.2 Power Cords

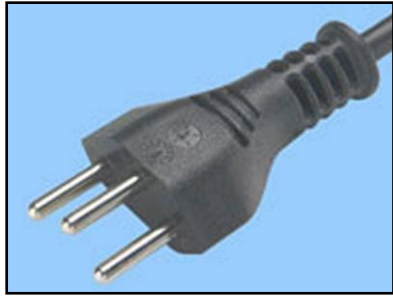
The C14 power entry receptacle on the back panel of the control unit accepts any power cord with a standard C13 plug (see Figure 7-1). A wide range of power cord options are described below.



Figure 7-1. Standard C13 Plug (left) and C14 Control Unit Power Entry Receptacle (right)

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

 <p>This cord is for use in South Africa.</p> <p>Description Part Number Power Cord (South Africa) EWM18B8SA</p>	 <p>This cord is for use exclusively in Israel.</p> <p>Description Part Number Power Cord (Israel) EWM18B8IL</p>
 <p>This cord is for use exclusively in Japan.</p> <p>Description Part Number Power Cord (Japan)EWM18B8JP</p>	 <p>This cord is for use exclusively in Argentina.</p> <p>Description Part Number Power Cord (Argentina)EWM18B8AR</p>
 <p>This cord is for use exclusively in Denmark.</p> <p>Description Part Number Power Cord (Denmark)EWM18B8DK</p>	 <p>This cord is for use in Australia & New Zealand.</p> <p>Description Part Number Power Cord (Australia) EWM18B8NZ</p>



This cord is for use exclusively in Switzerland.

Description	Part Number
Power Cord (Switzerland).....	EWM18B8CH



This cord is for use exclusively in Italy.

Description	Part Number
Power Cord (Italy)	EWM18B8IT

7.3 Power Supplies

The rotator control unit has two internal DC power supplies. One of these power supplies is 30 VDC (100 W) and the other is 24 VDC (60 W). These power supplies are located within the high voltage section of the control unit (see Figure 7-2).



DANGER:

High voltage. Risk of electric shock.

High voltages are present inside the control unit at the power entry module and on the two internal power supply modules as shown in the shaded and outlined portion of the image below.

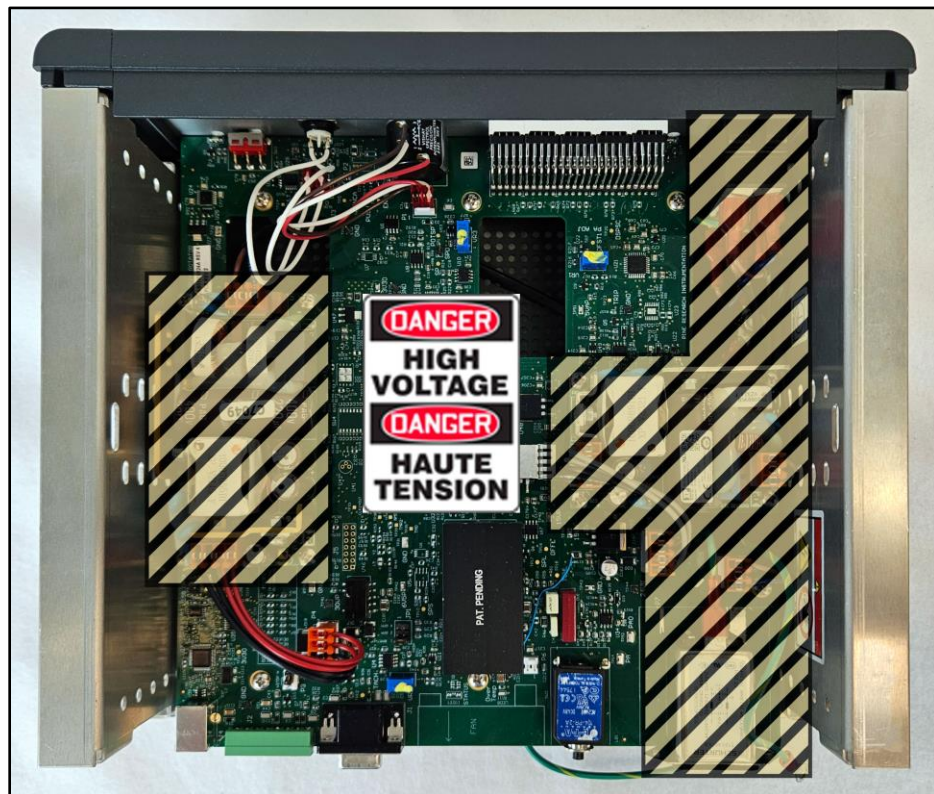
KEEP HANDS AND TOOLS AWAY FROM THE POWER ENTRY MODULE AND THE TWO POWER SUPPLY MODULES!

DANGER:

Haute Tension. Risque de décharge électrique.

Des tensions élevées sont présentes dans l'unité de commande au niveau du module d'entrée d'alimentation et sur les deux modules d'alimentation internes tel qu'indiqué dans la portion grisée et mise en évidence de l'image ci-dessous.

GARDEZ VOS MAINS ET VOS OUTILS ÉLOIGNÉS DU MODULE D'ENTRÉE D'ALIMENTATION ET DES DEUX MODULES D'ALIMENTATION!



**CAUTION:**

Static electricity may damage electronic components.

Ensure proper grounding when handling static sensitive components by wearing a grounding strap.

ATTENTION:

L'électricité statique est susceptible d'endommager les composants électriques.

Veillez à disposer d'une mise à la terre appropriée lorsque vous manipulez des composants sensibles en portant un ruban de mise à la terre.

