



Setup Guide For Squidstat Potentiostats as a Bipotentiostat for Rotating Ring-Disk Electrode (RRDE) Experiments

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January 2024

Preface

This application note explains the general use of two Squidstat potentiostats in a bipotentiostat configuration.

- Cyclic voltammetry is collected at the disk while collecting chronoamperometry data at the ring.
- Rotator speed is set manually.



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 - **Final Standard Configuration** – Page 12
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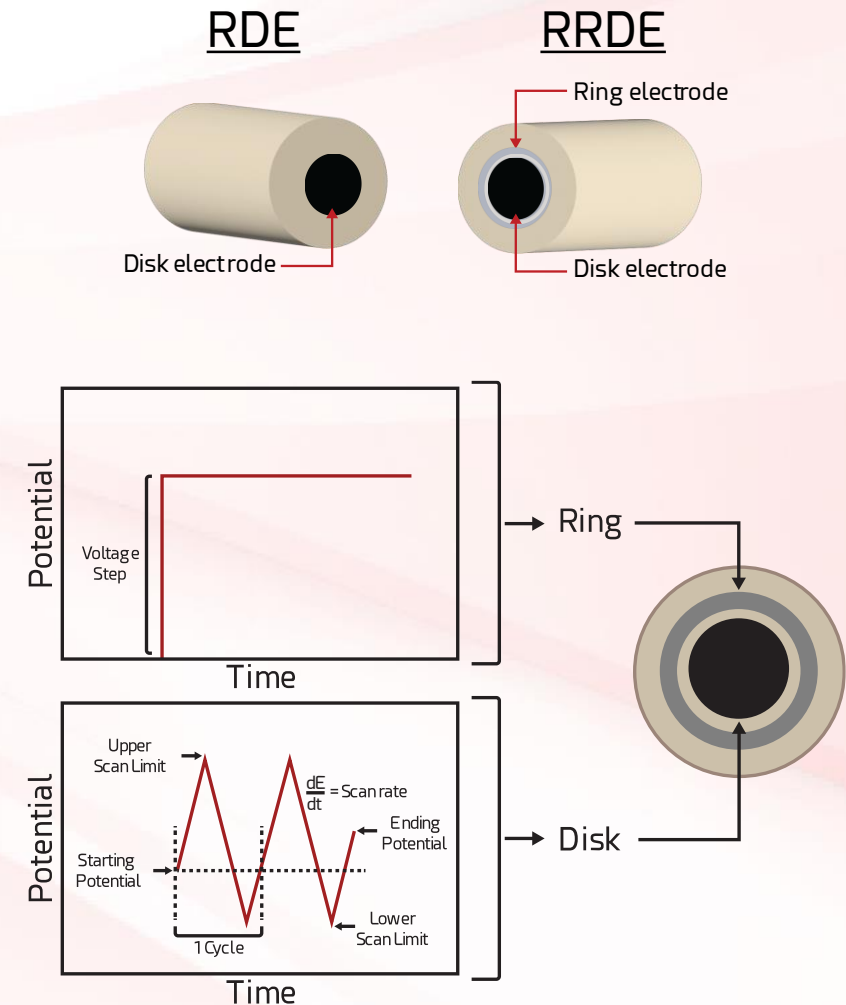
Abbreviations

- Counter Electrode – **CE**
- Counter Sense Electrode – **CSE**
- Reference Electrode – **RE**
- Working Electrode – **WE**
- Working Sense Electrode – **WSE**
- Open Circuit Potential – **OCP**
- Rotating Ring-Disk Electrode – **RRDE**
- Squidstat User Interface – **SUI**

Step By Step Guide using Two Squidstat Instruments as a Bipotentiostat with Prebuilt RRDE Experiments

