Admiral Instruments
Test the potential™

Squidstat Operating Manual

Updated Jan 2025

Admiralinstruments.com

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1 Support Information and Warranty Statement

1.1 Customer Support

If you have any questions or require support with your Squidstat software or hardware, we're here to help! Please reach out to Admiral Instruments using the contact information below:

Email: support@admiralinstruments.com

Phone: +1 (480) 256-8706

Address: 4666 South Ash Avenue
Tempe, Arizona 85282
USA

Calls are answered Monday through Friday from 9:00 A.M. to 5:00 P.M. GMT/UTC-07:00. Our customer service team will respond to all email and phone inquiries within one business day. Technical support services are always provided free of charge.

To help us resolve your issue as efficiently as possible, please include the following details in your initial message:

- A detailed description of the problem, including the steps leading up to the issue and any troubleshooting actions you've already tried.
- The raw data (.csv) files and corresponding Experiment Settings (.txt) files related to the issue.
- The log files generated during the timeframe of the issue (the log files are located in the file directory /Documents/Admiral Instruments/Logs).
- A picture of your test setup along with a description of the cell, if possible.
- The serial number of your device, found on the rear panel or in the Devices menu of the Squidstat software when the device is connected.
- The operating system (Windows, Mac, or Linux) and software version you're using, which is visible at the top of the Squidstat software and under "Release Notes" in the More Options tab.

1.2 Recalibration

Your Squidstat ships fully calibrated and ready to use – however, all electronic devices change gradually with age. To best support you, Admiral Instruments gives you the flexibility to evaluate the DC accuracy of your Squidstat's calibration without shipping the Squidstat back to us. The application note "Understanding and Validating Potentiostat DC Accuracy," available on our website, provides a step-by-step guide to help you determine if your Squidstat needs recalibration. After following the guide, you may choose the next steps that correspond to your time and budget requirements.

Two of our main goals are to give you control over performance and to enhance your productivity. If your Squidstat's accuracy has changed with age, Admiral Instruments offers two types of recalibration services: 1) offset correction using the Squidstat Support Utility, and 2) factory recalibration. Offset correction is a simple procedure that corrects measurement offsets due to age-related component drift. This type of DC correction only takes a few minutes with the Squidstat Support Utility software, and you can get back to using your Squidstat right away. Please refer to the Sections 13.4.1 Current Tare and 13.4.2 DC Voltage Tare in this operating manual for a step-by-step guide on performing offset corrections. Please note that if the measured DC offset exceeds 3% of the current or voltage range, it may not be fully corrected by the tare functions in the Squidstat Support Utility. If the offset correction tare fails, please contact us for further assistance. Offset correction may, but does not guarantee, improvement in applied setpoint performance. If your Squidstat shows setpoint performance anomalies that persist after offset correction, you may contact us for a quote regarding factory recalibration.

If you choose factory recalibration, your Squidstat will be shipped to Admiral Instruments for a thorough evaluation, including checks for hardware damage. Factory recalibration offers a more comprehensive service than offset correction, as it includes full DC recalibration (both measurement and setpoint offset correction) and AC recalibration. If you require maximum precision, we recommend choosing factory recalibration over offset correction, as it ensures your Squidstat is fully recharacterized and performing to the highest standards.

1.3 Warranty and Disclaimers

Admiral Instruments warrants that Squidstat instruments are free from defects in material and workmanship under normal use for a period of two years from the date of delivery. During this warranty period, Admiral Instruments will repair or replace, at no charge, any Squidstat potentiostat, Squidstat Cycler, Admiral Instruments brand power supply, or part that proves defective due to improper material or workmanship under normal use and maintenance.

Admiral Instruments will bear the cost of shipping a defective device during the warranty period, except in cases where the customer has failed to disclose issues that would reasonably have been apparent (e.g., liquid damage or unauthorized modifications), in which case the customer will be responsible for shipping costs if they wish to have the unit returned.

In addition, for any repairs performed by Admiral Instruments or an authorized distributor, a 90-day warranty will be provided for the repair. This warranty applies even if the instrument was out of warranty when the repairs were initiated.

Exclusions

The warranty policy does not apply to the following:

- Squidstat channel cables, cable clips, multi-purpose battery test fixtures, Squidstat Inkboards, or consumables such as electrodes.
- Damage resulting from improper installation, operation or storage.
- Damage incurred during transportation unless shipped by Admiral Instruments or an authorized distributor.
- Damage resulting from misuse, including failure to follow the user manual and operating instructions.
- Damage resulting from non-observance of the safety and operational guidelines as outlined in the manual.

- Environmental damage, including liquid exposure, exposure to extreme temperatures, or chemical exposure.
- Operation outside the specified environmental or operational conditions (e.g., humidity, atmosphere).
- Unauthorized repairs or modifications not performed by Admiral Instruments or an authorized distributor.
- Unauthorized opening of the instrument.
- Use of a power supply other than the one provided by Admiral Instruments.
- Use with incompatible software or firmware.
- Power supplies not manufactured by Admiral Instruments.

Disclaimers and Limitations of Liability

1. **Disclaimer of Implied Warranties**: We do not provide any warranties beyond what's explicitly stated in this warranty policy. This means we can't guarantee the product will meet every specific need or expectation, unless we've mentioned it explicitly in writing.
2. **Limitation of Liability**: Admiral Instruments is not responsible for any indirect damages or losses that happen because of issues with the product. This includes things like loss of business, lost profits, or any other costs that aren't directly related to fixing or replacing the product. Even if we were informed of the potential for these losses, we cannot be held responsible.
3. **Exclusive Remedy**: If the product has a defect covered under warranty, we will either repair or replace it for you, at no cost. This warranty covers the product itself, and our responsibility is limited to the original purchase price.
4. **Conditions for Coverage**: This warranty is void if the product is operated outside its intended use, is subject to improper installation or maintenance, or if it is altered, opened, or modified in any way. Warranty coverage is determined at the sole discretion of Admiral Instruments. If, upon examination, the product is found to be outside of warranty, Admiral Instruments reserves the right to decline coverage, even if prior approval was given before the product was returned.

High Power Disclaimer

Admiral Instruments is not liable for any damages or injuries resulting from the use of high-power equipment, including but not limited to Squidstat instruments and their external power supplies. Customers must ensure that all equipment used complies with the specified ratings and standards provided by Admiral Instruments. Failure to use appropriate equipment may result in damage to the instrument, injury, or other hazards, and will void the warranty. Admiral Instruments strongly recommends consulting with qualified personnel for the installation and operation of high-power equipment.

Software Compatibility Disclaimer

Admiral Instruments tests hardware and software compatibility and interoperability with PCs running Windows 7 and above, macOS 10.15 and above, and Ubuntu 20.04 and above. However, Squidstat software is not guaranteed to work with all customer-supplied computers.

2 Safety

The following safety warnings pertain to all Admiral Instruments hardware and should be strictly adhered to at all times. Failure to do so can result in personal injury and/or damage to the hardware.

RISK OF ELECTRICAL SHOCK

To reduce the risk of electrical shock and personal injury, do not remove the cover or access internal hardware components. Admiral Instruments hardware should only be opened by qualified personnel certified by Admiral Instruments. Internal hardware components are not required to be serviced by users under normal operating conditions. If a Squidstat is suspected of requiring servicing or repair, please contact Admiral Instruments at:

Email: support@admiralinstruments.com

Phone: +1 (480) 256-8706.

RISK OF ELECTROSTATIC DISCHARGE

All Squidstat potentiostats and Squidstat Cyclers have electrostatic discharge protection up to 25 kV built in. However, care must still be taken to prevent possible damage from electrostatic discharge. When moving a Squidstat to a new location, first power off the instrument and then disconnect all cables connected to the main body and power supply (if applicable) before making the move.

GENERAL CONSIDERATIONS

Overvoltage and overcurrent protections are built in for all Squidstat potentiostat and Squidstat Cycler hardware. Despite these protections, users must still ensure to disconnect any device under test (DUT) from the instrument before powering it off. Not doing so creates an opportunity to discharge power from the DUT into the unit, which may cause damage. Users should not connect a DUT with voltage ranges beyond that of the compliance voltage for the Squidstat potentiostat or Squidstat Cycler in use.

Powered sense leads should not be connected or disconnected while the instrument is powered on. This includes Squidstat Plus models with serial numbers 1700 and above as well as the Squidstat Penta, Squidstat Decka, and Squidstat Venta.

ENVIRONMENTAL PRECAUTIONS

All Squidstat potentiostats and Squidstat Cyclers are designed for use in standard lab environments. Although direct use in field applications is suitable, the instruments were not specifically designed for use in field conditions. Do not expose the Squidstat or its cabling to any source of heat, liquid, or corrosive chemicals, and do not operate the unit outside the environmental limits below.

Operating Temperature Range	0°C to 40°C
Compatible Atmospheres	Ambient, N_2, Ar
Compatible Ambient Pressure	>50 mbar

SAFETY CERTIFICATIONS

All Squidstats automatically conform to the CE directives for low voltage electrical devices and thus have a CE marking on the rear panel of each instrument.

We also offer a Nationally Recognized Testing Laboratory (NRTL) certification label applied to any individual Squidstat in partnership with CSA Group. Please contact Admiral Instruments if you need to purchase a Squidstat with NRTL certification.

RESTRICTION OF HAZARDOUS SUBSTANCES

All Squidstats potentiostats and Squidstat Cyclers are built using lead-free components and solder following the Restriction of Hazardous Substances Directive.

GROUNDING

The Squidstat Solo, Penta, Decka, Venta, and the power supplies for the Squidstat Plus and Squidstat Cycler are safely grounded to Earth through the protective conductor in the AC power cable (Class I). The Squidstat Plus is grounded through its power supply (Class III), while the Squidstat Cycler is floating with respect to ground. Use only the power supply and power cabling provided by Admiral Instruments, and ensure it is connected to a properly grounded power outlet with a protective earth connection. See Section 8.8 for more information on grounding and the use of the ground/float switch for select models.

3 Unpacking and Parts List

3.1 Unpacking

All necessary hardware and software to operate a Squidstat are supplied by Admiral Instruments. Upon receiving your Squidstat, please check the packing list inside the shipment to ensure all the parts are present in the box. Contact Admiral Instruments immediately if any parts are missing or damaged.

3.2 Parts List for all Squidstat Models

The table below summarizes the contents of all standard Squidstat shipments by model.

Squidstat Model	Plus	Prime	Solo	Penta	Decka	Venta	Cycler*
Main Unit	1	1	1	1	1	1	1
External Power Supply	1	1	0	0	0	0	1
Channel Cables (5-leads per cable unless otherwise stated)	1 set of 3 (1 WE, 1 CE, 1 Sense)	4	1	1 set of 3 (1 WE, 1 CE, 1 Sense)	1 set of 3 (1 WE, 1 CE, 1 Sense)	1 set of 3 (1 WE, 1 CE, 1 Sense)	4
Power Source Cable	1	1	1	1	1	1	1
Potentiostat-to-Power Source Cable	1	1	0	0	0	0	0
USB Cable	1	1	1	1	1	1	1
Alligator Clips	5	20	5	5	5	5	20
Grounding Cable	0	0	0	1	1	1	0

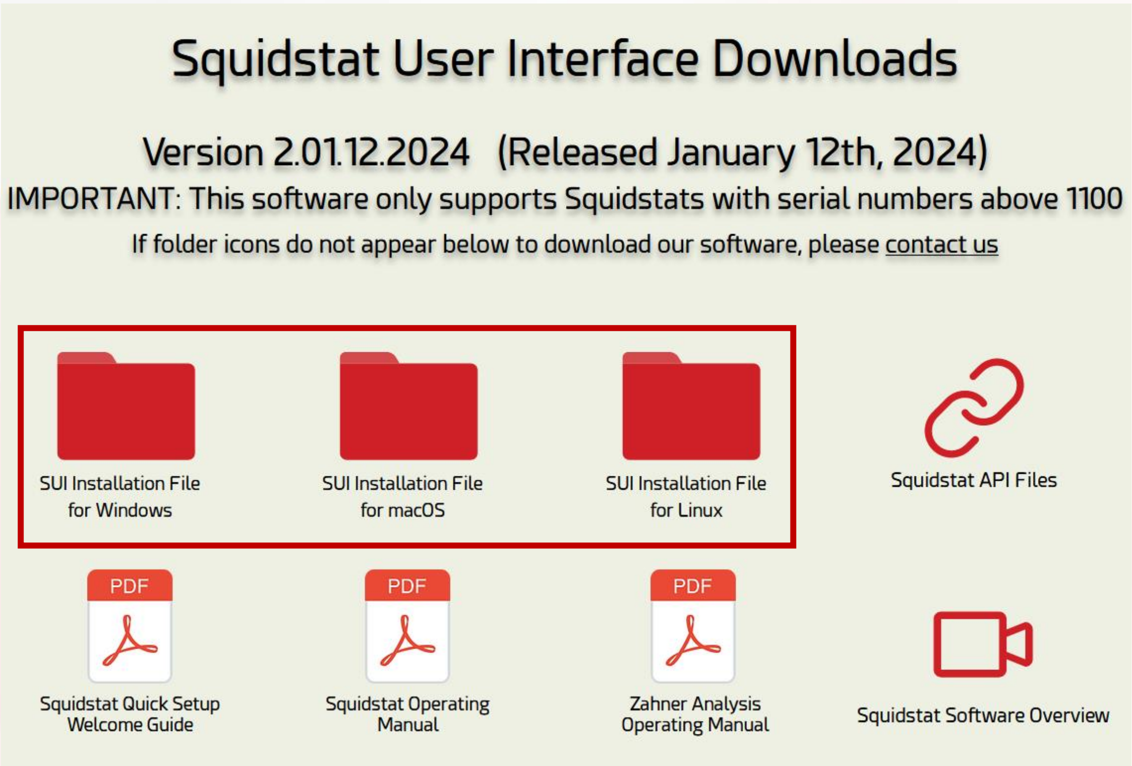
***** *Applies to the Squidstat Cycler Base Model only. Parts list for Customized Squidstat Cyclers will vary per order.*

Instructions for setting up hardware and detailed device specifications for each model can be found in the Hardware User Manual.

4 Installing the Squidstat User Interface (SUI) and Zahner Analysis (optional)

The Squidstat User Interface (SUI) is compatible with PCs running Windows 7 and above, macOS 10.15 and above, and Ubuntu 20.04 and above. Please follow the step-by-step instructions outlined below to install the SUI.

Step 1: Visit www.admiralinstruments.com/software, and scroll down to the "Squidstat User Interface Downloads" section. Click on the SUI installation package folder appropriate for your operating system.



Step 2: A popup will appear to download the Installation Wizard (.exe for Windows, .dmg for Mac, and .run or .tar.gz for Linux/Ubuntu) and an optional .zip file containing example experiment datafiles. Download the installation file, then double click the installer to begin.

Step 3: Follow the prompts from the Setup Wizard to install the Squidstat Drivers and Squidstat User Interface, and if selected, the optional Zahner Analysis software. The default installation directory is `C:\Program Files (x86)\Admiral Instruments\Squidstat` for the SUI and `C:\Program Files\Zahner\ZahnerAnalysis` for the optional Zahner Analysis software.

Step 4: After installation is complete, launch the SUI directly from the Installer or the Desktop shortcut.

Step 5: Turn on the Squidstat and plug the USB connection into your computer. It may take a few minutes for the Squidstat to be recognized by the computer. If the Squidstat is shipped with firmware incompatible with the installed version of the SUI, an automatic firmware update will be initiated, see **Section 6.1**. When the Squidstat is ready to be used, the text "Start working with [Model][serial number]" will appear in the bell icon at the top right of the SUI.

5 Updating the Squidstat User Interface (SUI)

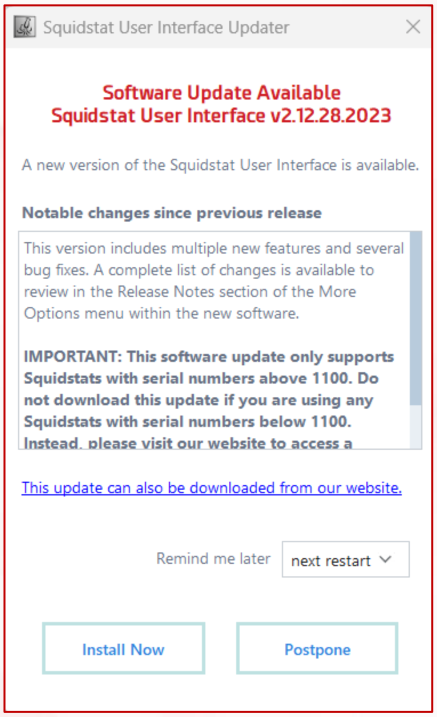
5.1 Manually Downloading and Installing New SUI Versions

The Admiral Instruments team is routinely adding and refining features and content in the SUI. Customers may choose to update the software as often as they wish by manually checking for new versions on the Admiral Instruments website.

Installation of manually downloaded versions of the SUI will be identical to installing the SUI for the first time (**Section 4**). This may be necessary, for example, in cases where a constant internet connection is not accessible for a specific computer running the SUI. In this case, the automated pop-up to install the newest version of the SUI will not be activated. Users will be required to physically transfer the installation files to the computer of interest.

5.2 Automatic Updates of the SUI from Pop-Up Messages

As of SUI version v2.06.15.2023, upon launching the software an automated check for a new version of the SUI on the Admiral Instruments website will occur. If an updated version is found, the user will be prompted to install the new SUI version. Users can either “Install Now” or “Postpone” the installation. The dropdown menu next to “Remind me later” provides four time-dependent reminders to install the new SUI version: next restart, 1 day, 5 days, and next release. Choosing “next release” is not recommended as the updates contain useful software feature additions and firmware upgrades that can improve hardware performance.



The procedure for updating to newer versions of the SUI is the same as installing the SUI for the first time (**Section 4**). During installation, existing instances of the SUI are automatically removed and replaced with the updated version. All contents within the Admiral Instruments folder, located by default in the Documents folder, will not be altered during the installation process, and no user data will be overwritten.

6 Updating Firmware on a Squidstat

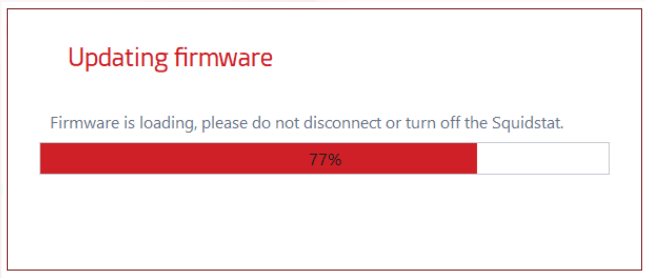
6.1 Automatically Updating the Firmware

Firmware updates are packaged with and are specific to each Squidstat User Interface (SUI) version. These updates are required for the Squidstat to properly communicate with each version of the SUI. Firmware updates will occur automatically when the SUI determines that an update is necessary for your instrument. To facilitate a seamless update process, follow the steps below, repeating steps 2 and 3 for each Squidstat that may require a firmware update.

Step 1: Launch the SUI

Step 2: Connect Squidstat to the computer via a USB cable

Step 3: Power on the Squidstat to initiate the automatic firmware update. During the firmware update, the following popup will be visible. The popup will close when the progress reaches 100%.