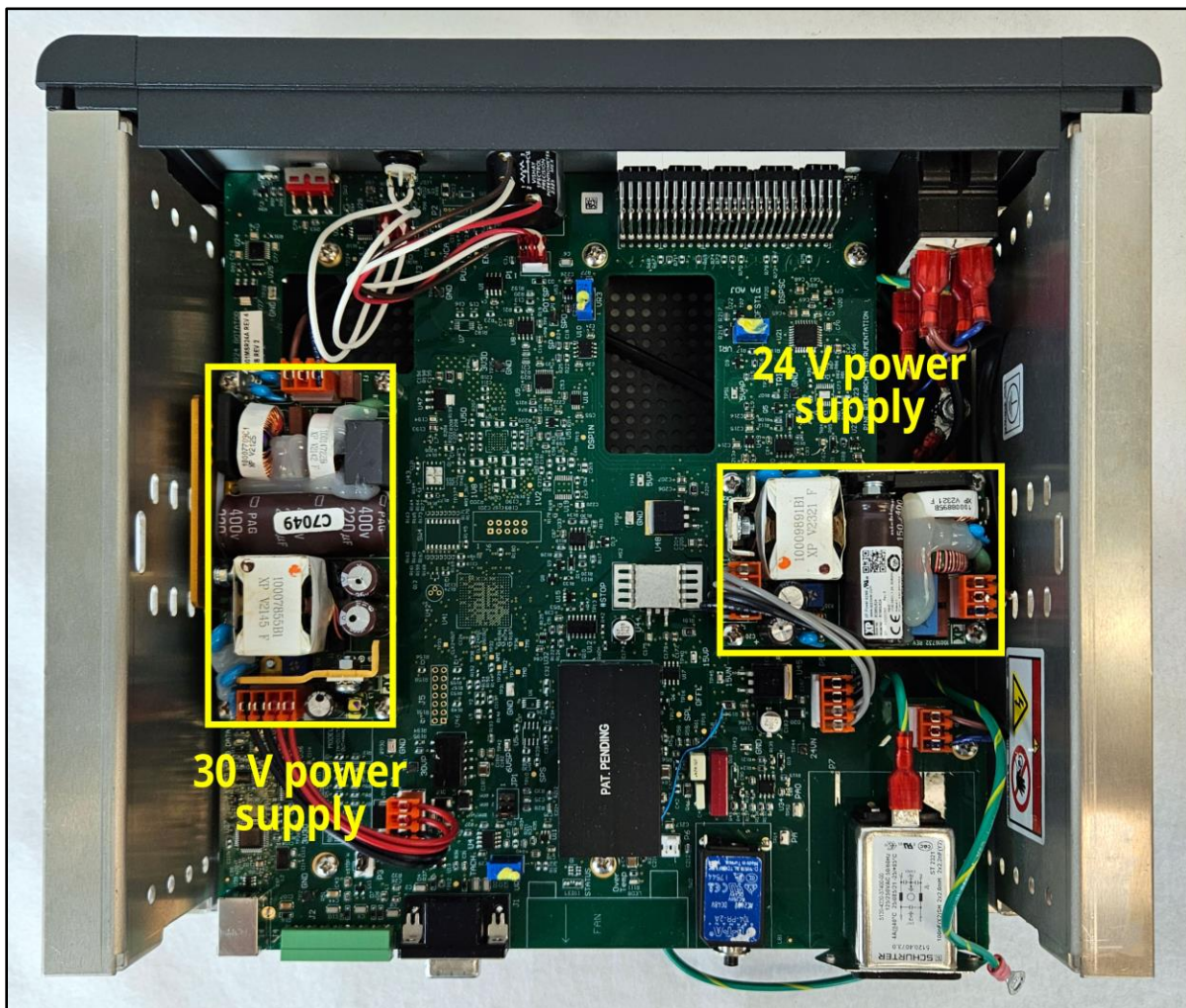


Figure 7-2. Location of High Voltage Power Entry and Internal Power Supplies



WARNING:

Risk of electric shock.

Disconnect power before servicing the control unit.

The shaded region above shows the location of the high voltage power entry and the two internal power supplies.

AVERTISSEMENT:

Risque de décharge électrique.

Déconnectez l'alimentation avant de procéder à l'entretien de l'unité de commande.

La région ombragée ci-dessus présente l'emplacement de l'entrée de puissance à haute tension et des deux alimentations internes.

In the (rare) event that it is necessary to replace one of the internal power supply units, it is important to carefully identify which type of power supply needs to be replaced. Replacement part numbers for each type of power supply are provided below (see Figure 7-3). Note that there are several connectors and mounting screws securing each power supply to the circuit board. Contact Pine Research for instructions on removing and replacing MSR evo power supplies.

<p>Internal Power Supply (30V)</p>	<p>Internal Power Supply (24V)</p>								
<table border="0"> <tr> <td>Description</td> <td>Part Number</td> </tr> <tr> <td>Power Supply (30 V, 100 W)</td> <td>AC01M30V</td> </tr> </table>	Description	Part Number	Power Supply (30 V, 100 W)	AC01M30V	<table border="0"> <tr> <td>Description</td> <td>Part Number</td> </tr> <tr> <td>Power Supply (24 V, 60 W)</td> <td>EE60W24</td> </tr> </table>	Description	Part Number	Power Supply (24 V, 60 W)	EE60W24
Description	Part Number								
Power Supply (30 V, 100 W)	AC01M30V								
Description	Part Number								
Power Supply (24 V, 60 W)	EE60W24								

Figure 7-3. Replacement Power Supplies for MSR evo Control Unit



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.