General Step-By-Step Guide

1. Set both instruments to float mode
2. Connect all four counter electrode leads
3. Connect both reference electrode leads
4. Connect two working electrode leads (**Potentiostat A**) for disk, connect two working electrode leads (**Potentiostat B**) for ring
5. Final Standard Configuration
6. Open RRDE pre-built experiment in SUI
7. Select **Potentiostat A** for disk and set potential limits for cyclic voltammetry experiment at disk
8. Select **Potentiostat B** for ring and set constant potential value for ring, start experiment
9. Set save location and begin data collection

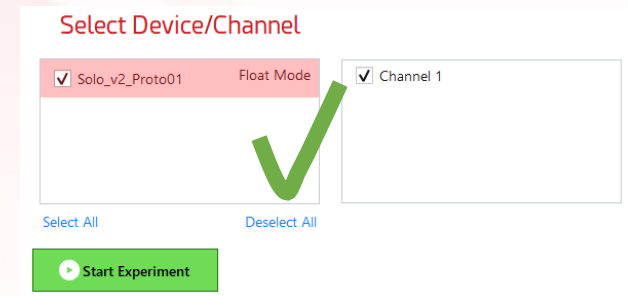
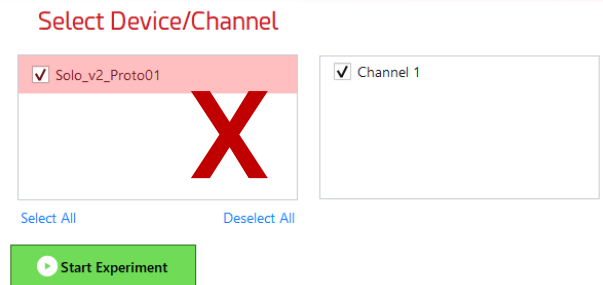


1. Set both instruments to float mode

- On the back of each instrument is a toggle switch to set the hardware to “Float” or “Ground” modes
- Move the switch to the “Float” mode position on **both** instruments

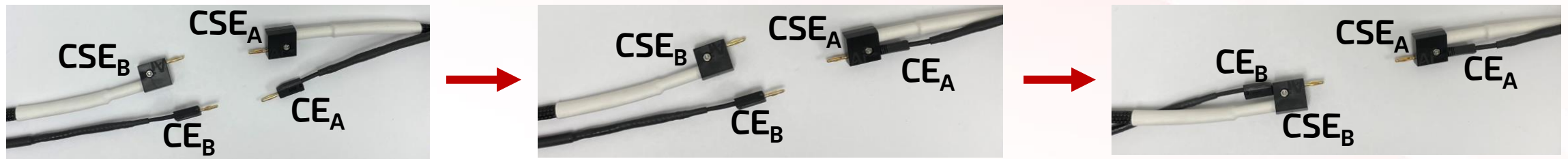
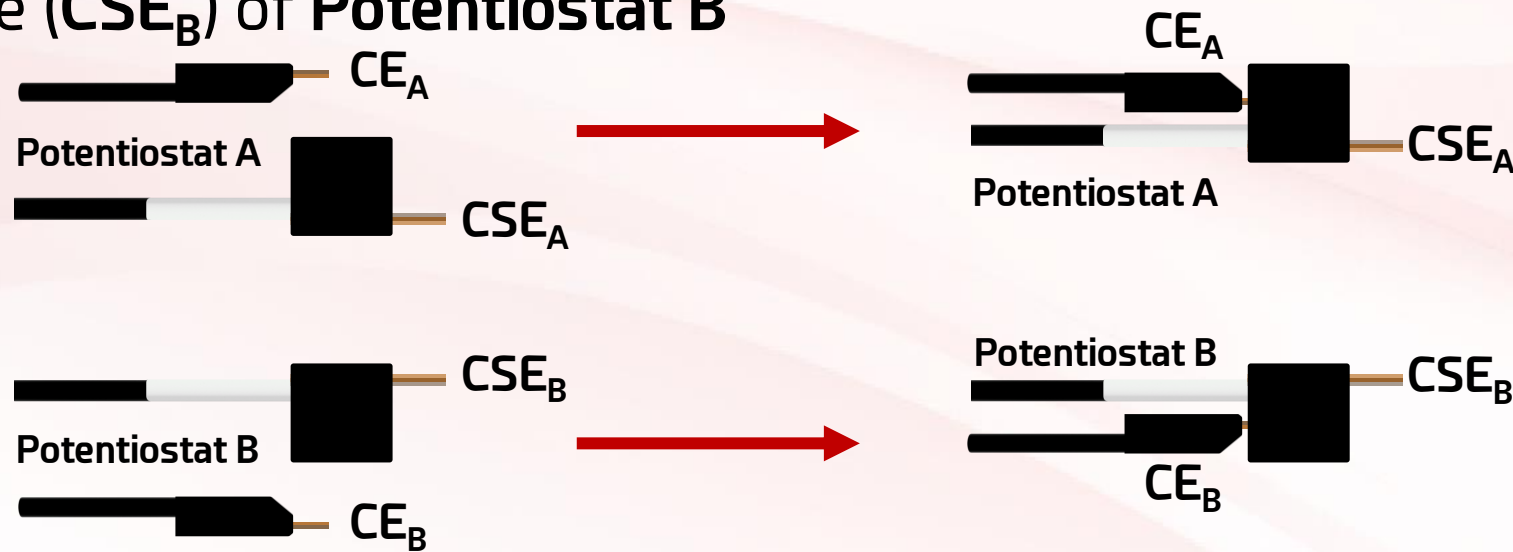