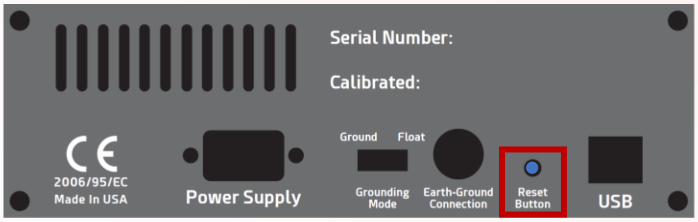


If multiple Squidstats with outdated firmware are connected and powered on before the SUI is launched, firmware updates for each instrument will occur in succession.

6.2 Manually Updating the Firmware

Manually updating the firmware should not be necessary under normal circumstances, however on rare occasions, a Squidstat may need to be reset to facilitate a firmware update. All Squidstat models can be reset except the Squidstat Cycler as it does not have a reset button. Follow the steps below to reset your Squidstat.

1. Power off the Squidstat.
2. Ensure the power supply is plugged in, the USB connection is plugged in, and the Squidstat software is open on the computer.
3. Press and hold the blue reset button located on the back of the instrument (outlined in red in the diagram below), and while continuing to hold down the reset button, press the front power button to turn on Squidstat.



4. If successful, a firmware update will begin automatically in the software.

7 Cabling

All Squidstat potentiostat and Cycler models come with a standard channel cable that is 1 m in length. Extended length cabling options are only available for the Squidstat Cycler Base Model and Customized Squidstat Cycler at two additional lengths, 2 m and 3 m. The Squidstat potentiostat models are unable to support longer cable lengths at the time of publishing, but that is subject to change. If you wish to purchase a longer length cable for the Cycler, please contact sales@admiralinstruments.com for pricing and additional information.

Identification and functional descriptions of all leads for the channel cabling provided with each Squidstat model is provided below.

7.1 Squidstat Prime and Squidstat Solo

Channel cabling for Squidstat Prime, Squidstat Solo, and first generation Squidstat Plus models (serial numbers below 1700) consists of five leads terminating in a 2 mm male/female banana plug. The cabling follows the wire and banana plug color coding scheme shown below.

Leads can either be connected to a cell individually or stacked together by inserting the banana pin of one lead into the hole on the back of the banana pin of another lead. Each lead is described below:

Working Lead (WE): Black Wire/Red Plug. Current carrying lead. Connects to the working electrode of an electrochemical cell.

Counter Lead (CE): Black Wire/Black Plug. Current carrying lead. Connects to the counter electrode of an electrochemical cell. Current flows only between the working and the counter leads.

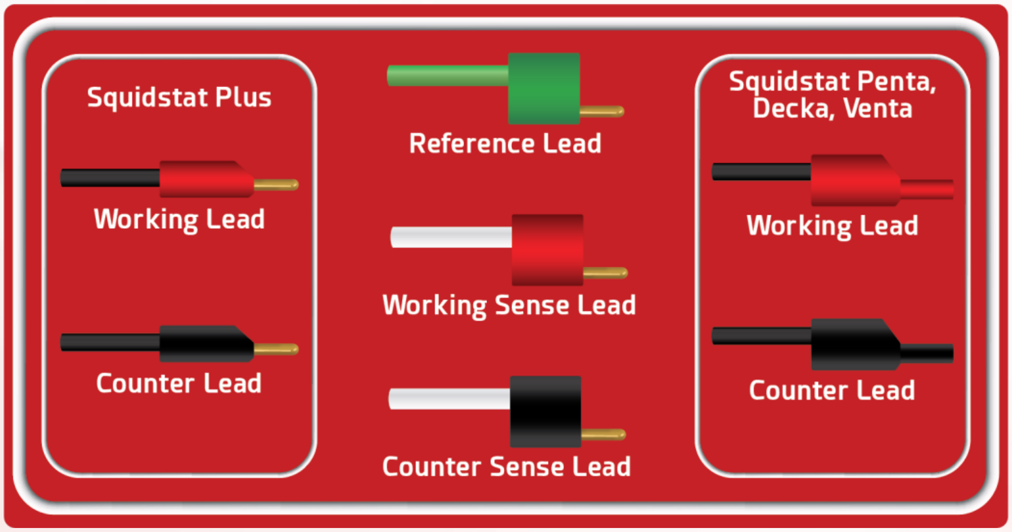
Working Sense Lead (WE-S): White Wire/Red Plug. Voltage sensing lead. Integral part of the feedback loop to control and measure the potential at the working electrode. Can be stacked with the working lead.

Counter Sense Lead (CE-S): White Wire/Black Plug. Voltage sensing lead. Usually connected to the counter electrode of an electrochemical cell. Can be stacked with the counter lead. However, since the counter sense lead is not a part of the feedback loop, it may be placed anywhere a voltage measurement is needed.

Reference Lead (RE): Green Wire/Green Plug. Voltage sensing lead. Serves as a reference point, representing the "zero" potential against which the working and counter electrode potentials are compared. Can be connected to either the reference electrode or the counter electrode of an electrochemical cell.

7.2 Squidstat Plus, Squidstat Penta, Squidstat Decka, and Squidstat Venta

The Squidstat Plus, Penta, Decka, and Venta models include a single-channel cable set with three separate cables that function as one five-lead unit: a working cable, a counter cable, and a sense cable. The sense cable contains three leads terminating in a 2 mm male/female banana plug with integrated sense circuitry. They are conformally coated for protection against ambient moisture and corrosion but are not designed to withstand direct liquid exposure. The working and counter cables for the Squidstat Plus also terminate in 2 mm banana plugs, while those for the Squidstat Penta, Decka, and Venta use 4 mm banana plugs. The cabling follows the color-coded wire and banana plug scheme shown below.



Leads can either be connected to a cell individually or stacked together by inserting the banana pin of one lead into the hole on the back of the banana pin of another lead. Each lead is described below:

Working Lead (WE): Black Wire/Red Plug. Current carrying lead. Connects to the working electrode of an electrochemical cell.

Counter Lead (CE): Black Wire/Black Plug. Current carrying lead. Connects to the counter electrode of an electrochemical cell. Current flows only between the working and the counter leads.

Working Sense Lead (WE-S): White Wire/Red Plug. Voltage sensing lead. An integral part of the feedback loop to control and measure the potential at the working electrode. Can be stacked with the working lead.

Counter Sense Lead (CE-S): White Wire/Black Plug. Voltage sensing lead. Usually connected to the counter electrode of an electrochemical cell. Can be stacked with the counter lead. However, since the counter sense lead is not a part of the feedback loop, it may be placed anywhere a voltage measurement is needed.

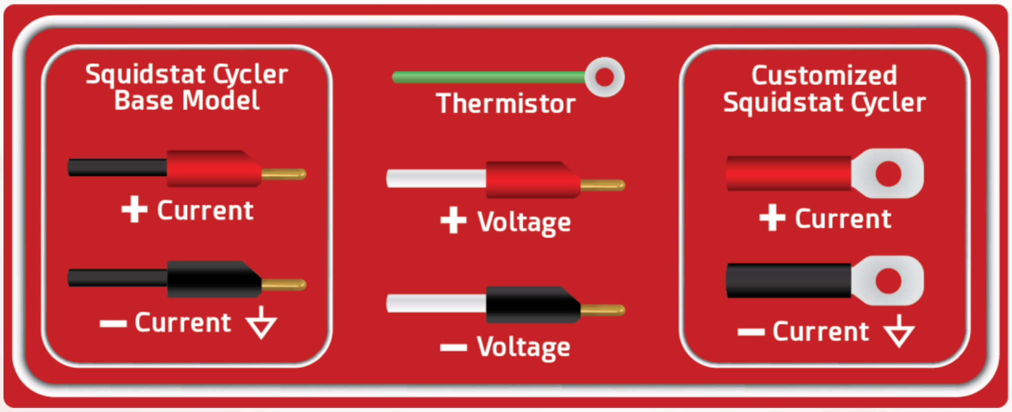
Reference Lead (RE): Green Wire/Green Plug. Voltage sensing lead. Serves as a reference point, representing the "zero" potential against which the working and counter electrode potentials are compared. Can be connected to either the reference electrode or the counter electrode of an electrochemical cell.

Squidstat Penta, Decka, and Venta models require a 2mm female to 4mm male banana plug adaptor (not provided) to stack leads. The sense lead should **ALWAYS** be plugged into the **BACK** of the current carrying lead. Not doing so could damage the sense lead from exposure to high currents!

7.3 Squidstat Cycler Base Model and Customized Squidstat Cycler

Channel cabling for the Squidstat Cycler Base Model consists of five total leads: four leads terminating in a 2 mm male/female banana plug and 1 thermistor lead terminating in a 3 mm ring.

Customized Squidstat Cyclers are shipped with three cables that function together as one channel cable with five leads: two separate current leads terminating in an M6 Lug connection, one sense lead cable consisting of two voltage leads terminating in a 2 mm male/female banana plug, and 1 thermistor lead terminating in a 3 mm ring. The diagram below shows the color scheme of the leads for all Squidstat Cyclers.



Squidstat Cycler Base Model leads can either be connected to a cell individually or stacked together by inserting the banana pin of one lead into the hole on the back of the banana pin of another lead.

Positive (+) Current Lead: Black Wire/Red Plug. Current carrying lead. Connects to the positive terminal of a cell. Analogous to the working lead.

Positive (+) Voltage Lead: White Wire/Red Plug. Voltage sensing lead. Connects to the positive terminal of a cell. Can be stacked with the positive current lead. Analogous to the working sense lead.

Negative (-) Current Lead: Black Wire/Black Plug. Current carrying lead. Connects to the negative terminal of a cell. This lead also functions as a signal ground, indicated by the ⏚ symbol. Current flows only between the Positive and negative current leads. Analogous to the counter lead.

Negative (-) Voltage Lead: White Wire/Black Plug. Voltage sensing lead. Serves as a reference point, representing the "zero" potential against which the positive voltage lead potential is compared. Connects to the negative terminal of a cell and can be stacked with the negative current lead. Analogous to the reference lead.

Thermistor Lead: Temperature sensing lead. Place anywhere a temperature measurement is required.

7.4 Extended Length Cabling

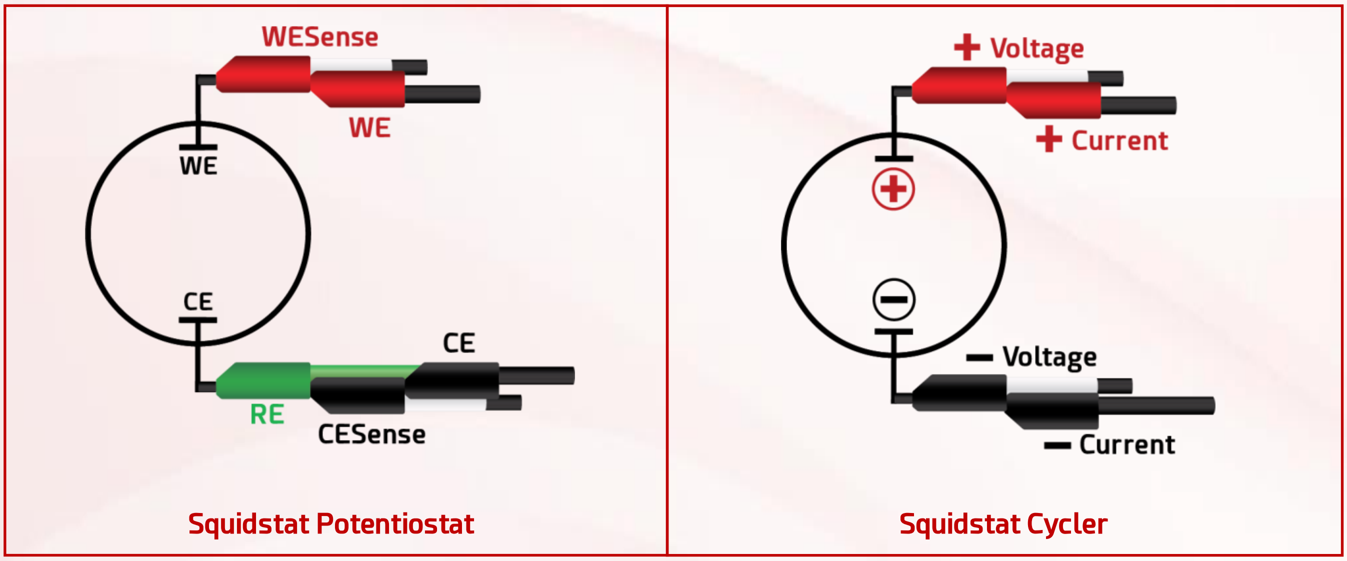
All Squidstat potentiostat and Cycler models come with a standard channel cable that is 1 m in length. Extended length cabling options are only available for the Squidstat Cycler Base Model and Customized Squidstat Cycler at two additional lengths, 2 m and 3 m. The Squidstat potentiostat models are unable to support longer cable lengths at the time of publishing, but that is subject to change. If you wish to purchase a longer length cable for the Cycler, please contact sales@admiralinstruments.com for pricing and additional information.

8 Cell Configurations

There are several ways to connect an electrochemical cell to a Squidstat potentiostat or Squidstat Cycler, depending on the number of electrodes and the type of measurement required. Common configurations are described below for two, three, and four-electrode configurations.

8.1 Two-Electrode Configuration

The two-electrode cell is the simplest electrochemical setup, consisting of three components: a working electrode (WE), a counter or auxiliary electrode (CE) and an electrolyte. Electrons, or electronic current, flow between the WE and CE through an external circuit consisting of a potentiostat. Ions, or ionic current, flow between the WE and CE through the electrolyte. Most electrochemical devices such as batteries and fuel cells are configured as two-electrode systems. This configuration is recommended for electrochemical cells with resistances > 1 kΩ.



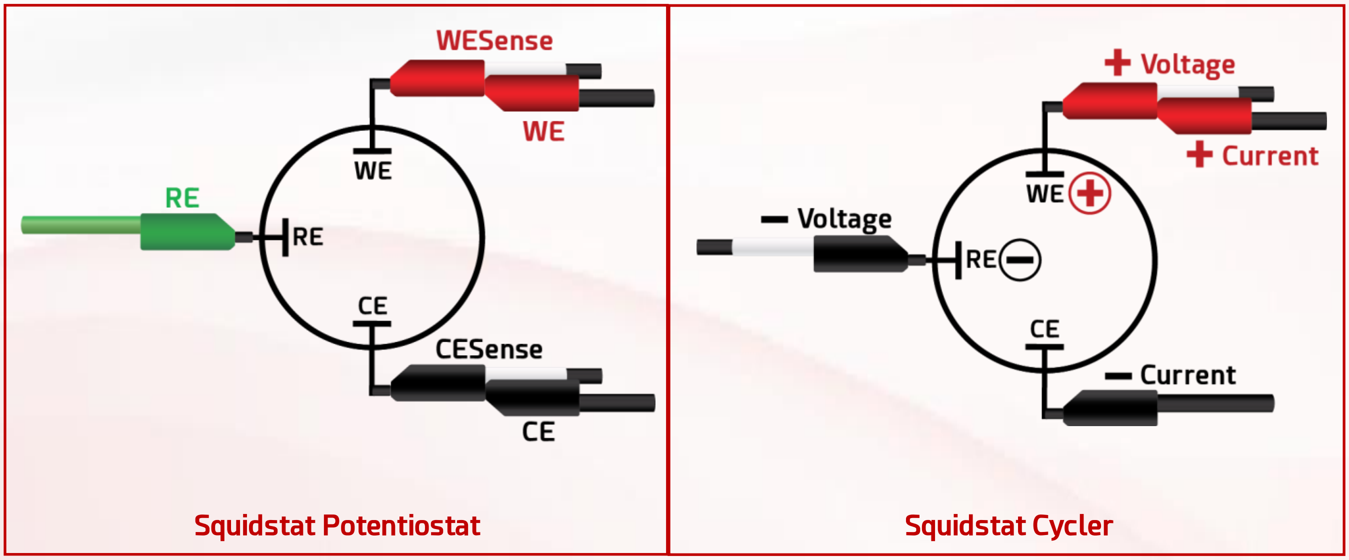
For a standard five-lead Squidstat potentiostat, the working lead and working sense lead are stacked together and connected to the WE. The counter lead, counter sense lead, and reference lead are stacked together and connected to the CE. This forces the reference and the counter leads to be at the same potential.

Similarly, to configure the Squidstat Cycler with four leads in a two-electrode configuration, the positive (+) voltage and (+) current leads are stacked and connected to the positive terminal, while the negative (-) voltage and (-) current leads are stacked and connected to the negative terminal.

The Squidstat Cycler operates in a unipolar configuration by default. Unless the instrument is configured for bipolar operation, always connect the positive leads (red) to the positive terminal and the negative leads (black) to the negative terminal of the cell.

8.2 Three-Electrode Configuration

The three-electrode configuration is the most popular configuration in conventional electrochemistry. The addition of an independent reference electrode (RE) allows for precise voltage control at the WE independent of current and ohmic drop. Current flows between the WE and the CE to hold the WE at the specified potential setpoint. Voltage changes at the CE during the experiment do not affect the potential at the WE, since the potential of the WE is applied or measured with respect to the RE. Little or no current flows through the RE.



For a standard five-lead Squidstat potentiostat, the working lead and the working sense lead are stacked and connected to the WE. The counter lead and counter sense lead are stacked and connected to the CE, and the reference lead is connected to the RE.

The Squidstat Cycler leads can also be arranged in a three-electrode configuration. The negative (-) voltage lead is connected to the RE, the positive (+) voltage and (+) current leads are stacked and connected to the WE, and the (-) current lead is connected to the CE. Unlike Squidstat potentiostats, Squidstat Cyclers do not have a counter sense lead, thus the potential at the CE is not reported.

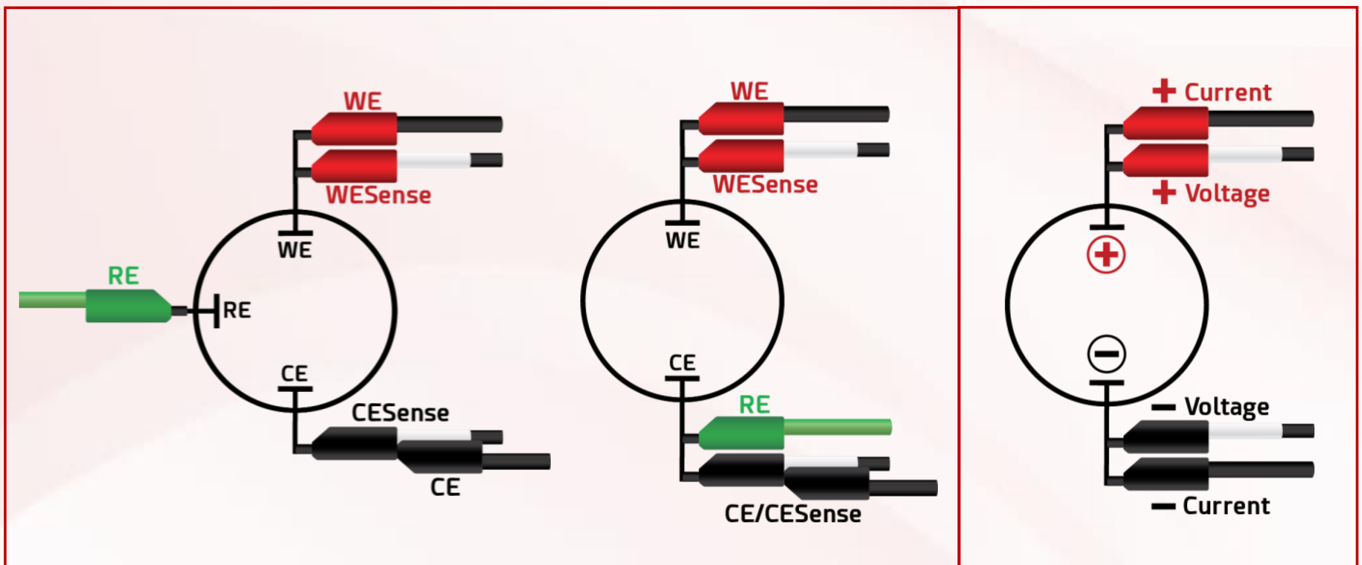
The Squidstat Cycler operates in a unipolar configuration by default. The 3-electrode configuration can only be employed if the potential of the WE is positive relative to the RE **and** the CE if the instrument is not configured in Bipolar mode.