8 Troubleshooting

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

8.1 Problem: Continuous Rotation at a High Rate

- If the rotation rate is extremely or uncontrollably high for more than four seconds (> 4 s), the motor will stop and the fault indicator LED will illuminate red.
- Check the motor control cable that 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 cause may be a faulty connection or wire. If this is the case, contact Pine Research.

8.2 Problem: No Rotation

- Confirm that the unit is connected to a live power outlet.
- Confirm that the power switch has not tripped and that it is in the "on" position. Confirm that the fan on the back of the control unit is running. Reset the switch if necessary.
- If the fault indicator LED is illuminated, check the back panel circuit breaker and reset the breaker if necessary.
- Check the motor control cable that 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.
- With the rotator in the "pause" state, check the RPM set point. Increase the set point by turning the rate control knob clockwise if it is currently set to 0 RPM.
- 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. First, turn off the rotator, and disconnect the power.



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.

**WARNING:**

Rotating shaft. Entanglement hazard.

Turn off the power to the rotator and disconnect the power cord from the power source before continuing with this procedure.

AVERTISSEMENT:

Arbre en rotation. Danger d'enchevêtrement.

Éteignez le rotateur et débranchez le cordon d'alimentation de la source d'alimentation avant de poursuivre cette procédure.

- Check for freedom of rotation of the shaft by manually attempting to rotate the shaft.
- Next, look inside the control unit and confirm that there are no loose connectors or damage to the printed circuit board.

8.3 Problem: System Power Loss

- The main power switch on the front panel is a circuit breaker that may trip and cause the system to lose power. To reset the breaker, turn the switch off and then turn the switch on again. Repeated tripping may indicate a more serious problem.

8.4 Problem: Back Panel Circuit Breaker Trips

- This breaker only trips if the current passing through the motor windings is high enough to potentially damage the motor. This could occur if the electrode is rotating in a particularly viscous liquid, if the shaft is rubbing against something, or if an applied periodic waveform controlling the rotation rate has too great an amplitude or frequency.
- This breaker (thermal type) is sized to limit the average motor current to within the motor specification. Running the motor at a high modulation frequency, or with large amplitude changes, or a combination of the two, may cause tripping. It may be necessary to reduce the modulation frequency and/or amplitude to prevent tripping of the breaker.

8.5 Problem: Motor Rotates Backwards

- When the rate control knob is in the full counterclockwise position, it is natural to expect that the rotation rate should be exactly zero. However, it is normal for there to be a small residual rotation rate, and often this is in the reverse direction. Note that the proper direction of rotation for the shaft is clockwise when viewed from above the motor.
- If a negative voltage signal is applied to the external I/O port on the back panel of the control unit, then the rotator will rotate backwards. If this is undesirable, reverse the polarity of the applied signal.

8.6 Problem: Excessive Audible Noise

- If the rotator has a standard lower bearing assembly with a stainless steel bearing, then this bearing may be corroded. If corroded, replace the entire lower bearing assembly.
- It is also possible for the internal spindle bearings to be worn. If this is the case, contact Pine Research.

8.7 Problem: Electrical Noise in Voltammograms (Environmental)

- 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.

8.8 Problem: Electrical Noise in Voltammograms (Grounding Issues)

- See Section 4.10 for more information on grounding strategies and definitions.
- Confirm that the earth ground connection on the rotator is connected to the chassis ground of the potentiostat.
- Confirm that all metal objects (such as cell clamps and ring stands) near the electrochemical cell are connected to the earth ground connection on the rotator.
- Confirm that all grounding connections are made to a common grounding point to avoid the formation of “grounding loops.” Note that grounding loops are sometimes non-obvious, especially when multiple instruments and computers are connected together.

8.9 Problem: Electrical Noise in Voltammograms (Brush Wear)

- Consider using a banana jumper cable to connect opposing brushes together. Two brushes in opposing contact may provide a better electrical connection.
- Inspect all brush contacts. Brushes should have a concave groove worn in them that exactly mates with the rotating shaft. The depth of this concave groove naturally increases over the lifetime of the brush. A new brush should be worn continuously for approximately eight hours to intentionally wear a groove into the brush to increase the surface area of the brush that is in contact with the shaft.

8.10 Problem: Electrical Noise in Voltammograms (Cell Connections)

- 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 that are shielded (coaxial) cables. Each electrode should be connected to the inner signal line of the coaxial cable (often using an alligator clip). The outer shield line of the coaxial cable is typically actively driven to a potential that protects the inner signal line. Do not ground such a driven shield line as it may cause the potentiostat to oscillate or malfunction.
- Confirm that any alligator clips being used for connection to the electrodes are not corroded and are securely fastened to the electrodes.

9 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.

10 Theory

10.1 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 techniques^[1] (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 forcing^[2] 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]

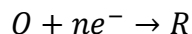
By far the most popular and widely used hydrodynamic methods are those that involve a rotating electrode. The rotating electrode geometries most amenable to mathematical modeling are the rotating disk electrode (RDE),^[9-14] the rotating ring-disk electrode (RRDE),^[15-26] and the rotating cylinder electrode (RCE).^[27-32] Researchers take advantage of the stable, 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 scientists^[44-69] 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.

10.2 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

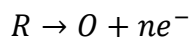
sufficiently negative potential is applied to the rotating electrode, the molecules (or ions) tend to be reduced.

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:

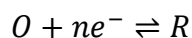


where R represents the reduced form of the molecule (or ion), O represents the oxidized form of the molecule (or ion), and n is the total number of electrons added to the molecule (or ion) when it is converted from the oxidized form (O) to the reduced form (R).