- The SUI software will show that the instrument has indeed been switched into float mode



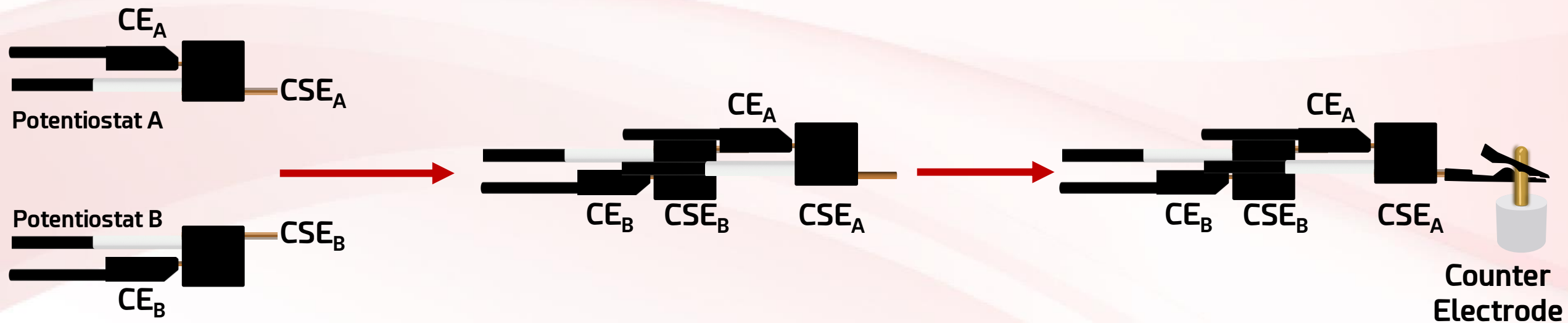
2a. Connections – Counter Electrodes

- Insert the counter electrode (CE_A) of **Potentiostat A** into the back of the counter sense electrode (CSE_A) of **Potentiostat A**
- Insert the counter electrode (CE_B) of **Potentiostat B** into the back of the counter sense electrode (CSE_B) of **Potentiostat B**