8.3 Two-Electrode and Three-Electrode Configurations with Four-Point Connections

In a four-point connection measurement, the voltage sense leads and current carrying leads are connected to the electrodes separately instead of in a stacked position. These configurations are recommended for impedance measurements of electrochemical cells with low resistance (≤ 1 kΩ). Separating the sense leads from the current carrying leads eliminates contact resistance which increases the measured impedance higher than the actual value.

For a standard five-lead Squidstat potentiostat, the working lead and working sense lead are connected separately to the WE. The counter lead and the counter sense lead can be stacked and connected to the CE, and the reference lead can either be connected to the CE for the two-electrode four-point configuration or to the RE for the three-electrode four-point configuration. Since the counter sense lead is not part of the voltage sense feedback loop, it can be stacked with the counter lead. However, for very low impedance cells, the counter sense lead can be removed from the circuit entirely.

For the 4-lead Squidstat Cycler, the positive (+) voltage and (+) current leads are connected separately to the positive terminal, while the negative (-) voltage and (-) current leads are connected separately to the negative terminal.

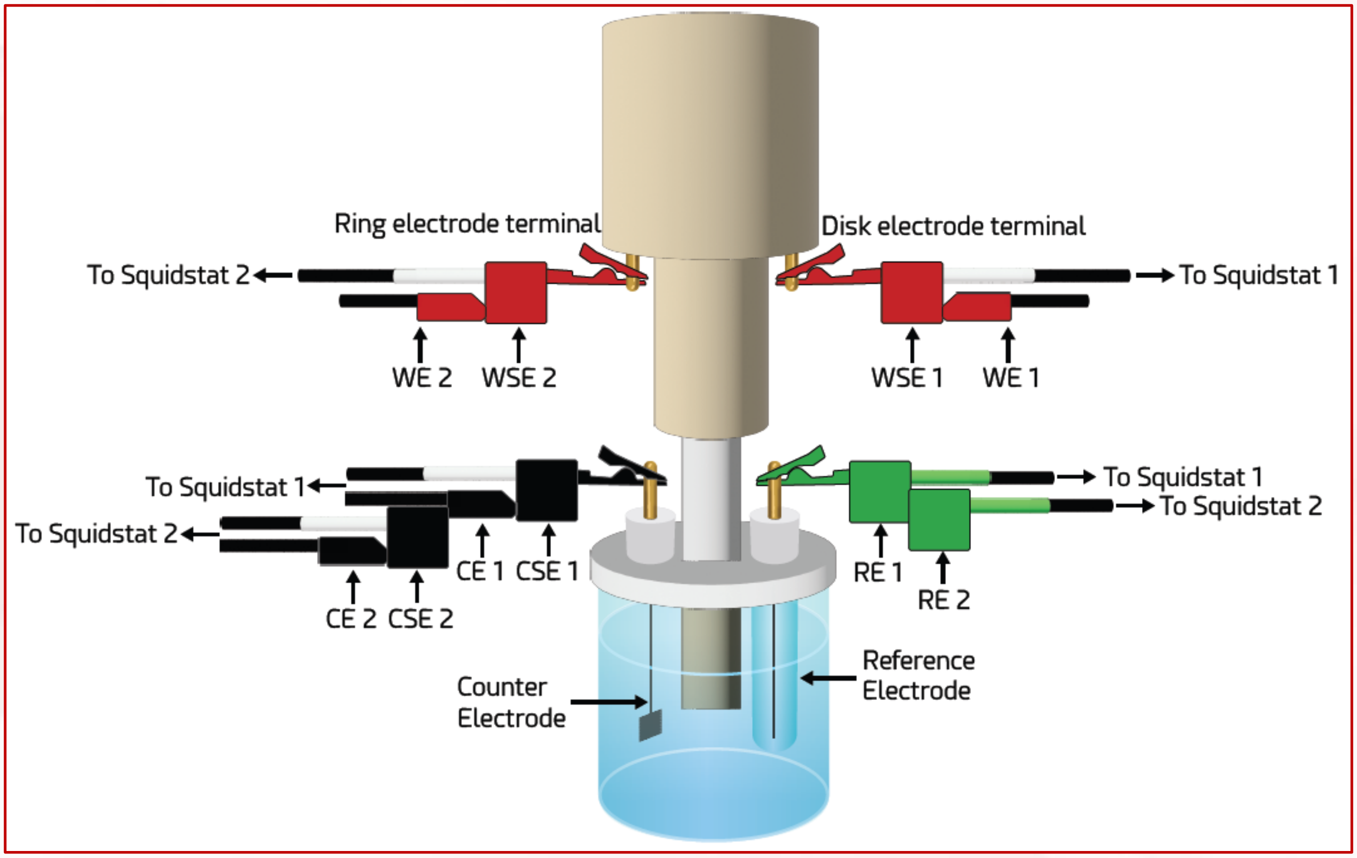


Squidstat Potentiostat | Squidstat Cycler

The sense leads measure the total resistance and voltage between the two connection points. For the most accurate measurements, the sense leads should be connected closer to the terminal than the current leads, as illustrated in the figure above.

8.4 Four-Electrode Bipotentiostat Configuration with Two Squidstats

A typical example of a 4-electrode bipotentiostat configuration is a rotating ring-disk electrode (RRDE). The cell configuration consists of two working electrodes (a disk and a ring), one reference electrode, and one counter electrode. Two Squidstat instruments are required to complete an RRDE experiment and each must be in float mode. The diagram below shows the reference leads from both instruments are stacked and connected to the same RE. All counter leads and counter sense leads are stacked together and connected to the same CE. The working lead and working sense lead from Squidstat-1 are connected to WE-1, while the working lead and working sense lead from Squidstat-2 are connected to WE-2. Please refer to the application note "Running rotating ring-disk electrode (RRDE) experiments with Squidstat potentiostats" for more information regarding completing an RRDE experiment using two Squidstats in a bipotentiostat configuration.



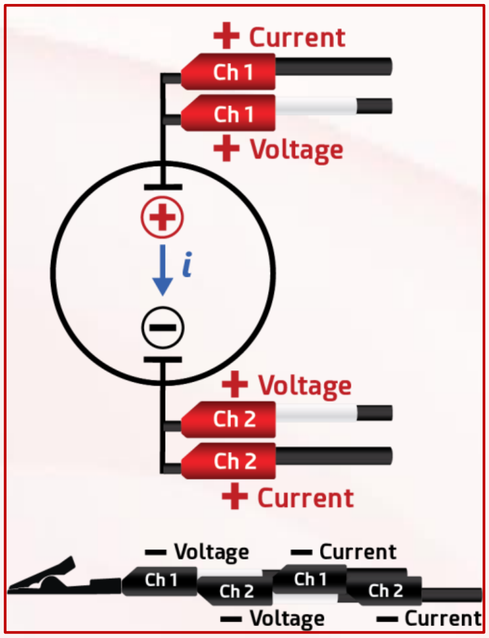
Two independent Squidstat potentiostats must be employed in a bipotentiostat configuration. Multi-channel Squidstat potentiostats such as the Squidstat Prime and Squidstat Cycler cannot combine two channels in a bipotentiostat configuration, as all channels share a common ground.

8.5 Multichannel Bipolar Operation (Squidstat Cycler Only)

Bipolar operation pairs two channels to extend the voltage range of a single channel, enabling operation at negative voltages. For example, when two channels on the Base Model Cycler are configured for bipolar operation, the resulting limits are ±5 V and ±5 A for the combined channel.

On models with more than two channels, only adjacent channels can be configured for bipolar operation. For instance, on a 4-channel Cycler, the two valid combinations are channels 1 and 2 or channels 3 and 4; other combinations are not supported.

The graphic below illustrates the lead setup for bipolar operation using channels 1 and 2. The positive (red) output leads connect to the battery or device under test, while the negative (black) output leads are stacked and set aside.



See Section 10.6.14 for instructions on how to enable bipolar operation in the Squidstat User Interface software.

The lower numbered channel (1 or 3) is always connected to the positive terminal of the battery or device under test, while the higher numbered channel (2 or 4) is always connected to the negative terminal.

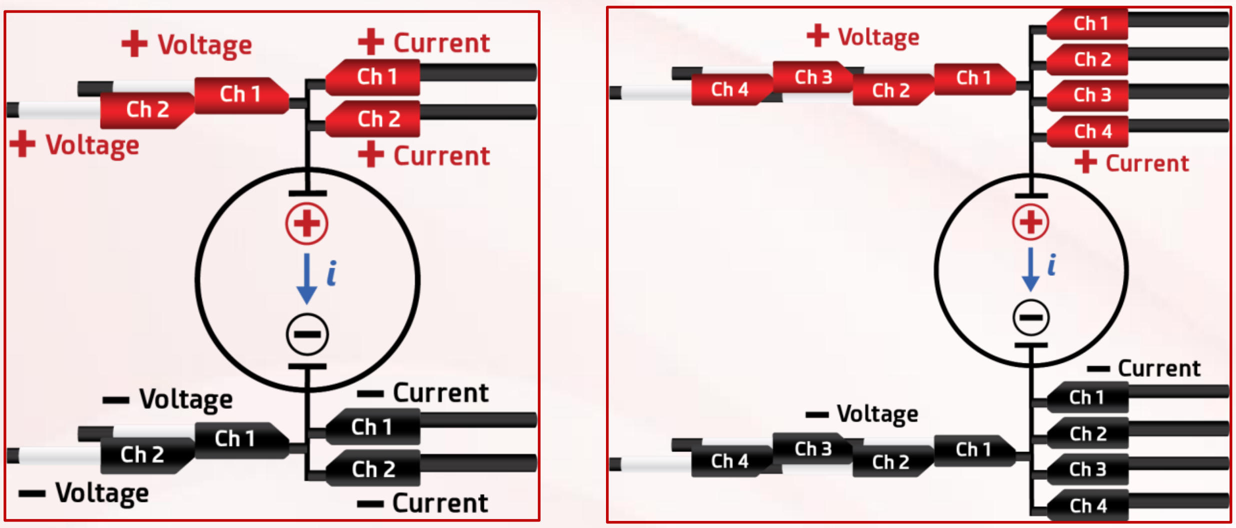
8.6 Multichannel Parallel Operation (Squidstat Cycler Only)

Parallel operation increases the maximum current output by combining multiple channels to function as a single channel. When channels are configured in parallel, the current limit becomes the sum of the current limits of all combined channels. Examples for the Base Model Squidstat Cycler:

- **2 channels** with ±5 A limits in parallel mode provide a single channel with ±10 A and 0-5 V.
- **3 channels** with ±5 A limits in parallel mode provide a single channel with ±15 A and 0-5 V.
- **4 channels** with ±5 A limits in parallel mode provide a single channel with ±20 A and 0-5 V.

> Only the current limit is increased by parallel operation, proportional to the number of channels linked. The voltage limit does not increase!

Any two or more channels can be combined in parallel, provided they are correctly configured in the Squidstat User Interface. See Section 10.6.14 for instructions on how to configure parallel operation in the software.



The graphic above illustrates two lead configurations: the left image shows parallel operation with two channels, while the right image shows parallel operation with all four channels. Voltage sense leads can be stacked, but current leads must be connected individually.

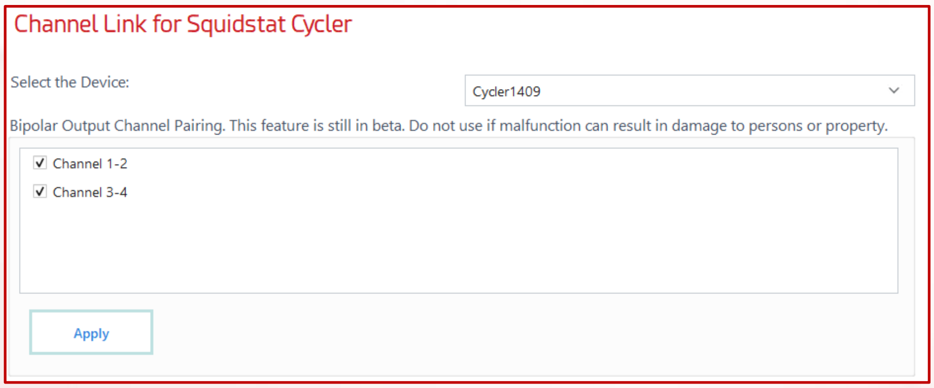
> The standard 2 mm alligator clips and cable wiring are not rated for more than 10 A, so the current leads **must** be connected individually!

8.7 Bipolar and Parallel Combined Operation (Squidstat Cycler Only)

If the Squidstat Cycler has at least 4 channels, it is possible to configure all 4 channels as a single channel with both bipolar and parallel operation. This provides access to negative voltages and also increases the maximum current limit. Using the Base Model Cycler (0-5 V, ±5 A per channel) as an example, when all 4 channels are configured for bipolar and parallel combined operation, the current and voltage limits are ±10 A and ±5 V, respectively.

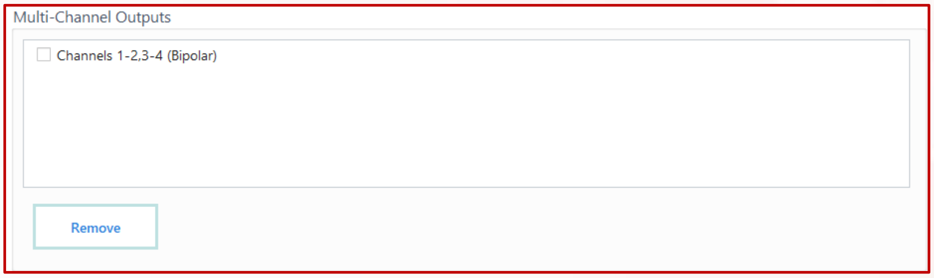
Multiple steps are required to enable this operating mode in the software, described below.

1. In the ***More Options*** tab, under the Beta Features menu, select **Channel Link for Squidstat Cycler**
2. Select Channel 1-2 and Channel 3-4 in the Bipolar Output Channel Pairing menu and click ***Apply***

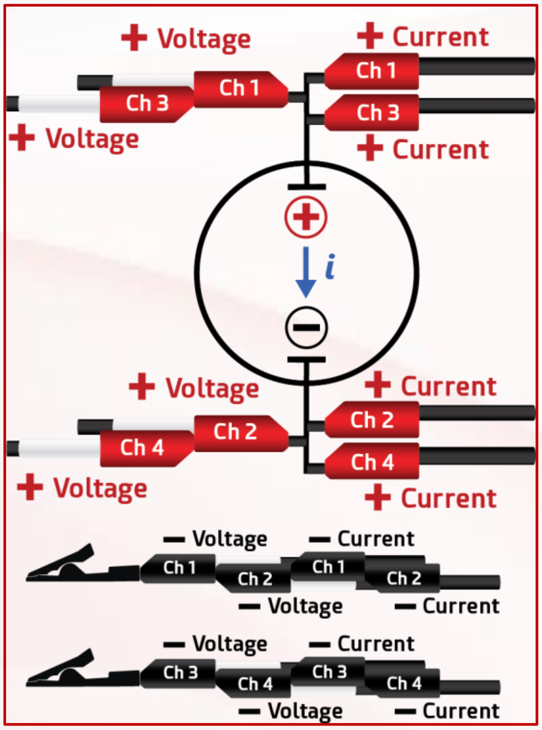


3. Select Channel 1-2 and Channel 3-4 in the Parallel Channel Output menu and click ***Add***

4. If completed successfully, the linked channels should appear in the Multi-Channel Outputs list as Channels 1-2,3-4 (Bipolar)

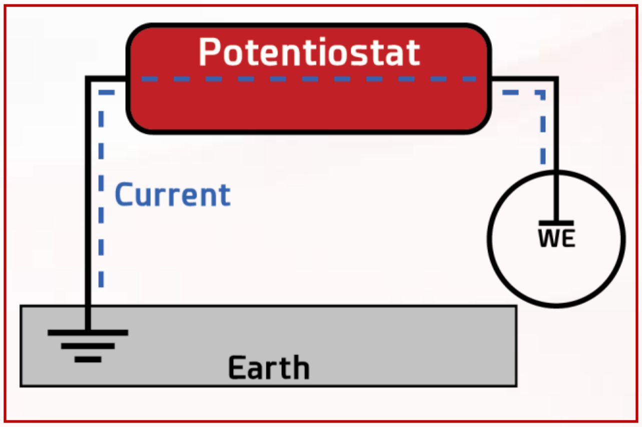


The graphic below shows how the leads should be connected to the cell for bipolar/parallel combined operation. As in bipolar mode, the red leads are connected to the cell, while the black leads are stacked and set aside. The lower numbered channels (1 and 3) are always connected to the positive terminal of the battery or device under test, while the higher numbered channels (2 and 4) are always connected to the negative terminal. As in parallel mode, the current carrying leads should be connected to the cell individually while the voltage sensing leads can be stacked.