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^0) that 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 mol/L, all gases are present at 1 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:

$$E_{Nernstian} = E^0 + \left(\frac{RT}{nF}\right) \ln\left(\frac{a_R}{a_O}\right)$$

where F is Faraday's constant ($F = 96485 \text{ C/mol}$), R is the ideal gas constant ($R = 8.3145 \text{ J/mol K}$), and T is the temperature (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 follows:

* 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.

$$E_{Nernstian} = E^0 + \left(\frac{RT}{nF}\right) \ln\left(\frac{C_R}{C_O}\right)$$

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 ($E_{Applied}$) that is more negative than the equilibrium electrode potential ($E_{Applied} < E_{Nernstian}$). Conversely, the half reaction can be driven in the oxidizing (anodic) direction by applying a potential more positive than the equilibrium electrode potential ($E_{Applied} > E_{Nernstian}$).

10.3 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 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/s) 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/s) swept in the negative (cathodic) direction (see Figure 10-1, left). As the applied potential approaches the standard electrode potential, a cathodic current is observed (see Figure 10-1, right). The cathodic current continues to increase as the potential moves past the standard electrode potential towards more negative potentials.

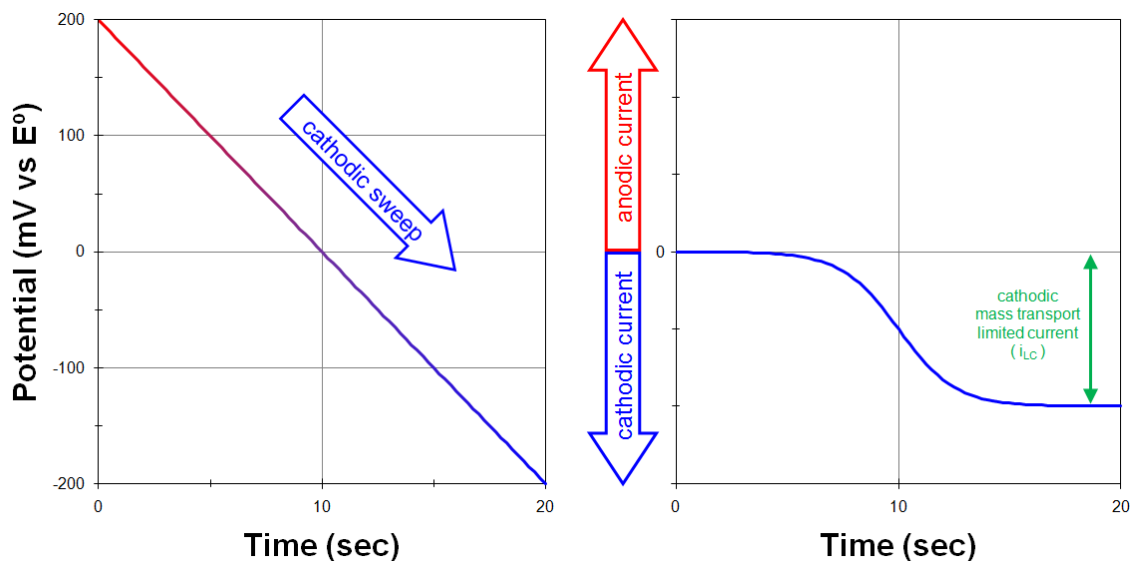
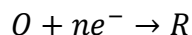


Figure 10-1. Response to a Potential Sweep (Cathodic) from a Solution Initially Containing only the Oxidized Form (O) with no Reduced Form (R)

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:

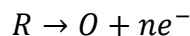


The observed cathodic current is the result of electrons flowing out of the electrode and into the solution. The rate of electron flow is limited only by how fast the oxidized form (O) can arrive at the electrode surface. The maximum current observed in this circumstance is called the cathodic limiting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

The cathodic sweep experiment described above (see Figure 10-1) 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 10-2, left) and an anodic current is observed (see Figure 10-2, 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}).

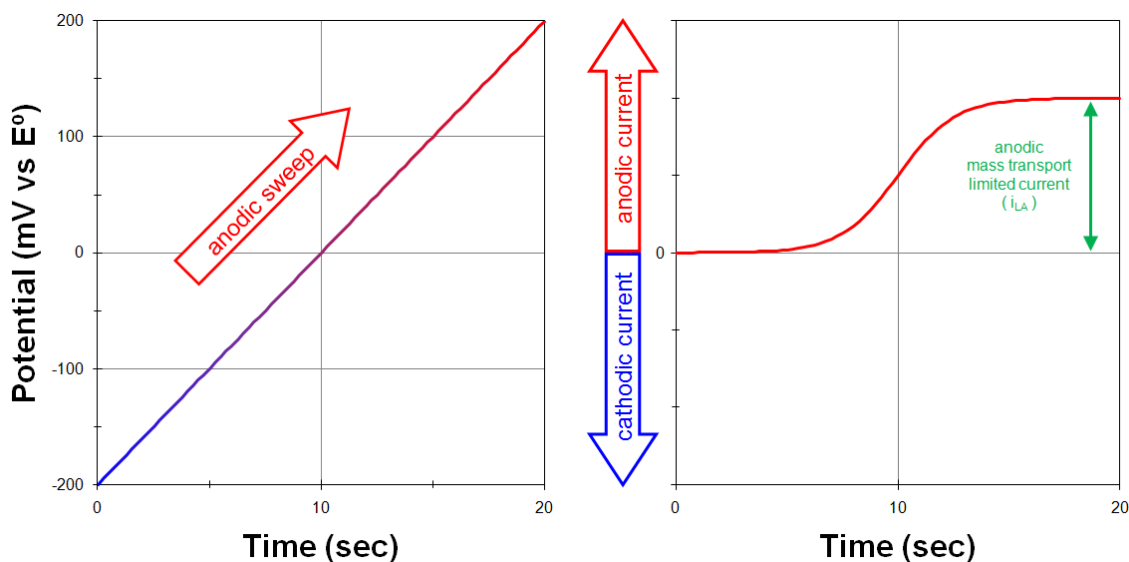


Figure 10-2. Response to a Potential Sweep (Anodic) from a Solution Initially Containing only the Reduced Form (R) with no Oxidized Form (O)

10.3.1 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 10-3, left), it is more common to plot current vs. potential (as shown in Figure 10-3, 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 not 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) potential 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 10-4, left) illustrates this convention, which is sometimes called the “North American” or “Texas” convention.