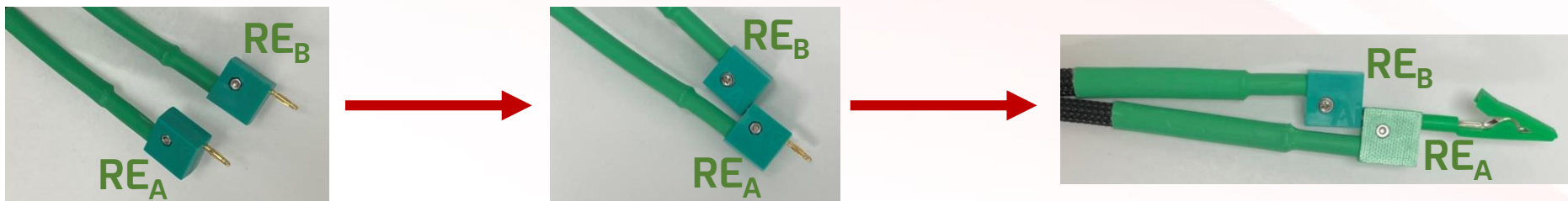
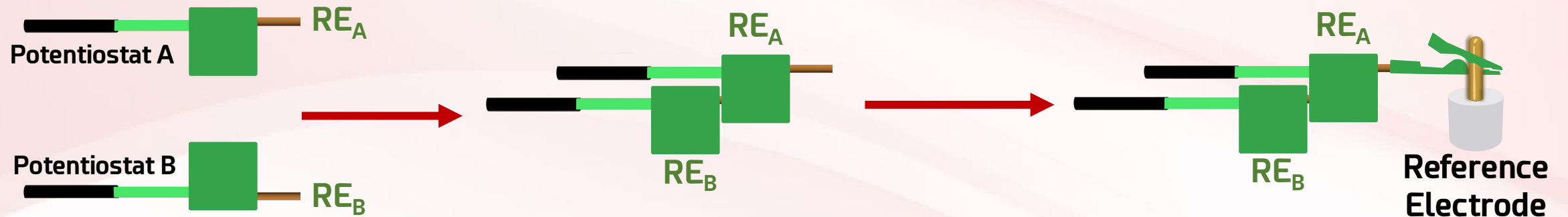
2b. Connections – Counter Electrodes

- Insert the counter sense electrode (CSE_B) of **Potentiostat B** into the back of CE_A
- Attach the four electrodes via CSE_A to the counter electrode



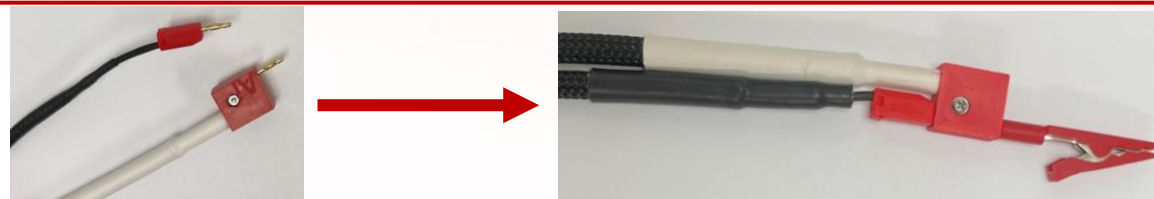
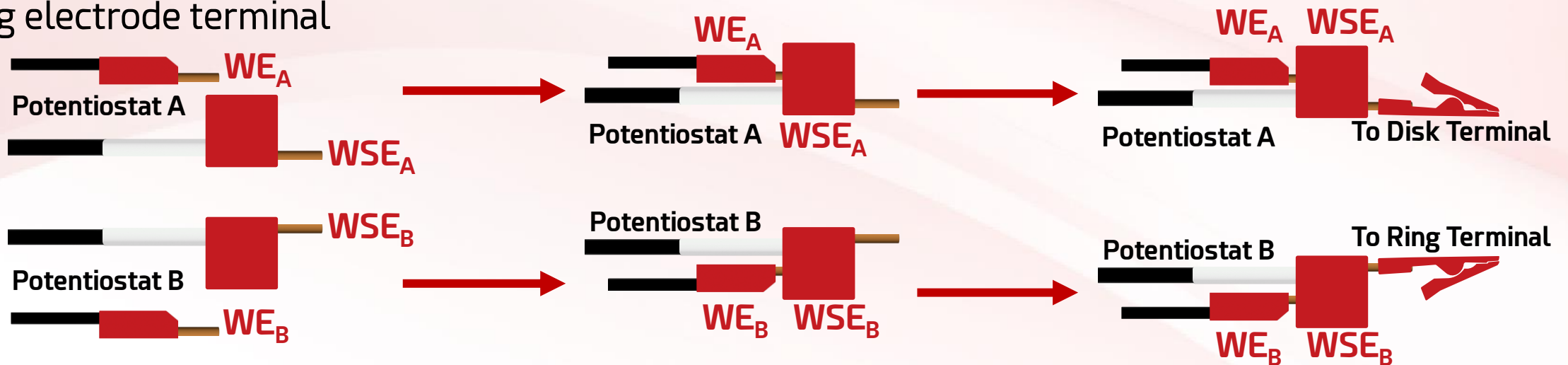
3. Connections – Reference Electrodes