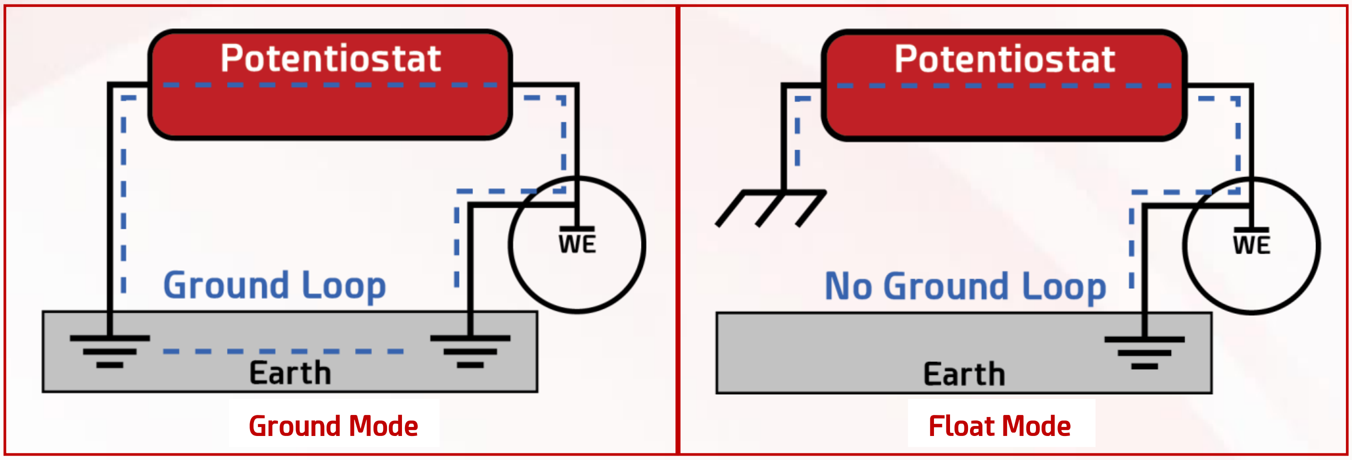


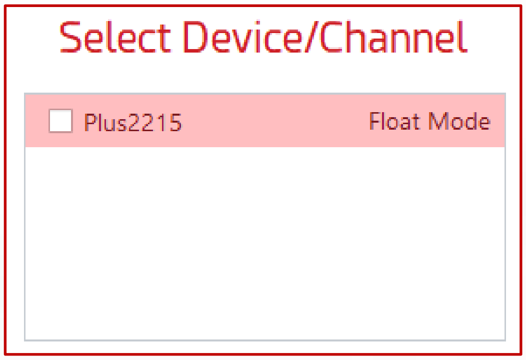
8.8 Ground and Float Mode

When a device is grounded, it is connected to an infinite source or sink of charge. The most common ground is the planet Earth, aptly named "Earth ground." The primary reason for grounding is safety: grounding provides a path for excess current in the case of an overvoltage event and reduces the buildup of electrostatic charge. The secondary reason for grounding is to define a zero-voltage reference point, which gives the basis to express voltage potentials relative to ground. All Squidstat potentiostats (excluding the Squidstat Cycler) can be configured to run in two grounding modes, ground and float, via a switch on the back panel of the instrument. In ground mode, the working electrode is Earth grounded (pictured right), while in float mode, the working electrode is connected to an internal ground.



For the majority of use cases, the Squidstat should be in ground instead of float mode, but there are exceptions when there is the potential for ground loops to form. For example, float mode should be used if a Squidstat and one of the electrodes it's connected to are both grounded through the common Earth ground via a power source, most often a common electrical outlet. In this configuration, current now has multiple pathways to ground, forming a conductive ground loop, shown in the diagram (left) below. The flow of current through this loop can cause a voltage drop, altering the zero-reference point and resulting in unstable and inaccurate voltage measurements. However, if the potentiostat is in float mode (pictured right), current can only travel on one pathway, and no ground loops form. Another instance where float mode should be used in the bipotentiostat configuration required for RRDE experiments, as both Squidstats are electrically connected through the working electrode.





When certain Squidstat models are in float mode, the software will display a “float mode” indicator next to the instrument's serial number (pictured left). There is no indicator when the switch is set to ground mode. This feature is available on Squidstat Plus models with serial numbers 1700 or greater, Prime models with serial numbers 1101 or greater, and Solo models with serial numbers 1260 or greater.

There is a 4mm female banana plug on the rear panel of all Squidstat potentiostats (excluding the Squidstat Cycler) which can be used to ground external objects such as a Faraday cage. This ground port is always grounded with respect to earth, regardless of whether the unit is in float or ground mode.

On Squidstat Penta, Decka, and Venta models, the D-sub connector for the sense cable can also be grounded to remove environmental noise, particularly for higher impedance (> 1 MOhm) test objects. At minimum, the D-sub connector should be grounded to the 4 mm grounding port on the rear panel of the instrument (pictured left). If environmental noise is still present, place the test object in a Faraday cage or other shielding and ground both the shielding and the D-sub connector to the grounding port (pictured right).

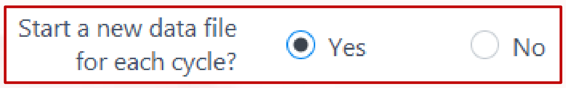
9 Electrochemical Methods – Pre-Built Experiments and Method Tiles

This section describes the default pre-built experiments found under the ***Run an Experiment*** tab and the method tiles available in the ***Build an Experiment*** tab. Both groups of experiments are presented here as many of the default pre-built experiments are also available as method tiles. The availability of each experiment in this section is defined as "Pre-Built" or "Method Tile".

9.1 General Notes - Pre-Built Experiments and Method Tiles

The following options can be found in multiple pre-built experiments and method tiles and are defined here once, for brevity.

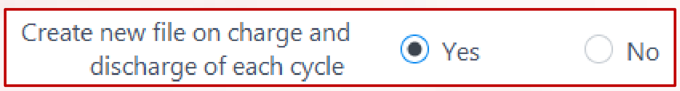
Start a new data file for each cycle?:



1. **Cyclic Voltammetry, Staircase Voltammetry, GITT, PITT, and Rotating Ring Disk Electrode**: Selecting "No" will produce one CSV file containing all data collected. Selecting "Yes" will split each cycle into individual CSV files.

2. **CC-CV Charge/Discharge and CP/CR Charge/Discharge**: The file splitting structure for these experiments depends on the selection for both **Start a new data file for each cycle** as well as the following option:

   Create a new file on charge and discharge of each cycle?:



   Start a new data file for each cycle will always supersede **Create new file on charge and discharge of each cycle.** The file output structure for all possible combinations is outlined below:

   - Selecting "No" for both options will produce one CSV file containing all data collected.
   - Selecting "Yes" for both options will produce one CSV file for each charge and discharge step in every individual cycle. i.e. 20 cycles = 40 total files.
   - Selecting "No" for **Start a new data file for each cycle** and "Yes" for **Create a new file on charge and discharge of each cycle** will produce two CSV files:
     - 1) all charge data collected
     - 2) all discharge data collected
   - Selecting "Yes" for **Start a new data file for each cycle** and "No" for **Create a new file on charge and discharge of each cycle** will produce a single file for each cycle containing both charge and discharge data.

With respect to: The relative potentials against which the working electrode potential is set. Users can select either “open circuit” or “reference.” Reference refers to the potential of the electrode where the reference electrode clip is attached. Open circuit refers to the open circuit potential, which is the potential of the working electrode (relative to the reference electrode) when there is no flow of current. For custom experiments containing multiple tiles, the open circuit potential is defined at the start of the experiment and redefined only when an open circuit potential tile is placed.

Current and voltage ranging: More than one current and voltage range is available to measure a wide scale of current and voltage magnitudes accurately. Although users can let the software automatically choose the current range(s) used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for Autorange and fixed range modes. Fixed range mode is useful to avoid noise created when the Squidstat switches between current ranges. Autorange is disabled for pulse and pulse voltammetry experiments to maintain measurement stability and accuracy, as rapid range changes can introduce artifacts or distortions in the fast, transient responses of these techniques.

Autorange: This is the default setting. The current range is automatically adjusted to match the measured signal's amplitude, optimizing accuracy and resolution. This allows the instrument to dynamically switch current ranges to capture both low and high signals without manual intervention.

Maximum current expected: Select the desired current range by entering a maximum current value. In fixed range mode, the Squidstat will not switch to a more sensitive range than specified; however, if the current exceeds the selected range, the Squidstat will automatically switch to a higher range to protect the hardware and prevent premature experiment termination—except during pulse and pulse voltammetry experiments, where the experiment will end if the current exceeds the set range.

C-rate as a unit: C-rate is a unit option for the current setpoint in galvanostatic DC experiments, representing the rate at which a battery is charged or discharged relative to its maximum capacity in mAh. For example, charging a 2800 mAh battery at 1C will fully charge it from 0% to 100% state of charge (SOC) in 1 hour using 2.8 A. Discharging the same battery at 1/5 C will drain it from 100% to 0% SOC in 5 hours using 560 mA. If C-rate is chosen as the unit for current, the battery capacity in mAh must be entered in the Experiment Settings pop-up when the experiment is initiated.

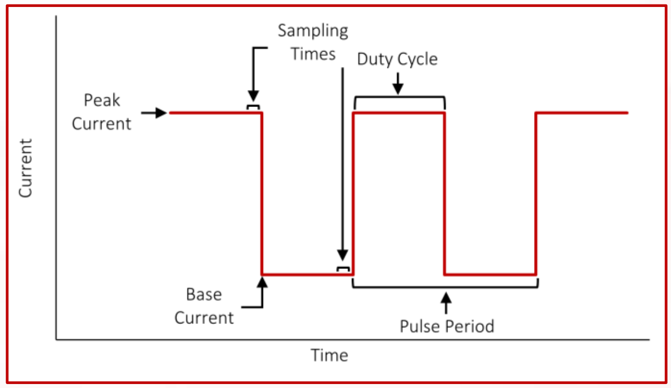
α-Factor (Sweep and Pulse Voltammetry): Due to the digital nature of modern potentiostats, an edge-free analog voltage or current ramp cannot be applied. Ramping is completed in very small steps. For example, during a voltage ramp, the potential is increased by a small step and held at that potential for a certain amount of time or step period. A user can choose how to sample data during the step period by varying the α-Factor, which is the ratio of the data sampling period to the step period. If the α-Factor is 25%, the last 25% of the recorded data are averaged and tabulated in the CSV file. If the α-Factor is 100%, all the data recorded during the step period is averaged and tabulated in the CSV file.

It is recommended to vary the α-Factor to get the best response during voltammetry and pulse experiments, including linear sweep voltammetry, cyclic voltammetry, normal pulse voltammetry, differential pulse voltammetry, square wave voltammetry, basic potential pulse, and basic current pulse.

Append data to the last CSV file that has matching column headers: This applies to **Method Tiles** only. If a user wants to append the data from this Method Tile to the CSV file from the previous Method Tile, select "Yes". If a user wants to create a separate CSV file for the method tile, select "No". In cases where this method tile is set to loop to run multiple times before moving on to the next tile, selecting "No" will create a new CSV file for each loop.

9.2 Basic Current Pulse – Method Tile

A constant current pulse is superimposed over a steady base current, with the pulse duration (duty cycle) expressed as a percentage of the pulse period. Voltage response is sampled and averaged over 10 µs at two points: at the end of the duty cycle and the end of the pulse period. The α-factor specifies the portion of this data to be averaged and reported, and the voltage response is plotted over time. The graphic to the right illustrates the current waveform applied to the working electrode.



General Options

Base current: The current applied during the "off" phase of the pulse period.

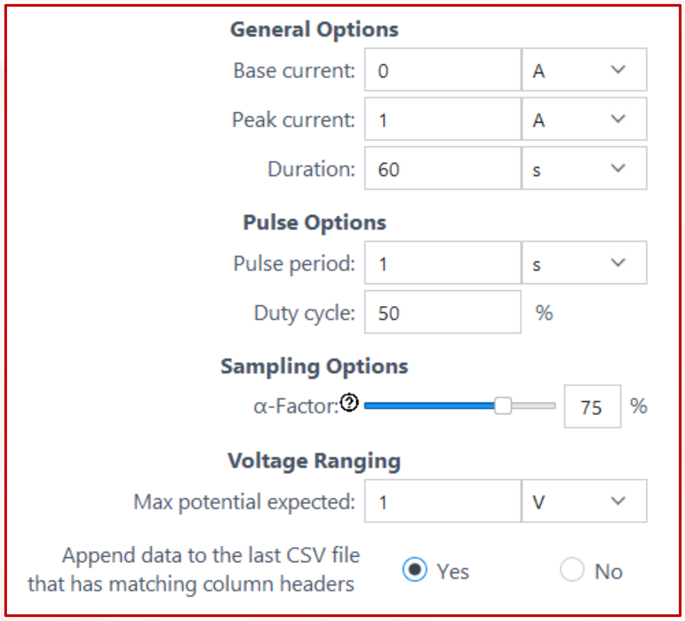
Peak current: The current applied during the "on" phase of the pulse period.

Duration: The total duration of the pulsed experiment.

Pulse Options

Pulse period: Total time for one pulse cycle. Includes time spent at both the peak and base current.

Duty Cycle: Percentage of one pulse period spent in the "on" or active phase. For a duty cycle of 25%, the peak current is applied for the first 25% of the pulse period, while the base current is applied for the remaining 75% of the pulse period.



Sampling Options

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

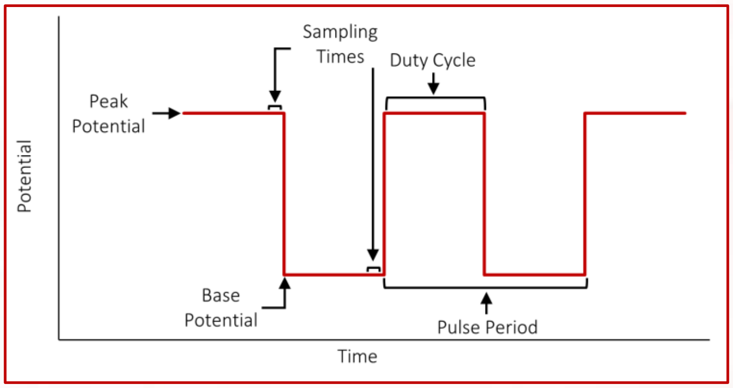
Voltage Ranging

Max potential expected: Designates which voltage range will be used to measure the voltage response. See general notes.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.3 Basic Potential Pulse – Method Tile

A constant potential pulse is superimposed over a steady base potential, with the pulse duration (duty cycle) expressed as a percentage of the pulse period. Current response is sampled and averaged over 10 µs at two points: at the end of the duty cycle and the end of the pulse period. The α-factor specifies the portion of this data to be averaged and reported, and the current response is plotted over time. The graphic to the right illustrates the potential waveform applied to the working electrode.



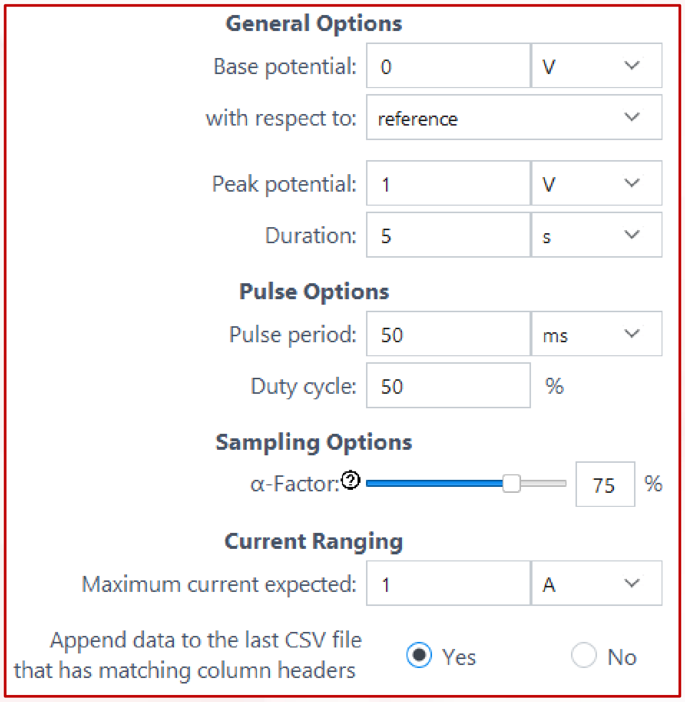
General Options

Base potential: The potential applied during the "off" phase of the pulse period.

With respect to: Reference or open circuit. See general note.

Peak potential: The potential applied during the "on" phase of the pulse period.

Duration: The total duration of the pulsed experiment. Must be longer than the pulse period.



Pulse Options

Pulse period: Total time for one pulse cycle. Includes time spent at both the peak and base potentials.

Duty Cycle: Percentage of one pulse period spent in the "on" or active phase. For a duty cycle of 25%, the peak potential is applied for the first 25% of the pulse period, while the base potential is applied for the remaining 75% of the pulse period.

Sampling Options

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

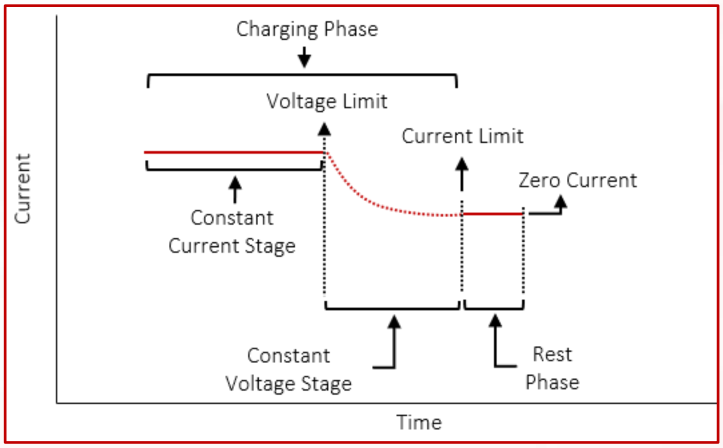
Current ranging

Maximum current expected: Determines which current range is used to measure the current response. See general notes.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

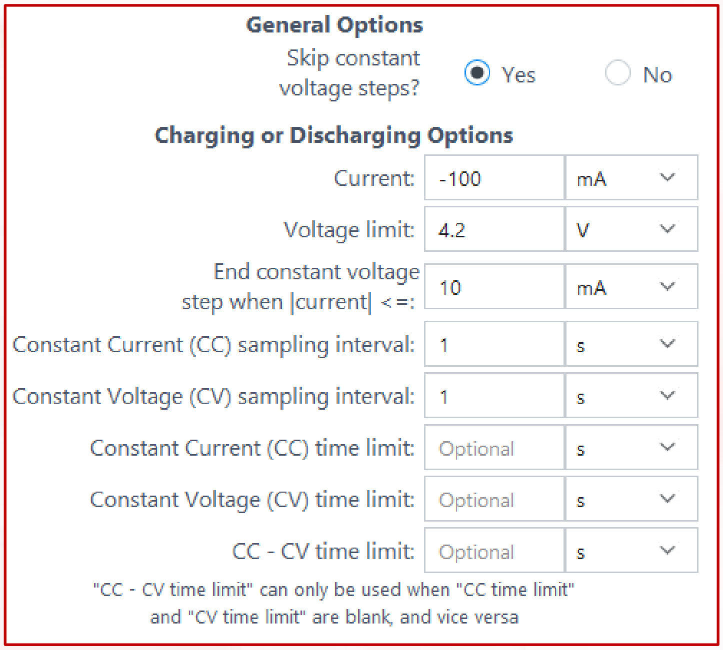
9.4 CC-CV Charge or Discharge – Method Tile

This method tile will charge or discharge an electrochemical device under test (DUT). For each charge and discharge phase, the electrochemical device undergoes a constant current (CC) segment followed by a constant voltage (CV) segment. The graphic to the right shows the waveform of current applied to the working electrode for CC-CV Charge or Discharge, using Charge as an example. During the rest period, the potentiostat is in open circuit potential mode.



General options

Skip constant voltage steps?: Select "Yes" to skip the constant voltage (CV) segment, or "No" to perform this step.



Charging or Discharging Options

Current: Current applied during charging or discharging. Input a positive (+) current value to charge and a negative (-) current value to discharge.

Voltage limit: Current is applied to charge or discharge the DUT until this voltage is reached.

End constant voltage step when |current| <=: Charging or discharging will stop if the absolute value of the current is less than or equal to this value when maintaining the voltage limit during the constant voltage step.

Constant Current (CC) sampling interval: The time or voltage interval between two consecutive data points during the constant current phase.