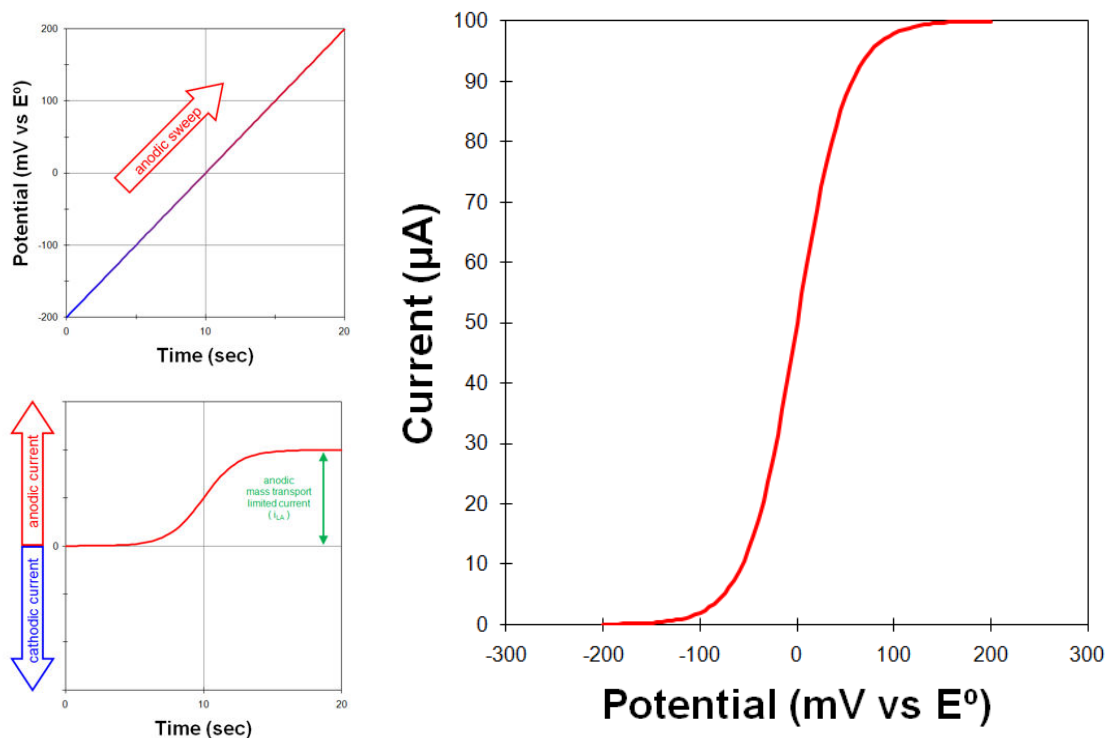


Figure 10-3. A Voltammogram is a Plot of Current versus Potential

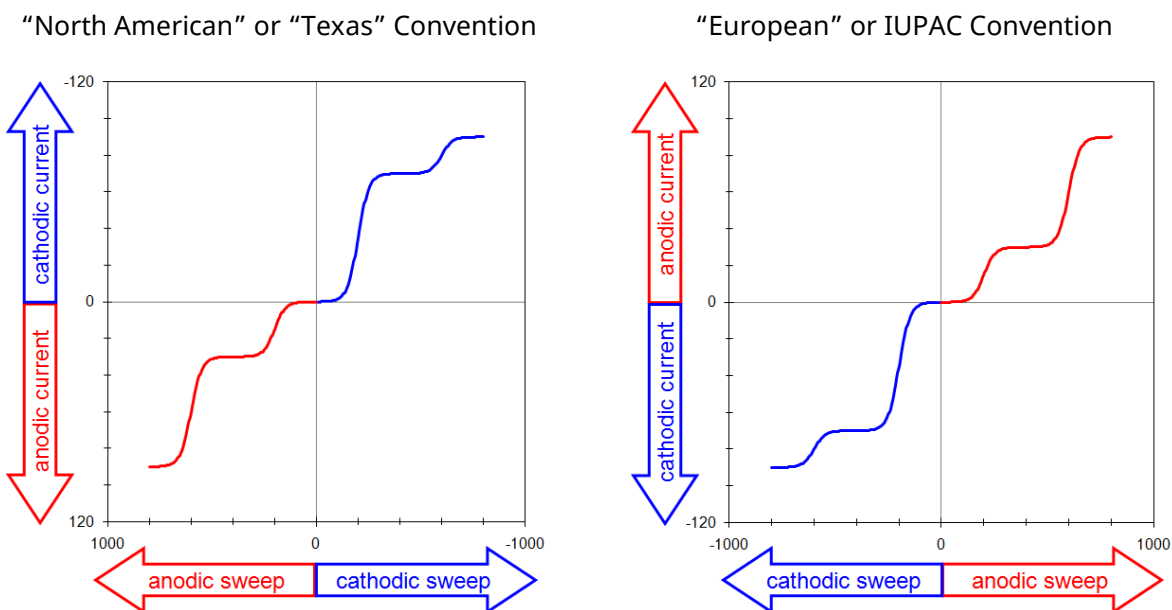


Figure 10-4. Two Popular Voltammogram Plotting Conventions

The same data may be plotted using the “European” or IUPAC convention (see Figure 10-4, 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/IUPAC 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/IUPAC 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.