- Insert the reference electrode (RE_B) of **Potentiostat B** into the back of the reference electrode (RE_A) of **Potentiostat A**
- The reference electrode (RE_A) of **Potentiostat A** should terminate with a clip to attach to the shared reference electrode



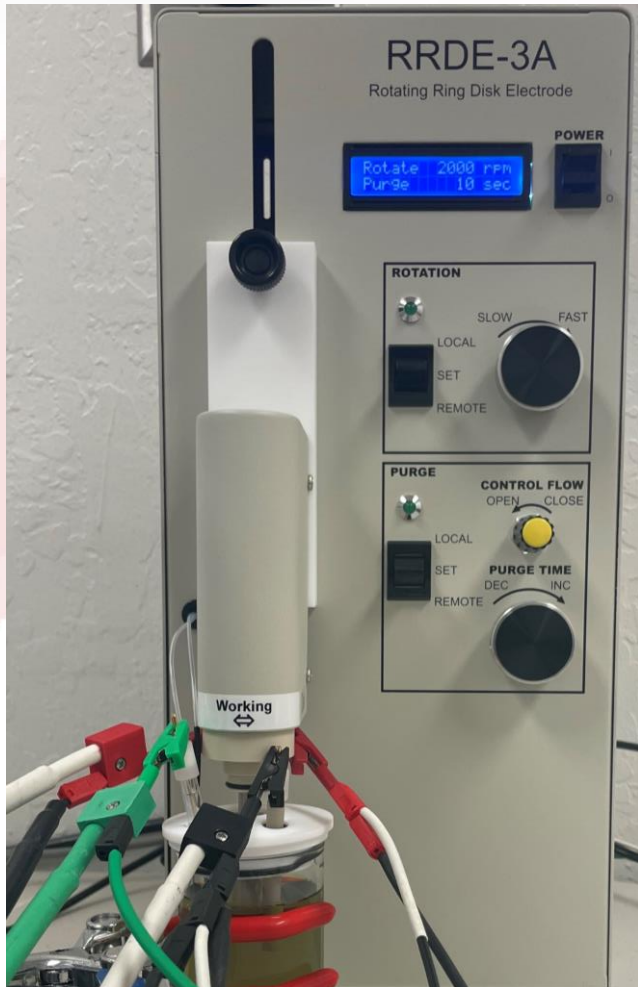
4. Connections – Working Electrodes Standard Configuration

- Insert the working electrode (WE_A) of **Potentiostat A** into the back of WSE_A
- Insert the working electrode (WE_B) of **Potentiostat B** into the back of WSE_B
- The working sense electrode (WSE_A) of **Potentiostat A** should terminate with a clip to attach to the disk electrode terminal
- The working sense electrode (WSE_B) of **Potentiostat B** should terminate with a clip to attach to the ring electrode terminal