Constant Voltage (CV) sampling interval: The time interval between two consecutive data points during the constant voltage phase.

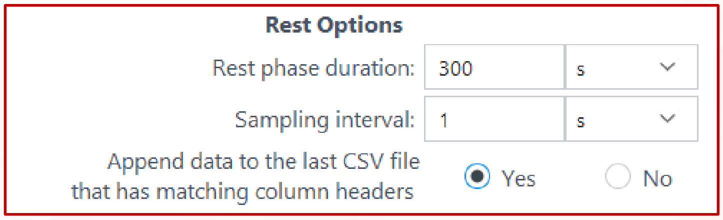
Constant Current (CC) time limit: Stops CC phase if the voltage limit is not met by this time. Potentiostat will then switch to the CV phase. *If this option is enabled the CC-CV time limit must be blank*!

Constant Voltage (CV) time limit: Stops CV phase if the current limit is not met by this time. Potentiostat will then switch to the rest phase. *If this option is enabled the CC-CV time limit must be blank*!

CC-CV time limit: If both the CC and CV phases are not completed by this time, the potentiostat will then switch to the rest phase. *If this option is enabled both the CC time limit and the CV time limit must be blank*!

Rest Options

Rest phase duration: Amount of time the Squidstat will spend recording the open circuit potential after the constant voltage segment during charge or discharge.

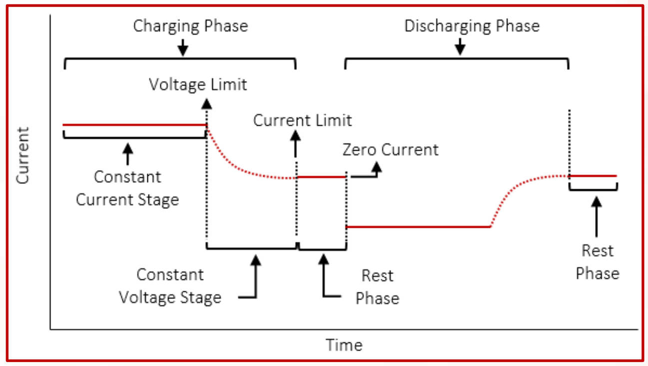


Sampling interval: The time interval between two consecutive data points during the rest phases.

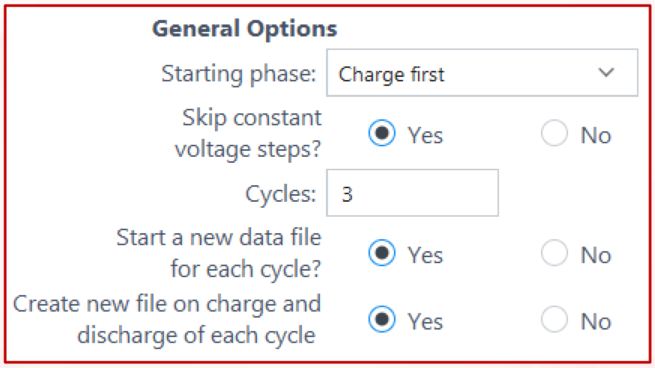
Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.5 CC-CV Charge/Discharge – Pre-Built Experiment and Method Tile

This experiment charges and discharges an electrochemical device under test (DUT). For each charge and discharge phase, the electrochemical device undergoes a constant current (CC) stage followed by a constant voltage (CV) stage. The CC stage ends when the voltage limit is reached. During the rest period, the potentiostat is in open circuit potential mode. The graphic to the right shows the waveform of current applied to the working electrode for CC-CV Charge/Discharge.



General options



Starting phase: Select whether the cycle begins by charging or discharging.

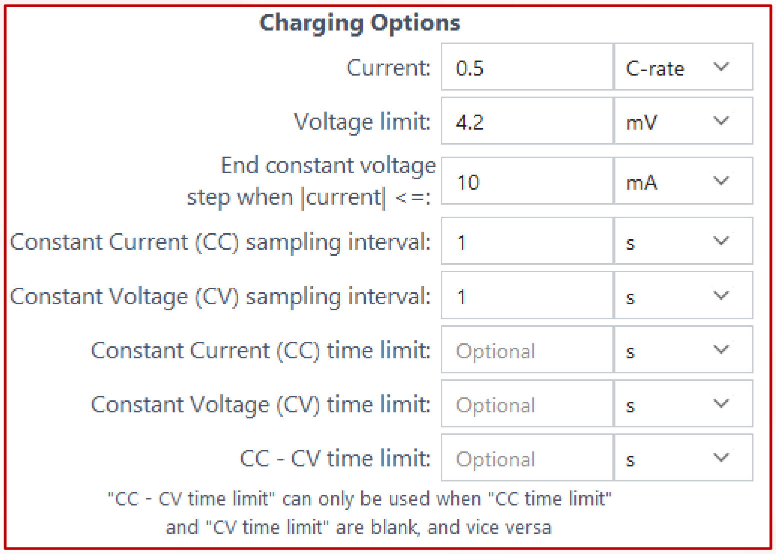
Skip constant voltage steps?: Select "Yes" to skip the constant voltage (CV) segment, and "No" to perform CC-CV charge/discharge.

Cycles: The number of times a user wants to run the charging/discharging cycles before the experiment ends.

Start a new data file for each cycle?: Separate data collection per cycle. See general notes.

Create new file on charge and discharge of each cycle?: Separate charge and discharge data. See general notes.

Charging Options



Current: Current applied during charging.

Voltage limit: Charging current applied until this voltage is reached.

End constant voltage step when |current| <=: Charging will stop if the current is equal or lesser than this value when maintaining the upper voltage limit for the rest phase duration.

Constant Current (CC) sampling interval: The interval between two consecutive data points during the constant charging phase.

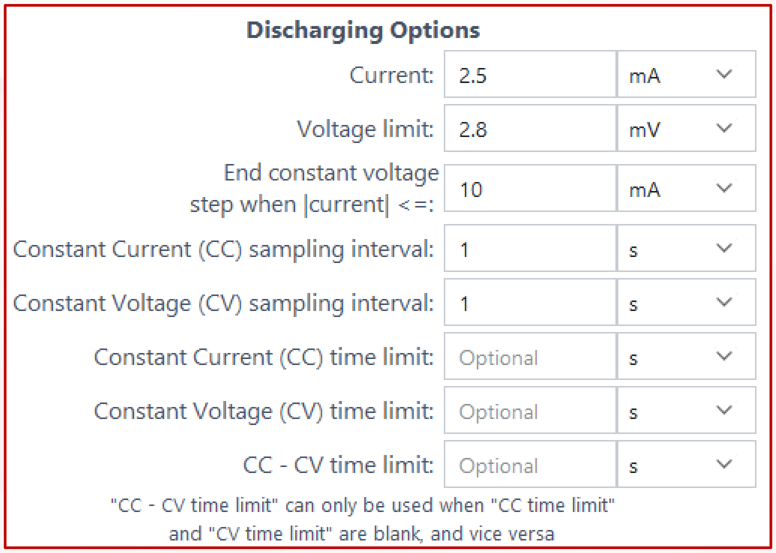
Constant Voltage (CV) sampling interval: The interval between two consecutive data points during the constant voltage phase.

Constant Current (CC) time limit: Stops CC phase if the voltage limit is not met by this time. Potentiostat will then switch to the CV phase. *If this option is enabled the CC-CV time limit must be blank*!

Constant Voltage (CV) time limit: Stops CV phase if the current limit is not met by this time. Potentiostat will then switch to the rest phase. *If this option is enabled the CC-CV time limit must be blank*!

CC-CV time limit: If both the CC and CV phases are not completed by this time, the potentiostat will then switch to the rest phase. *If this option is enabled both the CC time limit and the CV time limit must be blank*!

<u>Discharging Options</u>



Current: Current applied during discharge.

Voltage limit: Discharging current applied until this voltage is reached.

End constant voltage step when |current| <=: The Squidstat will stop discharging if the current is equal or lesser than this value when maintaining the lower voltage limit for the rest phase duration.

Constant Current (CC) sampling interval: The interval between two consecutive data points during the constant charging phase.

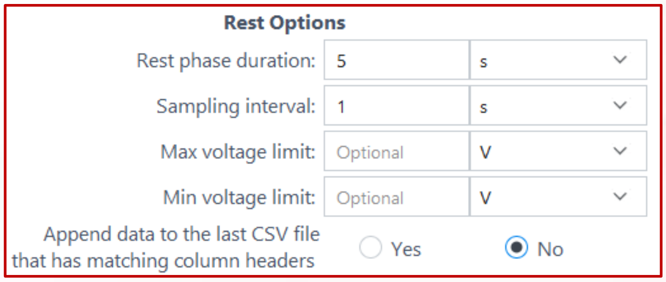
Constant Voltage (CV) sampling interval: The interval between two consecutive data points during the constant voltage phase.

Constant Current (CC) time limit: Stops CC phase if the voltage limit is not met by this time. Potentiostat will switch to the CV phase. *If this option is enabled the CC-CV time limit must be blank*!

Constant Voltage (CV) time limit: Stops CV phase if the current limit is not met by this time. Potentiostat will switch to the rest phase. *If this option is enabled the CC-CV time limit must be blank*!

CC-CV time limit: If both the CC and CV phases are not completed by this time, the potentiostat will switch to the rest phase. *If this option is enabled both the CC time limit and the CV time limit must be blank*!

Rest Options



Rest phase duration: Amount of time the Squidstat will spend at open circuit after the constant voltage segment during charge/discharge.

Sampling interval: The time interval between two consecutive data points during the rest phases.

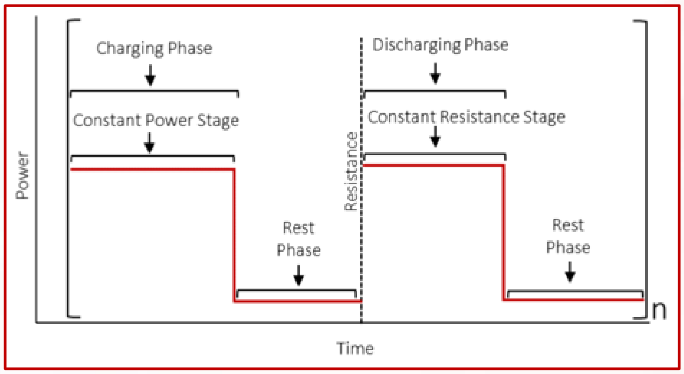
Max voltage limit: The rest phase will end if the voltage rises above this value.

Min voltage limit: The rest period will end if the voltage drops below this value.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.6 CP/CR Charge/Discharge – Pre-Built Experiment

This experiment discharges an electrochemical device with a constant resistance (CR) load and charges the device with a constant power (CP) source. The accompanying graphic to the right shows a profile of power and resistance applied during charging and discharging cycles, respectively, vs. time. During the rest period, the potentiostat is in open circuit potential mode.



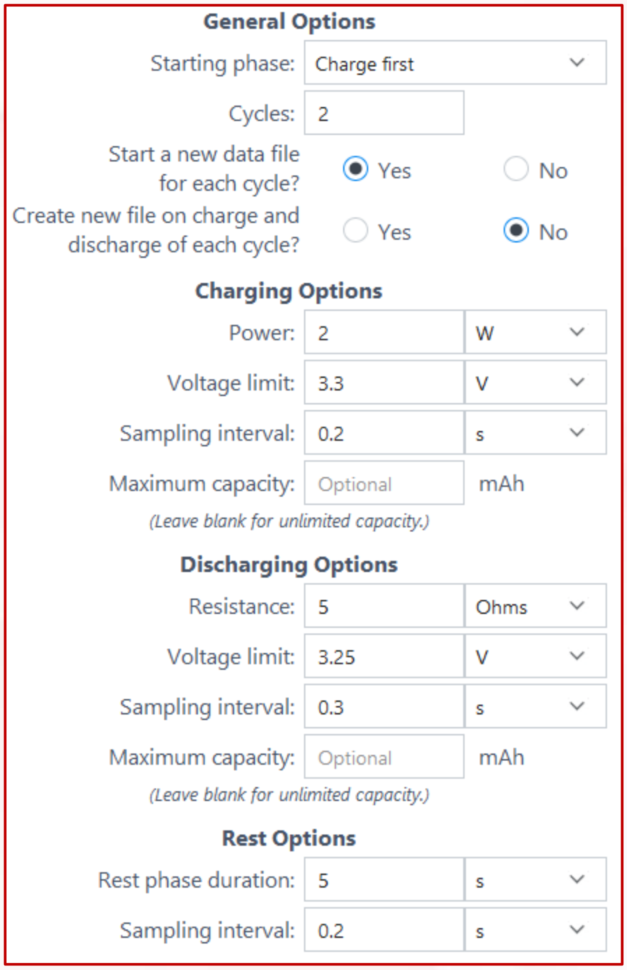
General options

Starting phase: Select whether the cycle begins by charging or discharging.

Cycles: The number of times a user wants to run the charging/discharging cycles before the experiment ends.

Start a new data file for each cycle?: Separate data collection per cycle. See general notes.

Create new file on charge and discharge of each cycle?: Separate charge and discharge data. See general notes.



Charging Options

Power: The Squidstat will charge the electrochemical device at this power.

Voltage limit: The Squidstat will charge the electrochemical device until this voltage is reached.

Sampling interval: The time difference between two consecutive data points during the charge phase.

Maximum capacity: The charging phase will end if the capacity $(i \times time)$ is higher than this value.

Discharging Options

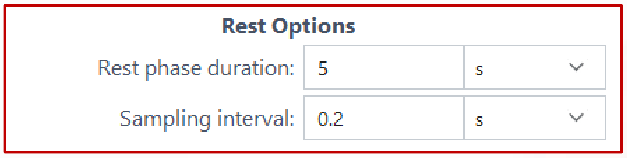
Resistance: The Squidstat will discharge the electrochemical device at this load.

Voltage limit: The Squidstat will discharge the electrochemical device until this voltage is reached.

Sampling interval: The time difference between two consecutive data points during the discharge phase.

Maximum capacity: The discharging phase will end if the capacity $(i \times time)$ is higher than this value.

Rest Options



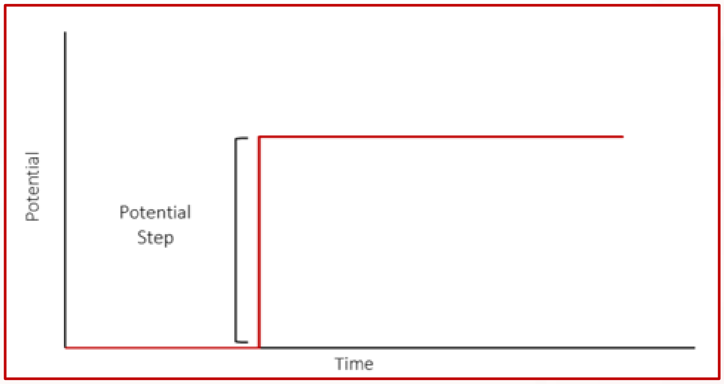
Duration: Time spent in open circuit potential mode after a charge/discharge cycle.

Sampling interval: The time difference between two consecutive data points during the rest period.

9.7 Chronoamperometry – Pre-Built Experiment

Constant Potential / Constant Potential (Advanced) - Method Tile

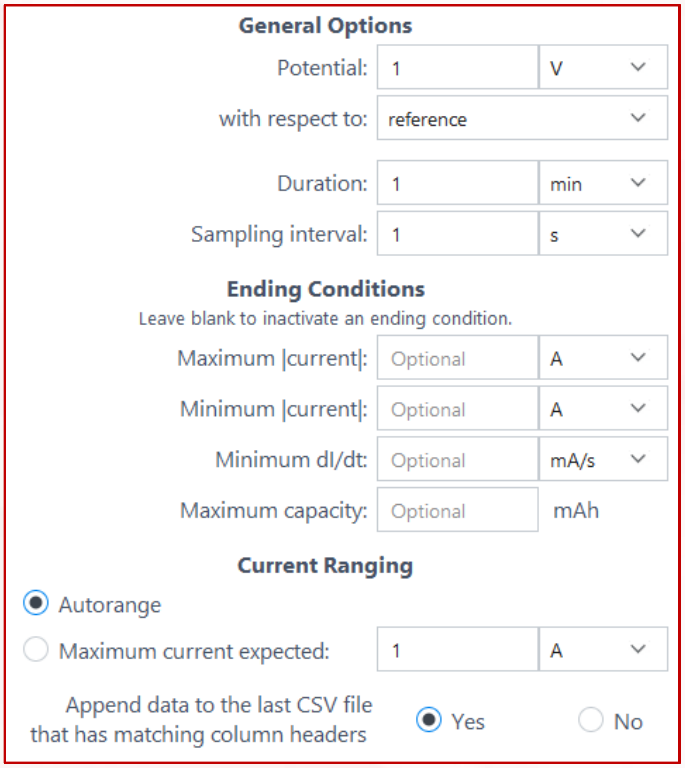
In this method, a constant voltage is applied for a specified duration and the current response is measured. The graphic to the right shows the potential waveform applied to the working electrode.



General Options

Potential: Constant applied voltage setpoint. Can be positive or negative.

With respect to: Reference or Open Circuit. See general notes.



Duration: The length of time that the potential will be applied.

Sampling Interval: The time difference between two consecutive data points.

Ending Conditions (Advanced Method Tile and Prebuilt Only)

Maximum |current|: Experiment will end if current goes above this absolute threshold.

Minimum |current|: Experiment will end if current goes below this absolute threshold.

Minimum dI/dt: Experiment will end if the change in current is below this threshold.

Maximum capacity: Experiment will end if the capacity $(i \times time)$ exceeds this value.

Current Ranging

Autorange: Allow potentiostat to choose current range. See general notes.

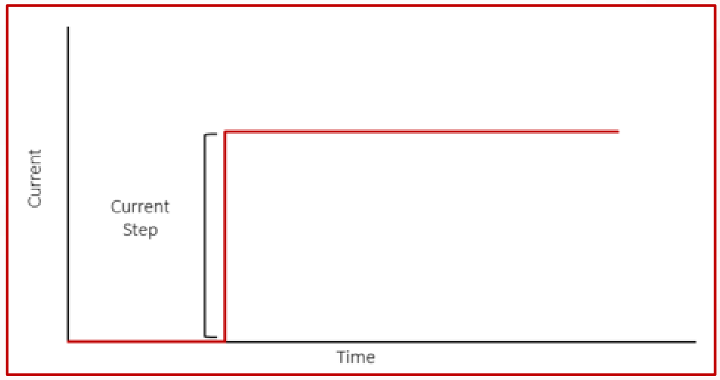
Maximum current expected: Set a fixed current range. See general notes.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.8 Chronopotentiometry - Pre-Built Experiment

Constant Current / Constant Current Advanced - Method Tile

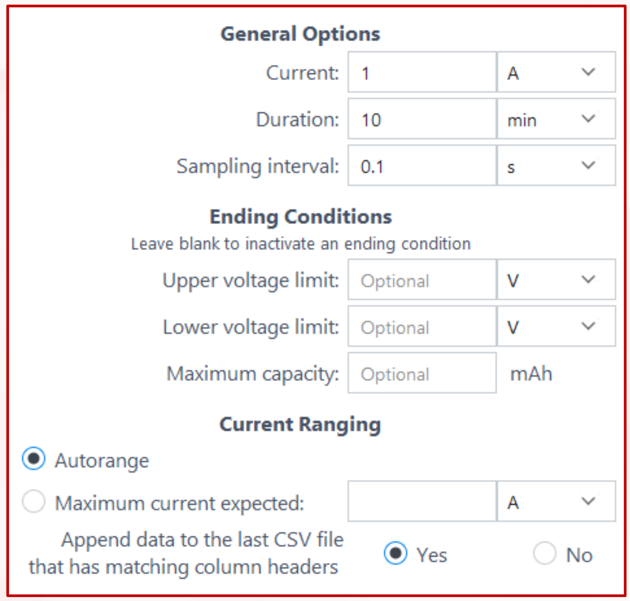
This method runs chronopotentiometry/constant current experiments. In this method, a constant current is applied for a specified duration while recording the potential at the working electrode. The graphic below shows the current waveform applied to the working electrode.