10.3.2 Measuring Limiting Currents

The theoretical voltammetric response from a rotating electrode is a symmetric sigmoid-shaped wave (like the ideal voltammograms shown in Figure 10-3 and Figure 10-4). 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 10-5). 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.

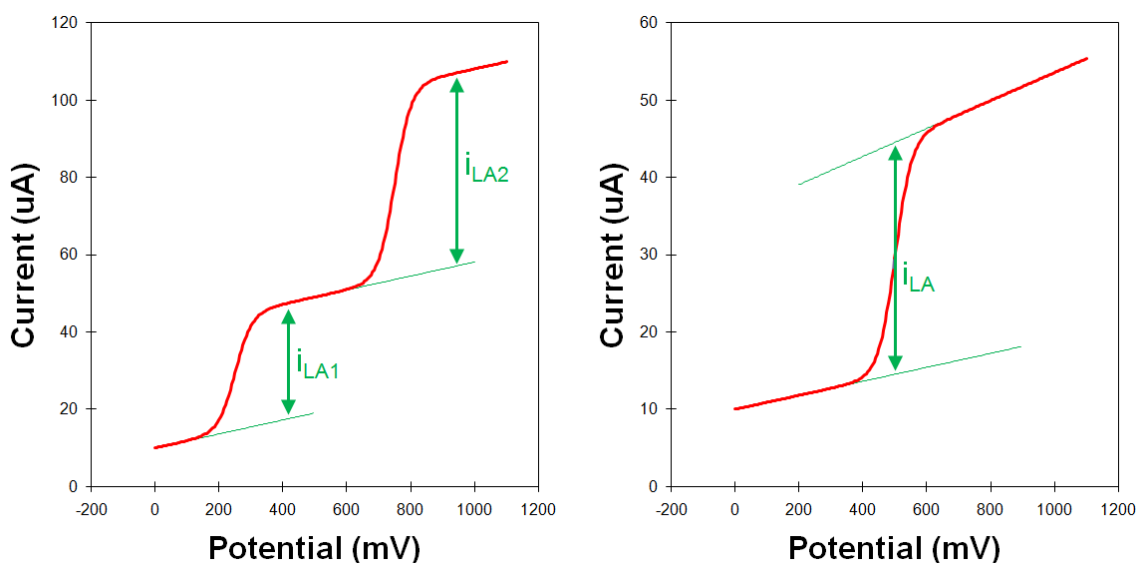


Figure 10-5. Sloping Backgrounds in Voltammograms

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

10-5, 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 10-5, 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 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 10-5, right).

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 10-6). 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.

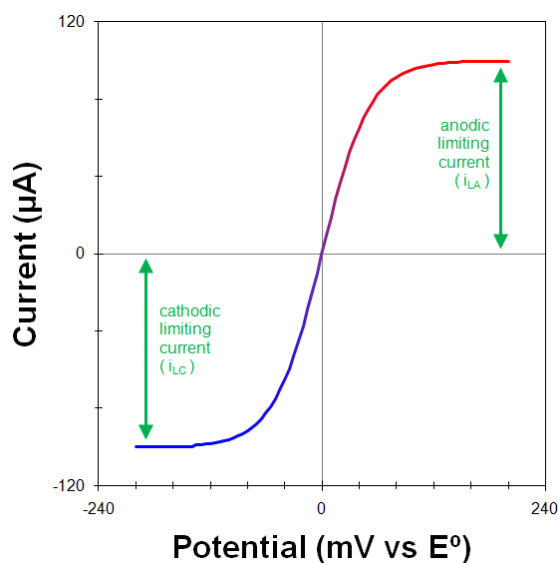


Figure 10-6. Voltammogram for a Solution Containing Both O and R

10.4 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 translated^[9] from Russian to English, and the RDE became more widely known^[11] to Western researchers. In the early 1960's, Stanley Bruckenstein^[10] at the University of Minnesota (and his students Dennis Johnson and Duane Napp) and Ronnie Bell^[12] 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 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):

$$\delta_H = 3.6(\nu/\omega)^{1/2}$$

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 in terms of the diffusion coefficient (D_F) of the molecule or ion as follows:

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

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]

$$i_{LC} = 0.620nFAD^{2/3}\nu^{-1/6}C_O\omega^{1/2}$$

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

$$i_{LA} = 0.620nFAD^{2/3}\nu^{-1/6}C_R\omega^{1/2}$$

where the concentration term (C_R) is for the reduced form rather than the oxidized form.

10.4.1 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 10-7, 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 10-7, right) increases linearly with the square root of the rotation rate (with a slope of $0.620nFAD^{2/3}\nu^{-1/6}C$) 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.