5. Final Standard Configuration

Complete Assembly



Disk Connection



Ring Connection



6. Preconfigured RRDE Experiment in SUI

Squidstat User Interface v2.11.13.2023

Run an Experiment | View Data | Multichannel Control | Build an Experiment | Manual Control | More Options

Categories

View All

Search

- Chronopotentiometry
- Constant Power
- Constant Resistance
- Cyclic Voltammetry
- Differential Pulse Voltammetry
- Galvanostatic EIS
- Potentiostatic EIS
- Galv. Intermittent Titration Technique
- Linear Sweep Voltammetry
- Multi-frequency Mott-Schottky
- Normal Pulse Voltammetry
- Open Circuit Potential
- Pot. Intermittent Titration Technique
- Rotating Ring Disk Electrode**
- Single-frequency Mott-Schottky

Rotating Ring Disk Electrode

This experiment runs the rotating ring disk electrode (RRDE) experiment. Two Squidstat potentiostats are required to run this experiment. A user can designate any Squidstat and its channel as either the **Disk Instrument** and **Disk Channel**, or the **Ring Instrument** and **Ring Channel**.

The potential of the disk electrode is swept back and forth between the **upper scan limit** and the **lower scan limit** at a constant **scan rate (dE/dt)** for a specified number of cycles. A user can select to scan to the **lower scan limit** or the **upper scan limit** first. The cycling scheme is as follows: **Starting potential** → [Scan limit 1 → Scan limit 2] n → **Ending potential**, where "n" is the number of cycles. The ring electrode is at a fixed **potential** with respect to the **reference** or **open circuit**.

7. Select Potentiostat A for the Disk

Parameters

Disk Parameters

Disk Instrument: Plus1894 ← Select **Potentiostat A** to be used at the Disk

Disk Channel: 1

Cycles: 1

Start a new data file for each cycle? Yes No ← This will produce a new .csv file for every cycle

Quiet time: 5 s ← Start of quiet time parameters
(time spent at the starting potential)

Quiet time sampling interval: 1 s

Starting potential: 0.6 V ← Start of cyclic voltammetry parameters
with respect to: reference

Potential Limit 1: 0.6 V ← Set potential vs reference or OCP
with respect to: reference

Potential Limit 2: -0.6 V

with respect to: reference

Ending potential: 0.6 V

with respect to: reference

Scan rate (dE/dt): 50 mV/s ← Set scan rate

Sample at interval of: 1 mV ← Set sampling rate

Disk Current Ranging

Autorange

Maximum current expected: 1 mA

- Ensure "Autorange" is **not** selected
- Set the current range to the maximum value expected for experiment

8. Select Potentiostat B for the Ring, Start Experiment

Ring Parameters

Ring Instrument: Plus1162

Ring Channel: 1

Potential: 1.0 V

with respect to: reference

Ring Current Ranging

Autorange

Maximum current expected: 10 mA

Start Experiment

Select **Potentiostat B** to be used at the Ring

Set potential (V) for the ring to be held at

Do not use "Autorange"
Set to maximum current for experiment

Click "Start Experiment"

- A pop-up window will appear
- Physical electrode parameters can be set
- Reference electrode type can be set
- Experimental notes such as which instrument is connected to which electrode can be easily saved
- Clicking "Apply" will save all information on this window to the experimental log and proceed
- Clicking "Do not apply, Skip" will not save any info on this window and proceed

Cell Parameters | IR Compensation | Global Limits

Cell Parameters

These parameters are automatically used to calculate values from raw data during an experiment, such as the current density from the current using the working electrode area.