Current: The constant current applied. Can be a positive or a negative value.

Duration: The length of time that the current will be applied.

Sampling Interval: The time difference between two consecutive data points.



Constant Current (Advanced) – Method Tile

Ending Conditions

Upper voltage limit: Experiment will end if current goes above this threshold.

Lower voltage limit: Experiment will end if current goes below this threshold.

Maximum capacity: Experiment will end if the capacity $(i \times time)$ exceeds this value.

Current Ranging

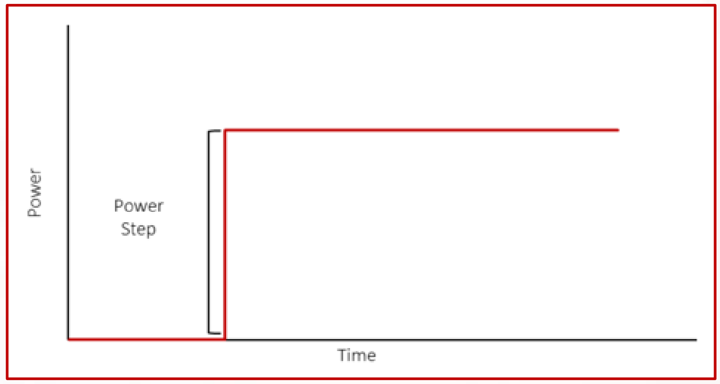
Autorange: Allow potentiostat to choose current range. See general note.

Maximum current expected: Set a fixed current range. See general note.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.9 Constant Power – Pre-Built Experiment and Method Tile

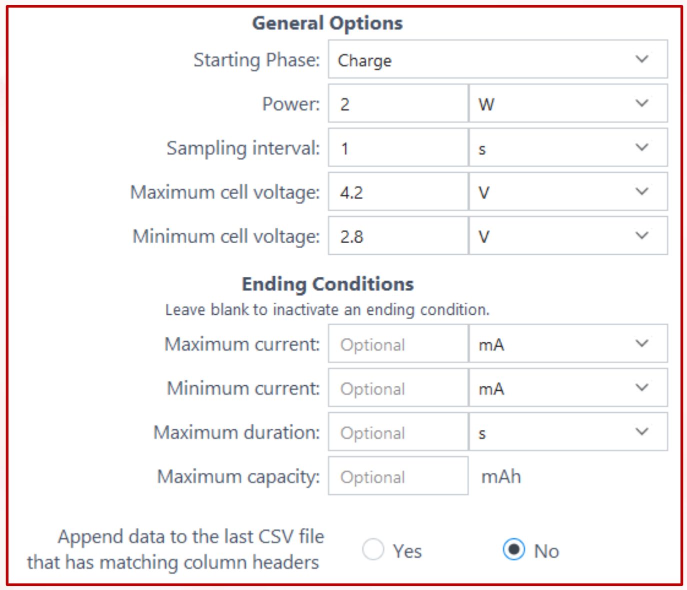
This method charges or discharges an electrochemical device (eg. Battery, Fuel Cells, Capacitors) under a constant power load. The graphic to the right shows a profile of power (P) during a constant power charge or discharge step.



Starting Phase: Select whether the cycle is to begin by charging or discharging.

Power: The Squidstat charges or discharges an electrochemical device at this power.

Sampling interval: The time or voltage difference between two consecutive data points for a given experiment.



Maximum cell voltage: Experiment will end if the cell voltage becomes higher than this value.

Minimum cell voltage: Experiment will end if the cell voltage becomes lower than this value.

Optional Ending Conditions

Maximum current: Experiment will end if the current rises above the specified value.

Minimum current: Experiment will end if the current dips below the specified value.

Maximum duration: The experiment will end at this specified duration time if no other ending conditions are met first.

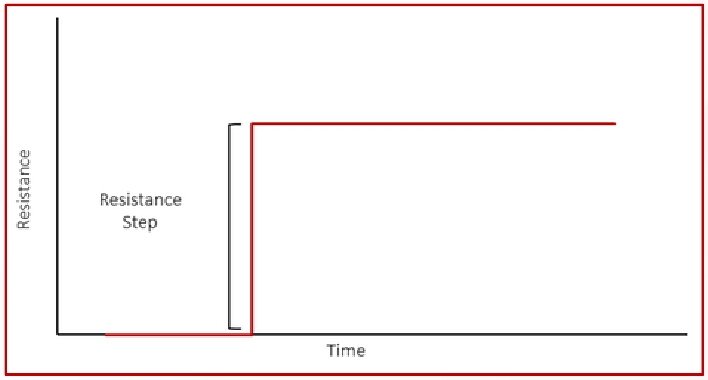
Maximum capacity: The experiment will end if the capacity $(i \times time)$ is higher than this value.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

Maximum and minimum current end conditions can only be used by the Squidstat Cycler Base Model and the Customized Squidstat Cycler!

9.10 Constant Resistance – Pre-Built Experiment and Method Tile

This method simulates a constant resistance load. The graphic to the right gives the waveform of applied resistance.

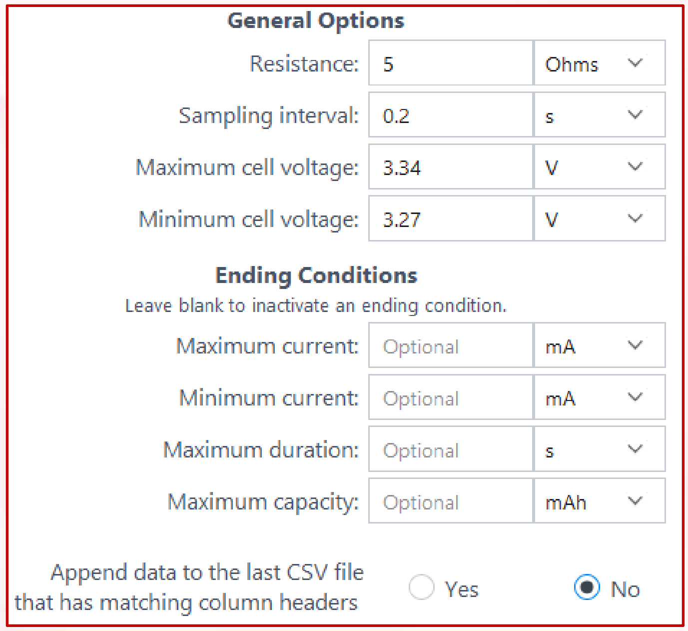


Resistance: Value of resistance to be applied.

Sampling interval: The time or voltage difference between two consecutive data points for a given experiment.

Maximum cell voltage: Experiment will end if the cell voltage becomes higher than this value.

Minimum cell voltage: Experiment will end if the cell voltage becomes lower than this value.



<u>**Optional Ending Conditions**</u>

Maximum current: Experiment will end if the current rises above the specified value.

Minimum current: Experiment will end if the current dips below the specified value.

Maximum duration: The experiment will end at this specified duration time if no other ending conditions are met first.

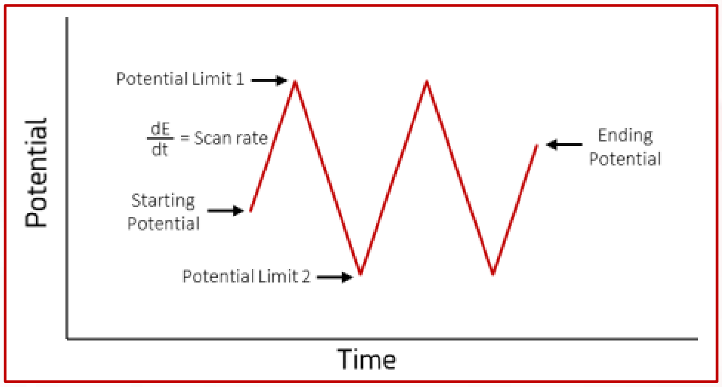
Maximum capacity: The experiment will end if the capacity ($i \times time$) is higher than this value.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

Maximum and minimum current end conditions can only be used by the Squidstat Cycler Base Model and the Customized Squidstat Cycler!

9.11 Cyclic Voltammetry – Pre-Built Experiment and Method Tile

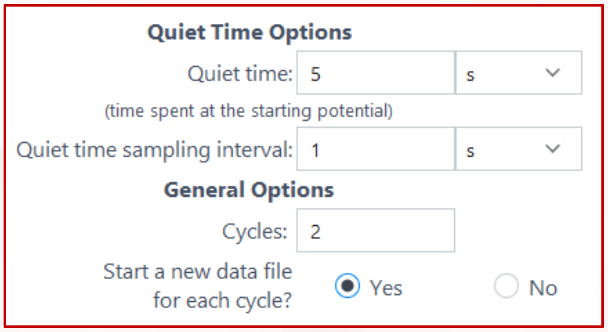
This method runs a cyclic voltammetry (CV) experiment. The potential of the working electrode is cycled between two potential limits or vertices at a fixed rate with respect to time and the current response is recorded. The graphic to the right shows the waveform of potential applied to the working electrode for CV.



Quiet Time Options

Quiet time: Time spent at the starting potential before the experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.



General Options

Cycles: The number of cycles to be performed. The cycling scheme is as follows, where "n" is the number of cycles:

$$Starting\ potential \rightarrow (Scan\ limit\ 1 \rightarrow Scan\ limit\ 2)_n \rightarrow Ending\ potential$$

Start a new data file for each cycle?: Separate data collection per cycle. See general note.

Scanning Options

Starting potential: This potential will be applied before scanning starts. This is also the starting point for the scan.

With respect to: Reference/OCP. See general note.

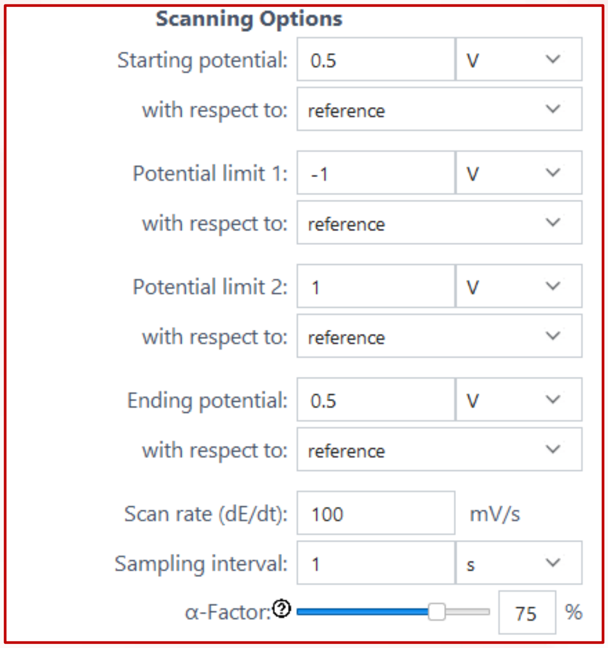
Potential Limit 1: The first potential limit which the potentiostat will scan too from the starting potential.

Potential Limit 2: The second potential limit which the potentiostat will scan too from Potential Limit 1.

Ending potential: The scan will end at this potential.

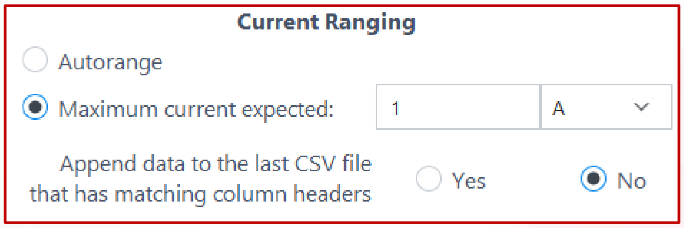
Scan rate: The rate of change of potential with respect to time.

Sampling interval: The time or voltage difference between two consecutive data points.



α-Factor: The percentage of data averaged during each sampling interval. See general notes.

Current Ranging



Autorange: Allow potentiostat to choose current range. See general note.

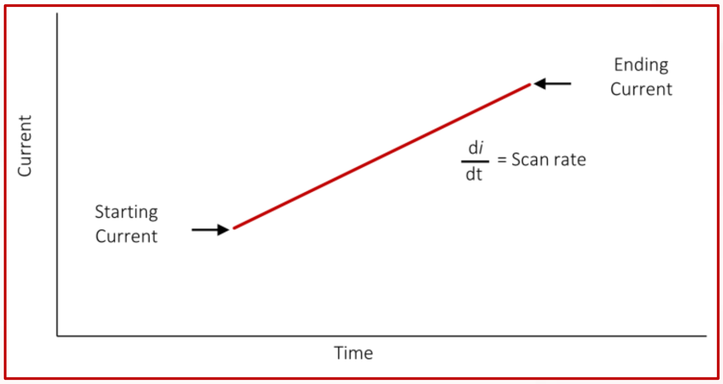
Maximum current expected: Set a fixed current range. See general note.

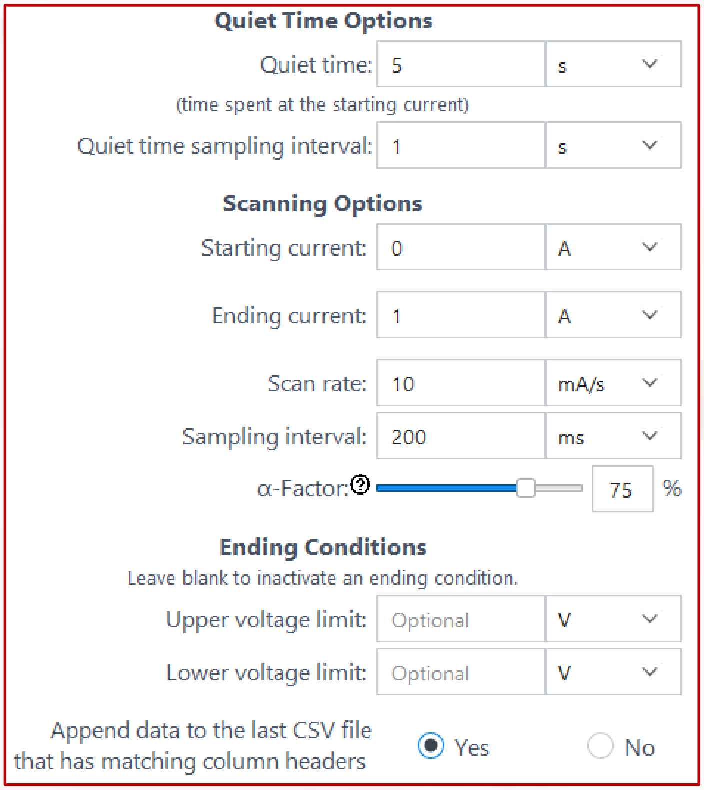
Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.12 Linear Sweep Amperometry – Pre-Built Experiment

DC Current Linear Sweep – Method Tile

This method runs linear current sweep voltammetry (LSV) and linear polarization. This is particularly useful for corrosion researchers wanting to create a Tafel plot. In this step, potential is scanned from a starting potential to the ending potential at a fixed scan rate. The graphic to the right shows the waveform of potential applied to the working electrode for a DC potential linear sweep.





Quiet Time Options

Quiet time: Time spent at the starting potential before the experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.

Scanning Options

Starting current: The scan starts at this current.

Ending current: The scan ends at this current.

Scan rate: The rate of change of current with respect to time.

Sampling interval: The time or current difference between two consecutive data points.

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

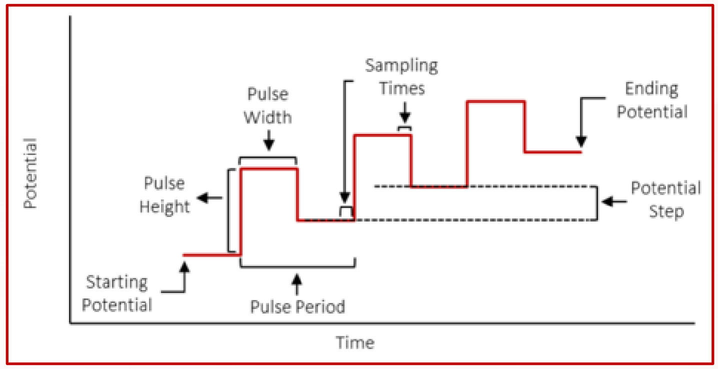
Ending Conditions

Upper Voltage Limit: Experiment will end if the voltage response becomes greater than this value.

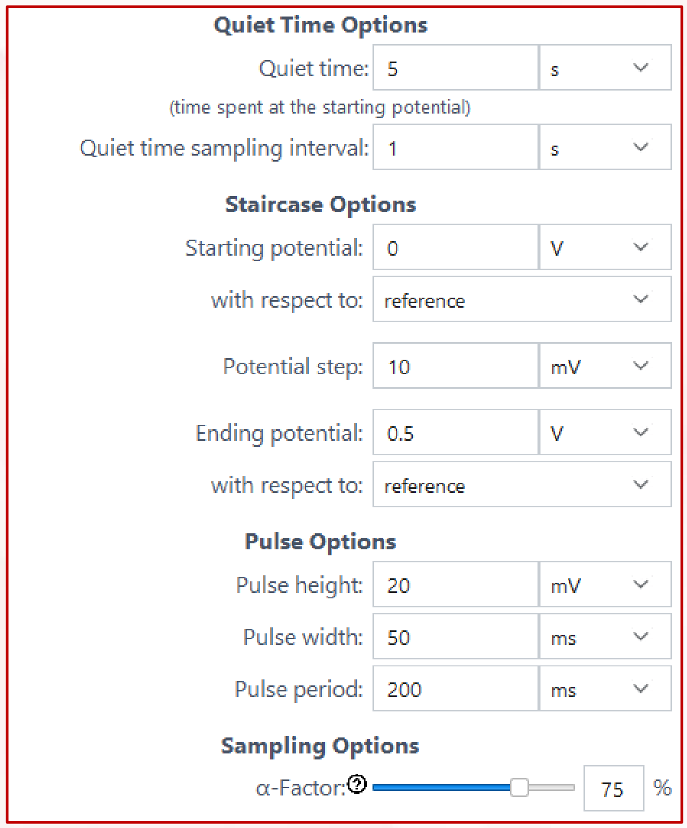
Lower voltage limit: Experiment will end if the voltage response becomes lower than this value.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.13 Differential Pulse Voltammetry - Pre-Built Experiment and Method Tile



This method will run a differential pulse voltammetry (DPV) experiment where pulses of potentials are superimposed on a potential staircase. Each potential step and pulse height retain a constant value. The current response is sampled at two points, one at the end of the pulse width $[i(\tau_1)]$ and another immediately before the pulse $[i(\tau_2)]$. The difference in current response sampled at these two points is plotted as a difference $\delta i = [i(\tau_1) - i(\tau_2)]$ versus the baseline potential. The graphic above shows the waveform of potential applied to the working electrode for DPV.



Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.

Staircase Options

Starting potential: The Squidstat will apply this voltage at the beginning of the scan.

With respect to: Reference/OCP. See general note.

Potential step: This is the difference between consecutive return potentials after the pulses.

Ending potential: The experiment will end when this potential is reached.

Pulse Options

Pulse height: The magnitude of each pulse.

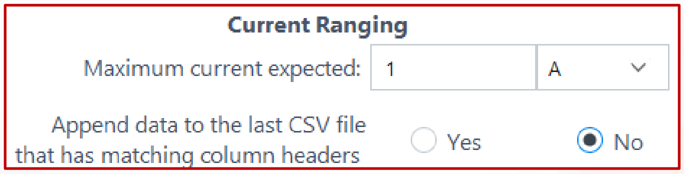
Pulse width. The potential pulse will be applied for this amount of time.

Pulse period: The duration between two consecutive pulses.

Sampling Options

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

Current Ranging

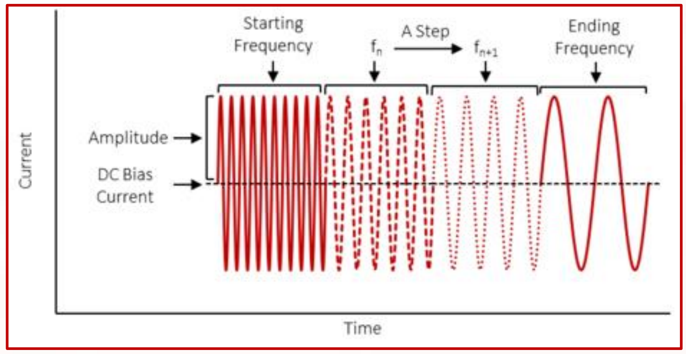


Maximum current expected: Set a fixed current range. See general note.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.14 Galvanostatic EIS - Pre-Built Experiment and Method Tile

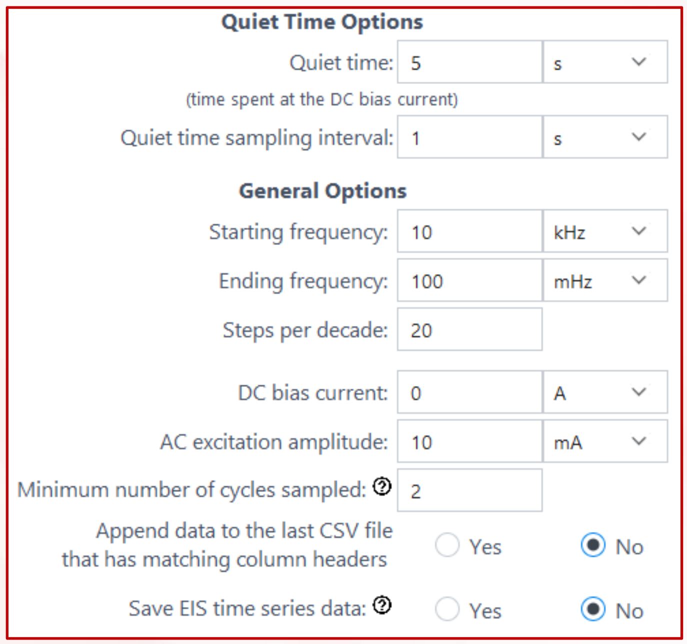
This method applies a galvanostatic AC sine wave and measures the resulting AC voltage response across a range of frequencies. Complex impedance data is reported at each frequency, offering insights into the electrochemical properties, such as resistance and capacitance, of the material. The graphic to the right shows the current waveform applied during gEIS.



Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.



General Options

Starting frequency: The frequency of the galvanostatic sine wave that will be applied first.

Ending frequency: The frequency of the galvanostatic sine wave that will be applied last.

Steps per decade: The number of galvanostatic sine waves applied for every decade change in frequency. The sine wave frequency will change logarithmically.

DC bias current: This current will be applied as a baseline current.

AC excitation amplitude: This is the amplitude of the galvanostatic sine waves.

Minimum number of cycles sampled: The minimum number of periods of applied sinusoidal current at each frequency. Increase this number if low frequency data is noisy.

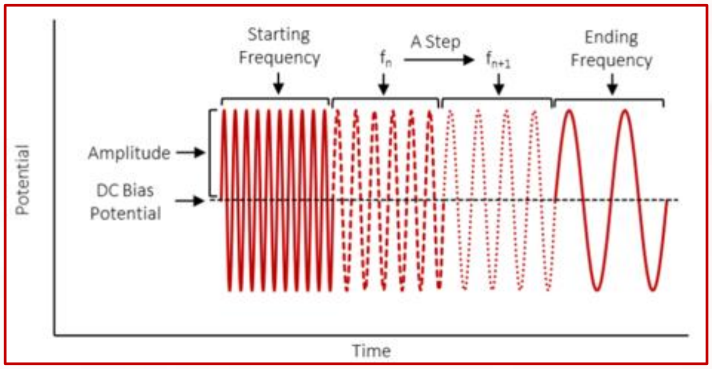
Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

Save EIS time series data: If enabled, the EIS time series data will be recorded in individual data files, one for each frequency. Enabling this option will result in large data files.

Save EIS time series data can only be used by the Squidstat Cycler Base Model and the Customized Squidstat Cycler!

9.15 Potentiostatic EIS - Pre-Built Experiment and Method Tile

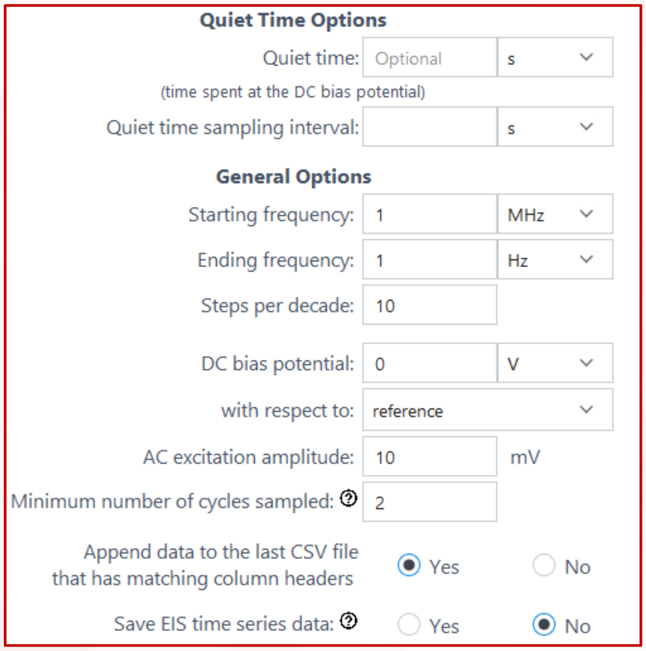
This method applies a potentiostatic AC sine wave and measures the resulting AC voltage response across a range of frequencies. Complex impedance data is reported at each frequency, offering insights into the electrochemical properties, such as resistance and capacitance, of the material. The graphic to the right shows the current waveform applied during pEIS.



Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time Sampling interval: The time or voltage difference between two consecutive data points for a given experiment.



General Options

Starting frequency: The frequency of the potentiostatic sine wave that will be applied first.

Ending frequency: The frequency of the potentiostatic sine wave that will be applied last.

Steps per decade: The number of logarithmically spaced sine waves applied for every decade change in frequency.

DC bias potential: This potential will be applied as the baseline potential with respect to Reference or OCP. See general notes.

AC excitation amplitude: This is the amplitude of the potentiostatic sine waves.

Minimum number of cycles sampled: The minimum number of periods of applied sinusoidal current at each frequency. Increase this number if low frequency data is noisy.

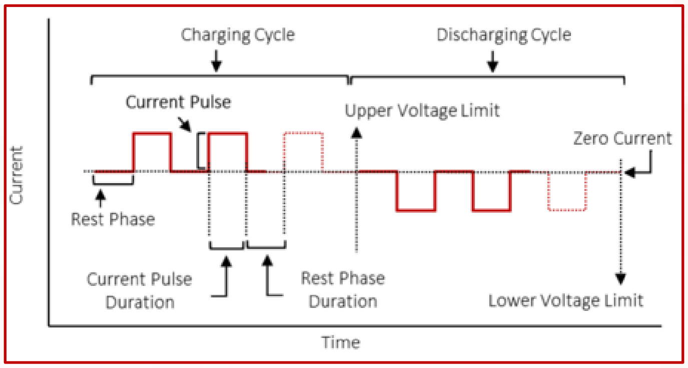
Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles. See general notes.

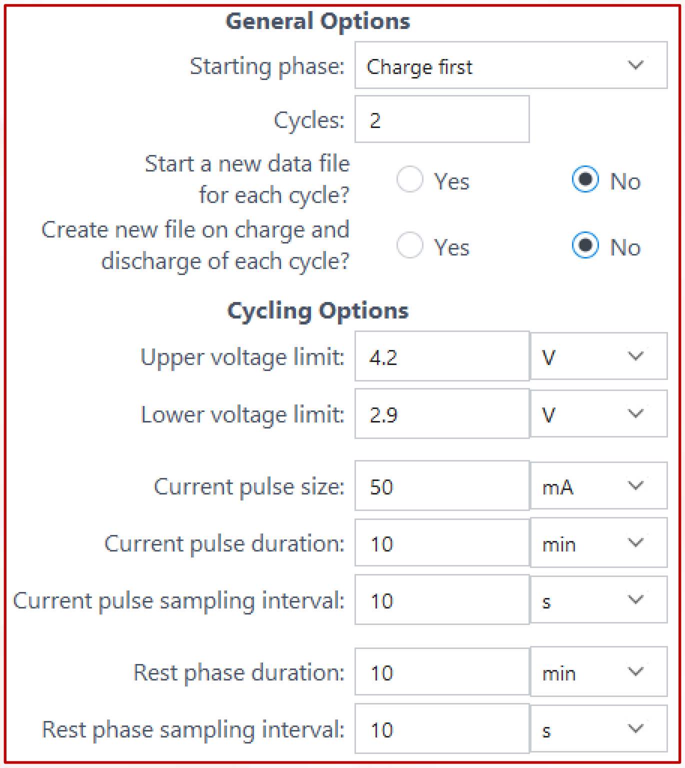
Save EIS time series data: If enabled, the EIS time series data will be recorded in individual data files, one for each frequency. Enabling this option will result in large data files.

Save EIS time series data can only be used by the Squidstat Cycler Base Model and the Customized Squidstat Cycler!

9.16 Galvanostatic Intermittent Titration Technique (GITT) - Pre-Built Experiment

This experiment charges and discharges a device under test. Both charging and discharging are completed by applying a series of current pulses of fixed amplitude and duration. After each current pulse, the cell undergoes a rest phase. The graphic to the right shows a waveform of current pulses applied to the working electrode during charging and discharging cycles.





General Options

Starting phase: Select whether the cycle begins by charging or discharging.

Cycles: The number of times that the device under test will be charged and discharged.

Start a new data file for each cycle?: Separate data collection per cycle. See general notes.

Create new file on charge and discharge of each cycle?: Separate charge and discharge data. See general notes.

Cycling Options

Upper voltage limit: The specified charging current is applied until this voltage is reached.

Lower voltage limit: The specified discharging current is applied until this voltage is reached.

Current pulse size: This is the magnitude of current applied during charging or discharging.

Current pulse duration: The Squidstat will apply the current pulse for this amount of time.

Current pulse sampling interval: The time difference between two consecutive data points during the current pulse duration.

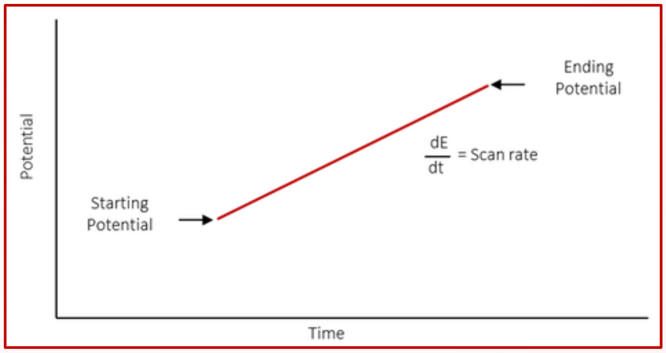
Rest phase duration: The amount of time between the end of a pulse and the beginning of the following pulse. During this time, Squidstat is in open circuit mode.

Rest phase sampling interval: The time difference between two consecutive data points during the rest phase duration.

9.17 Linear Sweep Voltammetry - Pre-Built Experiment

DC Potential Linear Sweep - Method Tile

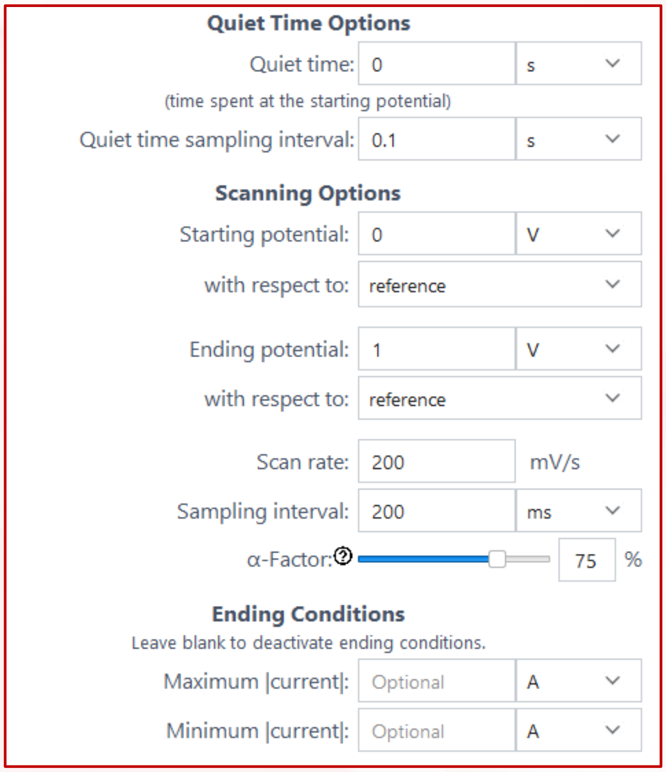
This method performs linear sweep voltammetry (LSV) and linear polarization, particularly useful for corrosion researchers creating Tafel plots. The potential is scanned from a starting potential to an ending potential at a fixed scan rate, as shown in the waveform graphic to the right.



Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.



Scanning Options

Starting potential: The scan starts from this potential.

With respect to: Reference/OCP. See general note.

Ending potential: The scan ends at this potential.

Scan rate (dE/dt): The rate of change of potential with respect to time.

Sampling interval: The time or voltage difference between two consecutive data points.

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

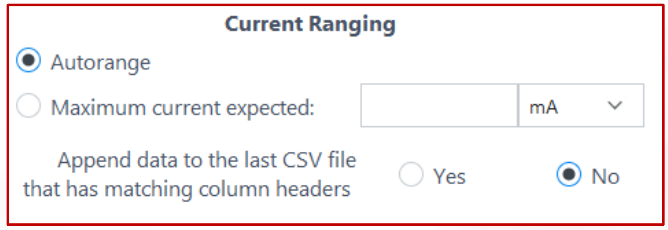
Ending Conditions

Maximum |current|: Experiment will end if the current response becomes greater than this value.

Minimum |current|: Experiment will end if the current response becomes lower than this value.

α-Factor: The ratio of the data sampling period to the step period, defining the portion of recorded data used for averaging. See general note.

Current ranging

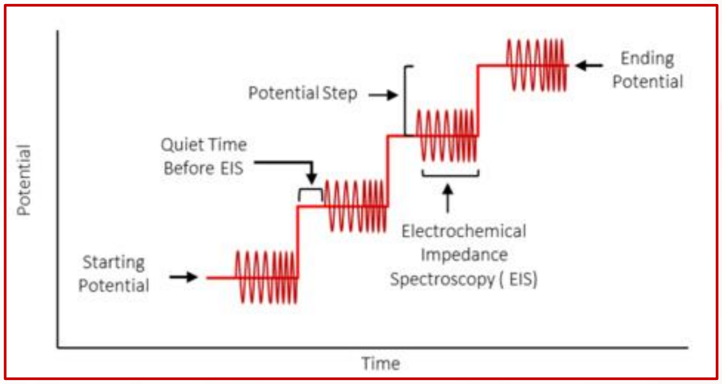


Autorange: Allow potentiostat to choose current range. See general note.

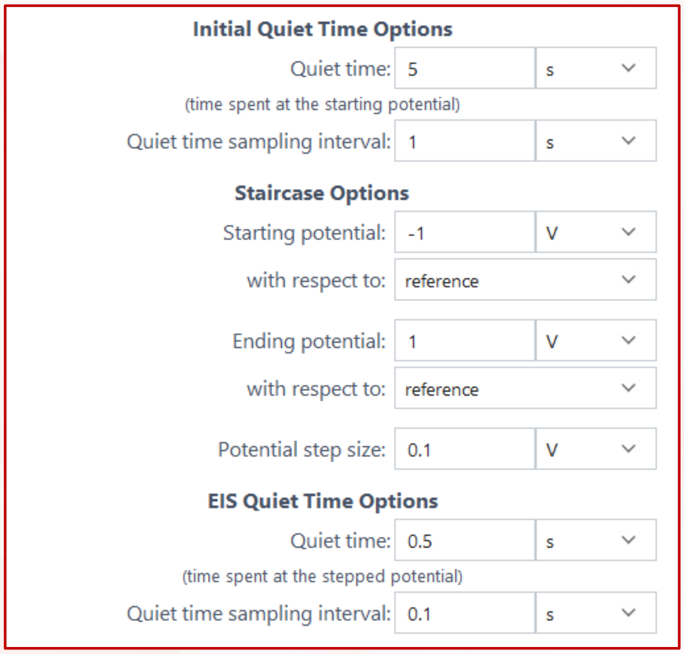
Maximum current expected: Set a fixed current range. See general note.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.18 Mott-Schottky (Staircase Potentiostatic EIS) - Pre-Built Experiment and Method Tile



This experiment sweeps the potential of the working electrode from the starting potential to the ending potential in fixed steps. Each potential step consists of a quiet time and a potentiostatic sinusoidal wave of fixed or variable frequency and fixed amplitude. The DC bias of the sinusoidal wave overlaps the potential step. A common application of this technique is to determine properties of semiconductors such as flatband potential, doping density, and Helmholtz capacitance. The graphic to the right shows the waveform of potential applied to the working electrode.



Initial Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.

Staircase Options

Starting potential: Potential applied at beginning of scan.

With respect to: Reference/OCP. See general note.

Ending potential. The experiment will stop after the EIS scan at this potential is completed.