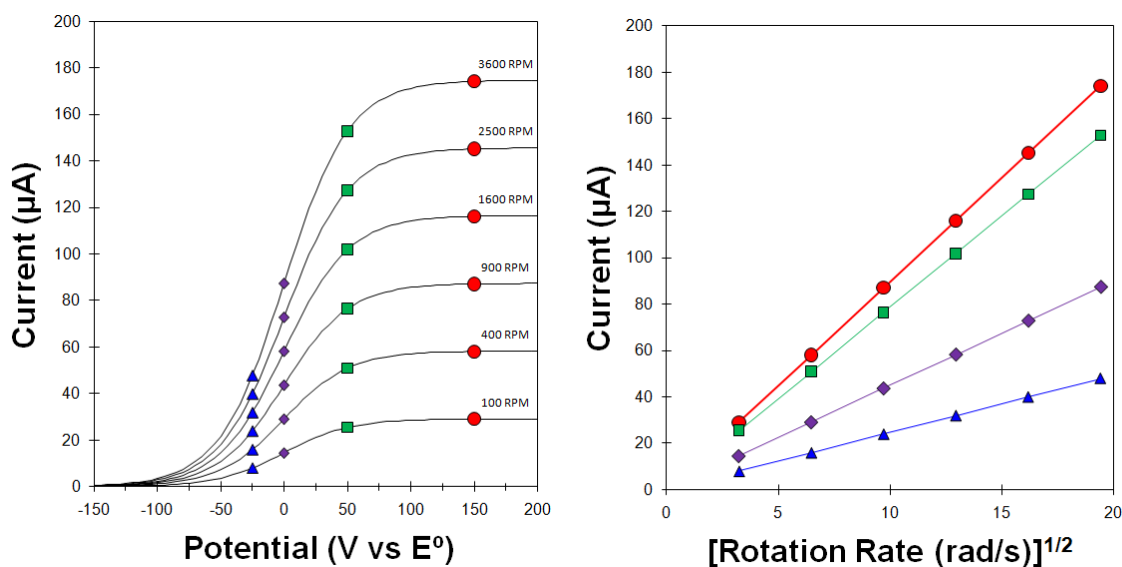


Figure 10-7. Levich Study - Voltammograms at Various Rotation Rates

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 10-7, right). But, 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.

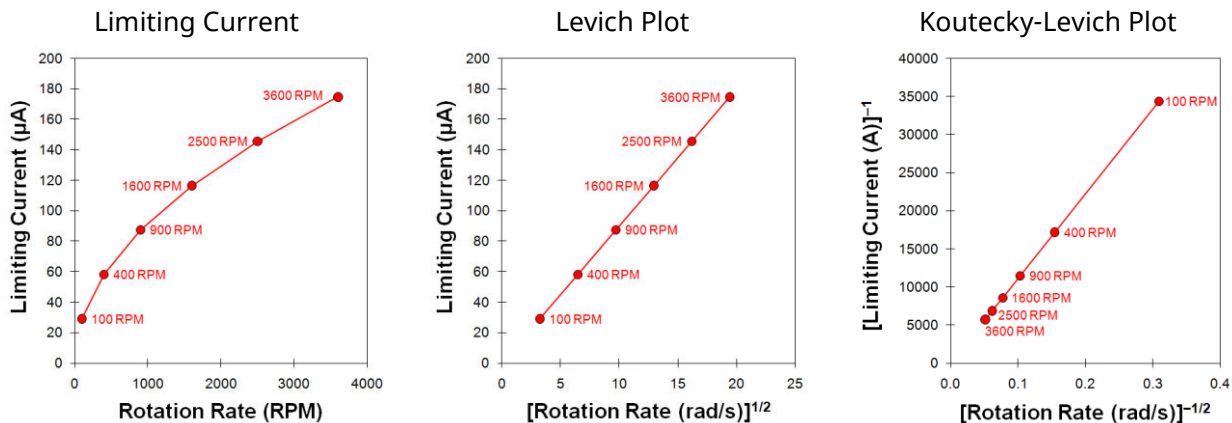


Figure 10-8. Levich Study – Limiting Current versus Rotation Rate

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 10-8, 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:

$$\frac{1}{i_L} = \left(\frac{1}{0.620nFAD^{2/3}\nu^{-1/6}C} \right) \omega^{-1/2}$$

A plot of reciprocal current versus the reciprocal square root of the angular rotation rate (see Figure 10-8, right) is called a Koutecky-Levich^[2,14] plot. 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.

10.4.2 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 analysis^[14] 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 10-7) with a set of voltammograms with sluggish kinetics (see Figure 10-9), 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.

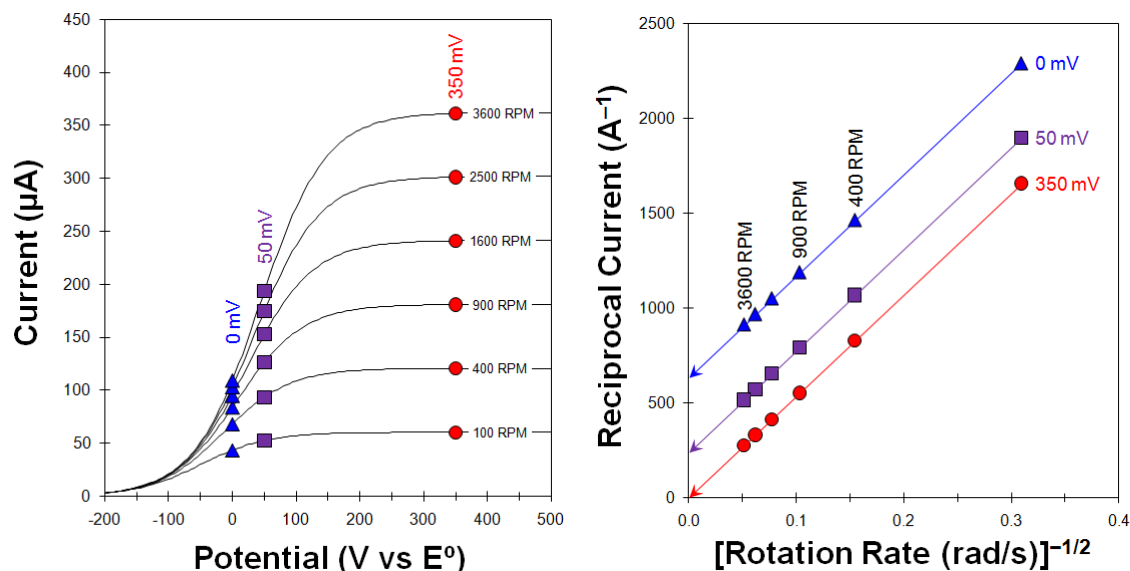


Figure 10-9. Koutecky-Levich Study – Voltammograms with Sluggish Kinetics

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 10-9, 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 10-9, right) when these sampled currents are plotted on a Koutecky-Levich Plot.