General Physical Parameters

Working Electrode Area: cm²

Working Electrode Mass: g

Working Electrode Volume: cm³

Battery Physical Parameters

Battery Capacity (mAh):

Reference Electrode

Common Reference Electrode: Ag/AgCl in saturated KCl

Other Reference Electrode:

Working Electrode vs. NHE (V): 0.1976

Apply All Settings to RRDE

Notes

RRDE Experiment
Plus1764 on Disk
Solo2102 on Ring

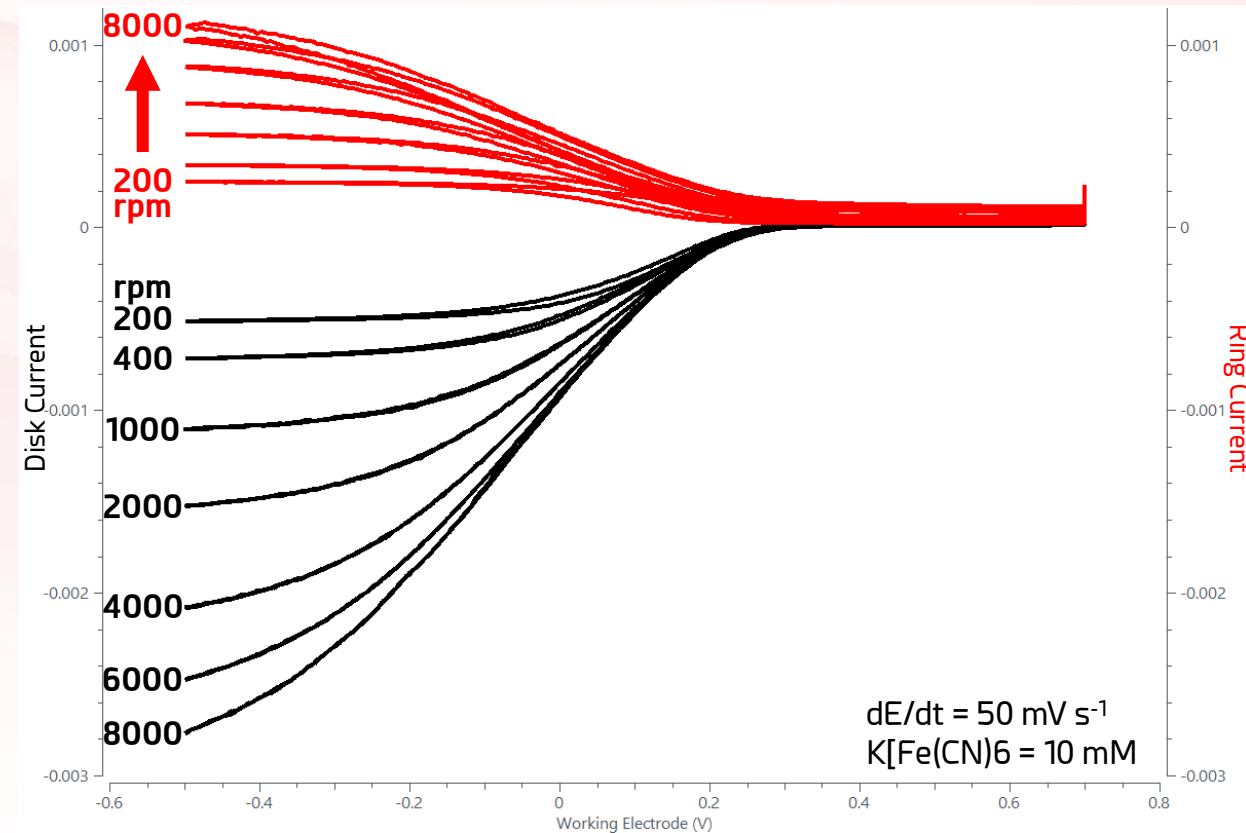
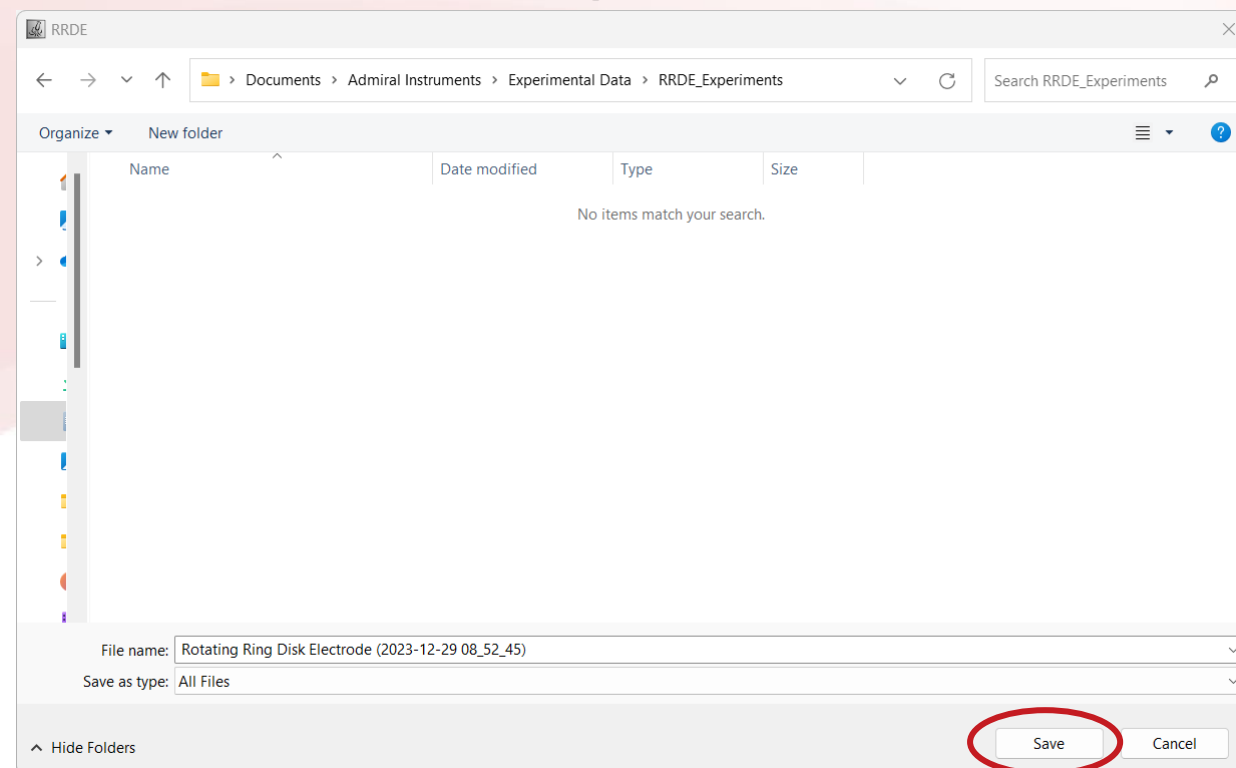
Save information for next experiment.

Apply | **Do not apply, Skip** | **Cancel Experiment** | **Clear All Inputs**

9. Set Save Location and Begin Data Collection

- A final pop-up window will appear.
- Set the file save location for a new experiment folder.
 - Default location is within the Admiral Instruments folder under Experimental Data
- New folder name will include experiment type and time stamp to avoid overwriting previously collected data

- Upon starting data collection, the SUI will switch to the "View Data" tab
- Experimental data can be viewed in real time for both the ring and the disk.



Performance Note

- Bipotentiostat mode was tested up to a current response at the disk of 5 mA (40 mA cm⁻²).
- Collection efficiencies matched or exceeded calculated theoretical values for the RRDE electrode in use from 100 to 6000 rpm.
- If unexpected behavior is observed above our benchmark values, please contact admiral instruments at contact@admiralinstruments.com

Summary

- This application note provided a step-by-step guide to operating two Squidstat potentiostats in a bipotentiostat configuration for RRDE applications.
- Preconfigured RRDE experiments were employed via the Squidstat User Interface (SUI)

Additional Resources for RRDE

- [RRDE Technical Application Note](#) – Admiral Instruments detailed overview of the fundamentals of RRDE experiments.

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