For the set of currents sampled on the limiting current plateau (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 10-8, 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 10-9, 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:

$$\frac{1}{i} = \frac{1}{i_k} + \left(\frac{1}{0.620nFAD^{2/3}\nu^{-1/6}C} \right) \omega^{-1/2}$$

Plotting the reciprocal current ($1/i$) against the reciprocal angular rotation rate ($\omega^{-1/2}$) yields a straight line with an intercept equal to the reciprocal kinetic current (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 that pertain to different electrochemical processes, can be found in the literature.^[14]

10.5 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 RRDE^[21-26] 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 that characterize a given ring-disk geometry are the collection efficiency^[23] and the transit time. The collection efficiency is the fraction of the material from the disk that 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.

10.5.1 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:

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

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:

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

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

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

10.5.2 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 (~10 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 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 (~50 mV/s) towards more negative potentials, and a cathodic current is observed that 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 10-10). 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.

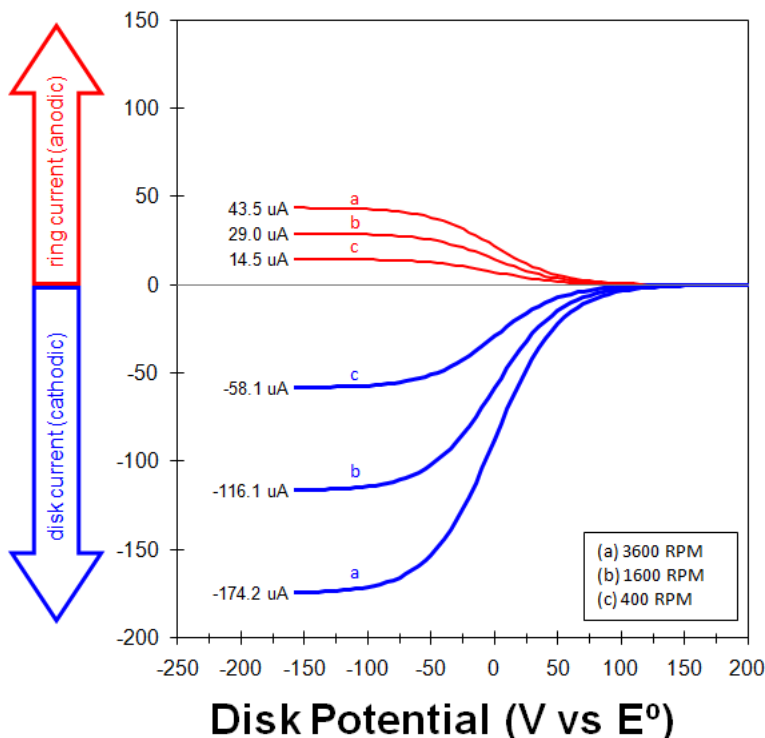


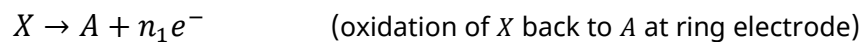
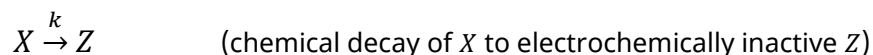
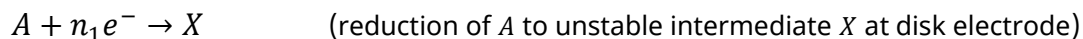
Figure 10-10. Rotating Ring-Disk Voltammograms at Various Rotation Rates

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 with opposite mathematical signs (anodic and cathodic), the collection efficiency is always expressed as a positive number.

$$N_{empirical} = -i_{limiting,ring}/i_{limiting,disk}$$

10.5.3 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 much X is being “collected” at the ring. There is also a competing chemical reaction that is capable of eliminating X before it has a chance to travel 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 ($N_{apparent}$):

$$N_{apparent} = -i_{ring}/i_{disk}$$

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 $N_{apparent} \approx N_{empirical}$, 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 rotate 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.

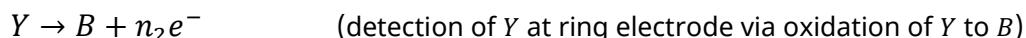
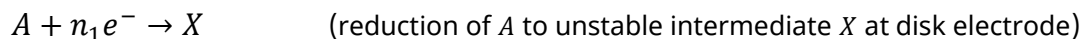
By recording a series of rotating ring-disk voltammograms at different rotation rates and analyzing the results, it is possible to estimate the rate constant (k) associated with the intermediate chemical decay pathway. Various relationships have been proposed for this kind of analysis,^[2] and one of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

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

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.

10.5.4 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 that 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:

$$\theta_{XY} = \left(\frac{1}{N_{\text{empirical}}} \right) \left(\frac{n_1}{n_2} \right) \left| \frac{i_{\text{ring}}}{i_{\text{disk}}} \right|$$

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 that 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]

10.6 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 researchers^[44-46] 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 turbulent^[31] 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, rotating 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 in terms of the concentration (C) and diffusion coefficient (D) of the molecule or ion being studied, Faraday's constant ($F = 96485$ C/mol), 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):

$$i_L = 0.0487nFAd_{cyl}^{0.4}D^{0.644}\nu^{-0.344}C\omega^{0.7}$$

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.^[29-32,44-69]

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11 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 that 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 electroanalytical 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.