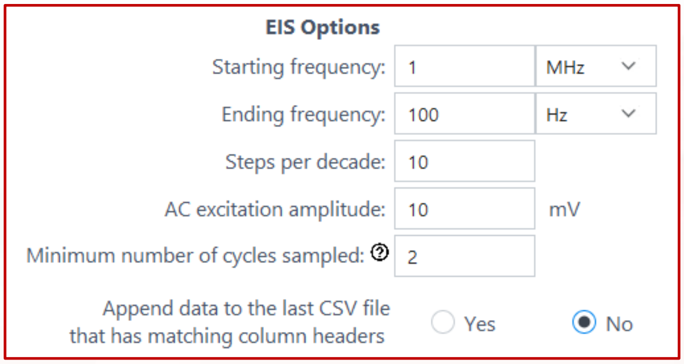
Potential step size: This is the magnitude of the potential step. The direction of the scan is determined by the starting and ending potentials irrespective of the sign of the potential step size.

EIS Quiet Time Options

Quiet time: Time spent at the starting potential following each potential step in the staircase.

Quiet time sampling interval: The time or voltage difference between two consecutive data points during quiet time.

EIS Options



Starting frequency: The frequency of the sine wave that will be applied first.

Ending frequency: The frequency of the sine wave that will be applied last. To run single-frequency Mott-Schottky, set the starting and ending frequency to the same value.

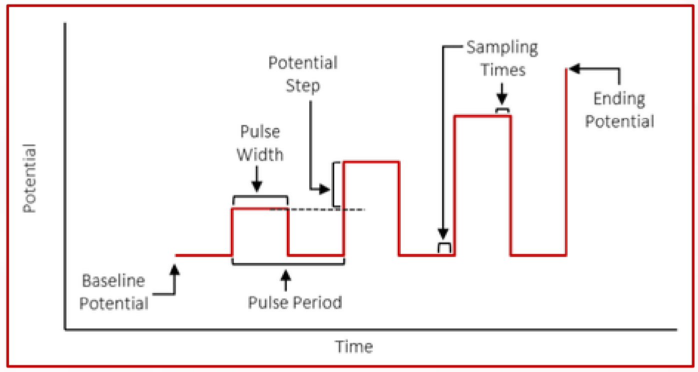
Steps per decade: The number of potentiostatic sine waves applied for every decade change in frequency. The frequency of the sine waves will change logarithmically.

AC excitation amplitude: This is the amplitude of the potentiostatic sine waves.

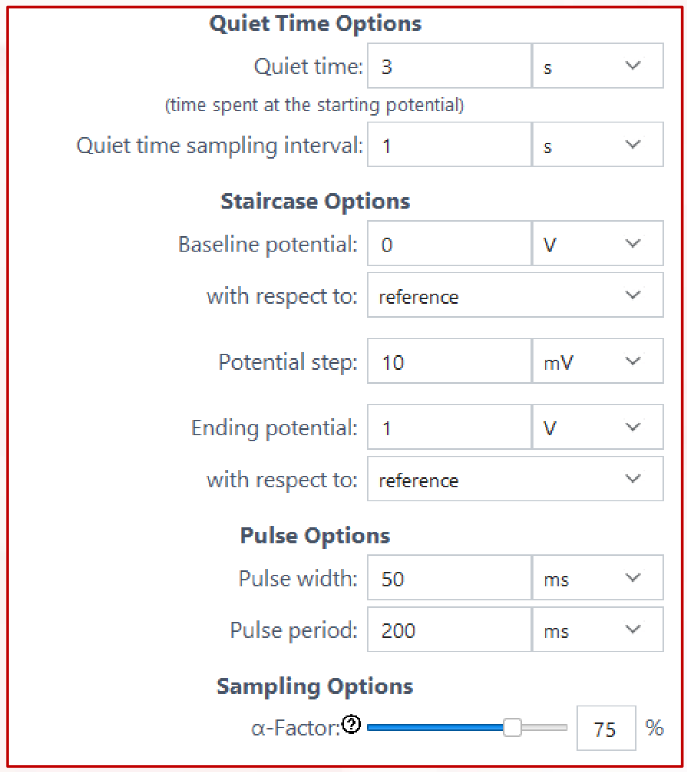
Minimum number of cycles sampled: The minimum number of periods of applied sinusoidal current at each frequency. Increase this number if low frequency data is noisy.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.19 Normal Pulse Voltammetry - Pre-Built Experiment and Method Tile



In this experiment, potential pulses are superimposed over a constant set potential. The baseline potential of each pulse does not change. The height of each subsequent pulse changes at a fixed interval. The pulse width and the pulse period are the same for each pulse. The current response is sampled at two points, one at the end of the pulse width $i(\tau_1)$ and another immediately before the pulse $i(\tau_2)$. The difference in current response sampled at these two points is plotted as a difference $\delta i = [i(\tau_1) - i(\tau_2)]$ against the potential of the pulse width. The graphic shows the waveform of potential applied to the working electrode.



Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.

Staircase Options

Baseline potential: The Squidstat will apply this value as a base potential.

With respect to: Reference/OCP See general note.

Potential step: This is the potential difference between consecutive potential pulses.

Ending potential. The experiment will end when this potential is reached.

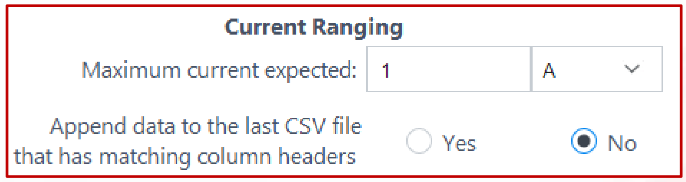
Sampling Options

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

Pulse Options

Pulse width. The potential pulse will be applied for this amount of time before returning to the base potential.

Pulse period: The amount of time between the start of consecutive pulses. This time includes the pulse width and the time at the base potential before the next pulse.



Current Ranging

Maximum current expected: Set a fixed current range. See general note.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

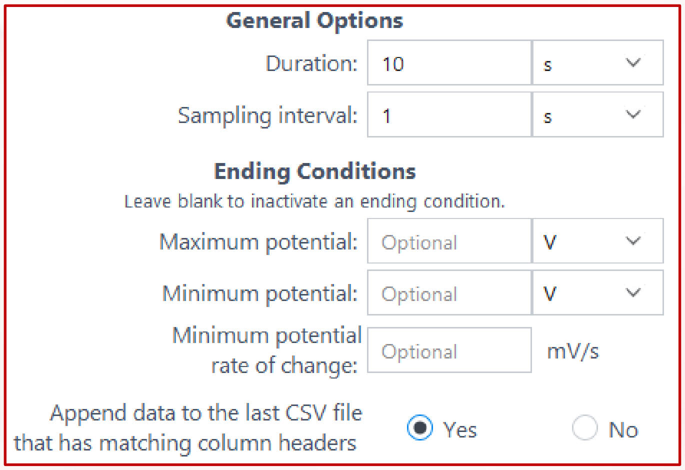
9.20 Open Circuit Potential - Pre-Built Experiment and Method Tile

This experiment measures the voltage at the working electrode relative to a reference electrode without applying any external current or voltage for a specified duration. The system records the natural electrochemical potential over time, providing insights into the stability and behavior of the electrode material in its native state.

General Options

Duration: The open circuit potential will be recorded over this length of time.

Sampling interval: The time difference between two consecutive data points.



Ending Conditions

Maximum potential: The experiment will end if the potential is higher than this value.

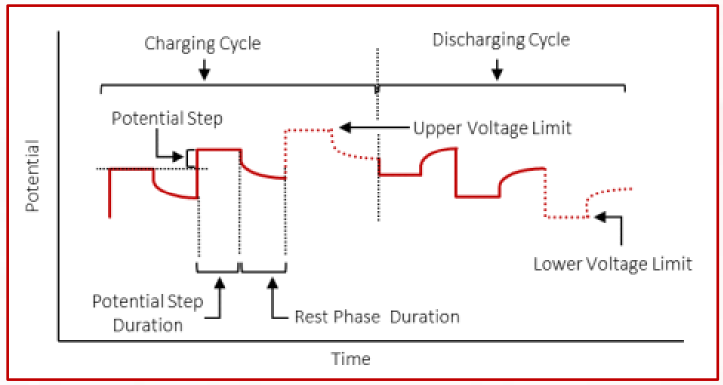
Minimum potential: The experiment will end if the potential is lower than this value.

Minimum potential rate of change: The experiment will end if the rate of change of potential is lower than this value.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.21 Potentiostatic Intermittent Titration Technique (PITT) - Pre-Built Experiment

This experiment charges and discharges a device under test (DUT). Both charging and discharging are done by stepping the potential of the cell by a fixed value for a fixed duration. Each potential step is followed by a relaxation period when the potentiostat is in open circuit mode. Therefore, the current response is obtained only from the pulse steps. The graphic to the right shows the waveform of potential applied to the working electrode during charging and discharging cycles.



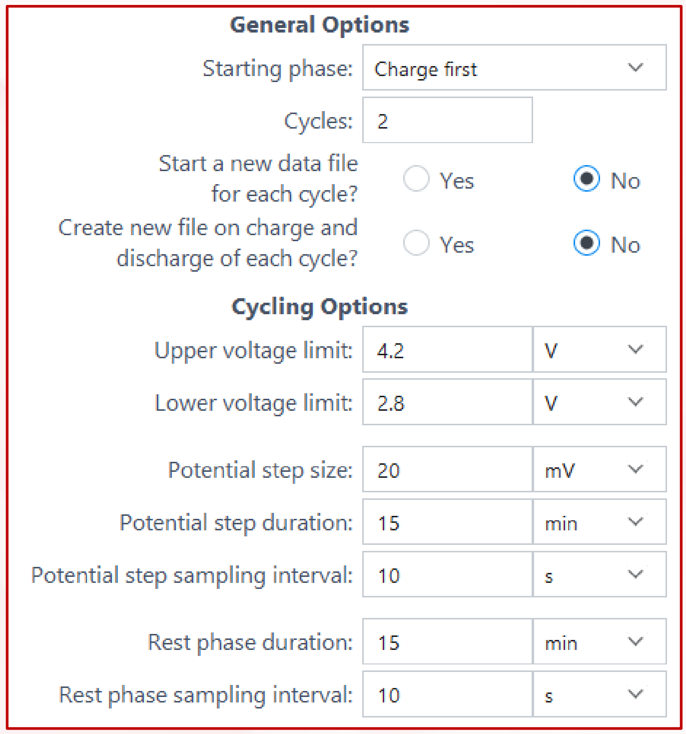
General Options

Starting phase: Select whether the cycle is to begin by charging or discharging.

Cycles: The number of times that the device under test will be charged and discharged.

Start a new data file for each cycle?: Determines output file structure. See general note

Create new file on charge and discharge of each cycle?: Determines output file structure. See general note.



Cycling Options

Upper voltage limit: The Squidstat will step the potential of the DUT until this voltage is reached during the charge phase.

Lower voltage limit: The Squidstat will step the potential of the DUT until this voltage is reached during the discharge phase.

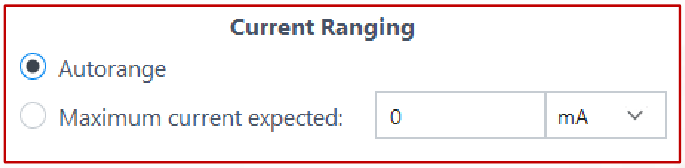
Potential step size: The magnitude of each potential step.

Potential step duration: The Squidstat will apply each potential step for this amount of time.

Potential step sampling interval: The time difference between two consecutive data points during the potential step.

Rest phase duration: The amount of time between the end of a potential step and the beginning of the next potential step. During this time, Squidstat is in open circuit mode.

Rest phase sampling interval: The time difference between two consecutive data points during the rest phase duration.

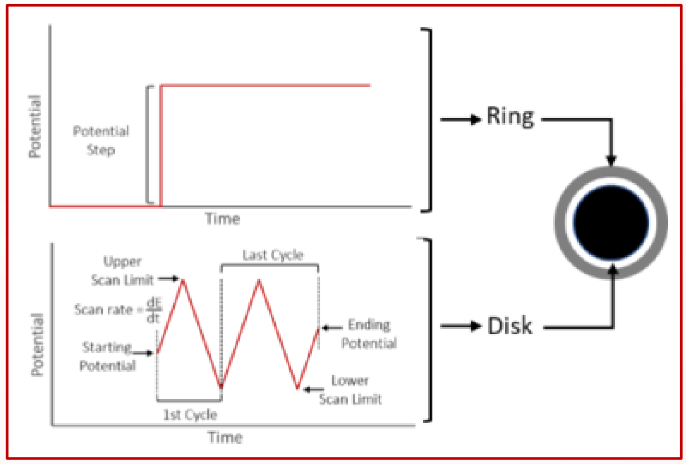


Current Ranging

Autorange: Allow potentiostat to choose current range. See general note.

Maximum current expected: Set a fixed current range. See general note.

9.22 Rotating Ring Disk Electrode (RRDE) - Pre-Built Experiment

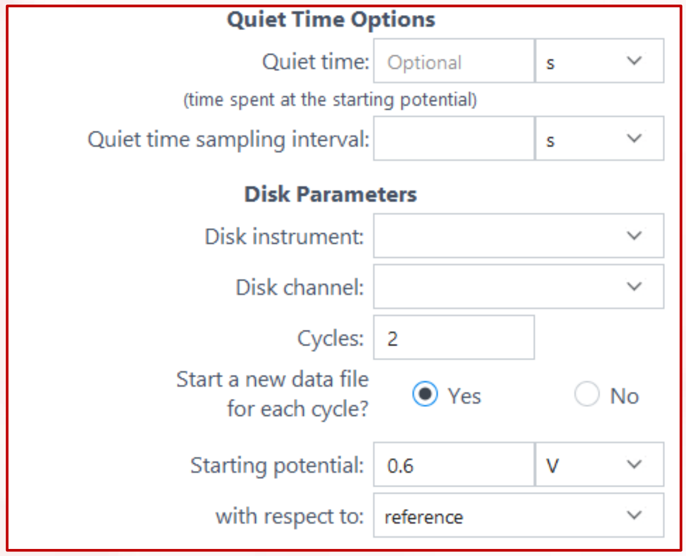


This experiment holds the ring electrode at a fixed voltage with respect to the reference electrode or open circuit. The disk is subjected to a cyclic voltammogram. Current measured at the disk and the ring are plotted against the potential of the disk electrode. The application note Running rotating ring-disk electrode (RRDE) experiments with Squidstat potentiostats is available as well as a startup guide for more information.

Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.



Disk Parameters

Disk instrument: Select which Squidstat will control the disk. Can be a single-channel or multichannel. If a Squidstat Prime is selected, the specific channel controlling the disk will need to be assigned.

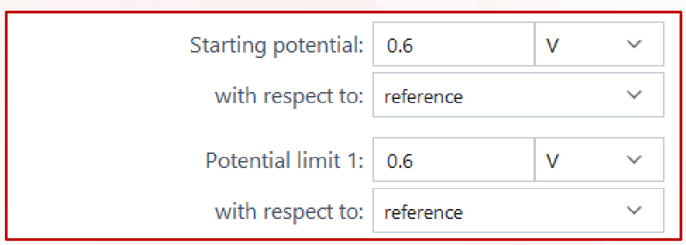
Disk channel: Choose which channel will control the disk. Defaults to channel 1 if a single-channel Squidstat is assigned.

Cycles: The number of cycles to be performed. The cycling scheme is as follows:

$$Starting\ potential \rightarrow (Scan\ limit\ 1 \rightarrow Scan\ limit\ 2)_n \rightarrow Ending\ potential$$

where “n” is the number of cycles.

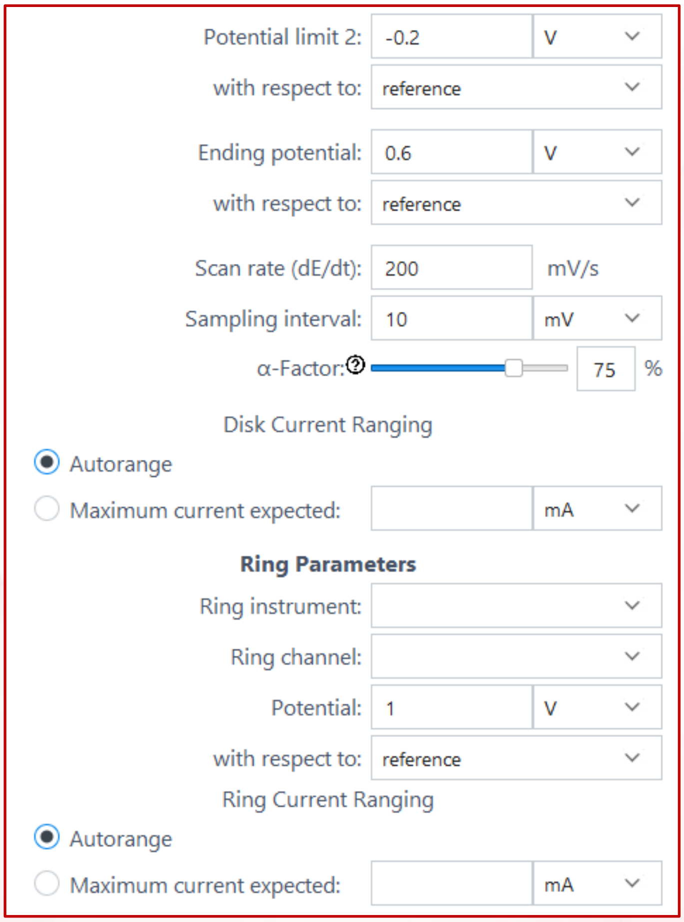
Start new CSV file for each cycle: Split data files by cycle. See general note.



Starting potential: The potential that is applied at the start of the scan. This can be set with respect to the open circuit potential or the reference potential.

With respect to: Reference/OCP. See general note.

Potential Limit 1: The potential limit to which the potentiostat will scan to from the starting potential. This value can be either more positive or more negative than the starting potential.



Potential Limit 2: The second potential limit which the potentiostat will scan to from Potential Limit 1.

Ending potential: The scan will end at this potential.

Scan rate (dE/dt): The rate of change of potential with respect to time.

Sampling interval: The time difference between two consecutive data points.

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

Disk Current Ranging

Autorange: Allow potentiostat to choose current range. See general note.

Maximum current expected: Set a fixed current range. See general note.

Ring Parameters

Ring instrument: Select which Squidstat will control the ring. Can be a single-channel or multichannel. If a Squidstat Prime is selected, the specific channel controlling the disk will need to be assigned.

Ring channel: Choose which channel will control the disk. Defaults to channel 1 if a single-channel Squidstat is assigned.

Potential: Constant potential applied at the ring electrode.

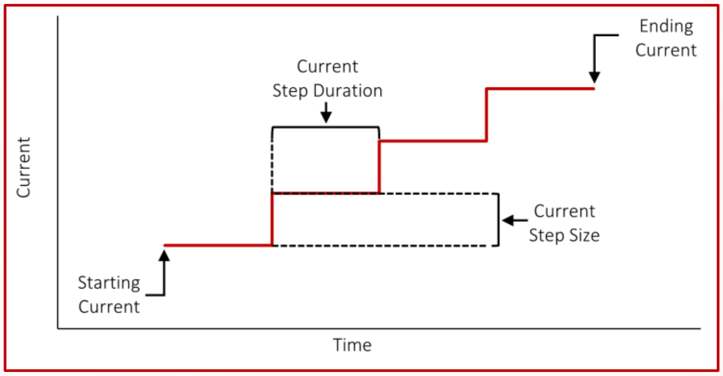
Ring current ranging

Autorange: Allow potentiostat to choose current range. See general note.

Maximum current expected: Set a fixed current range. See general note.

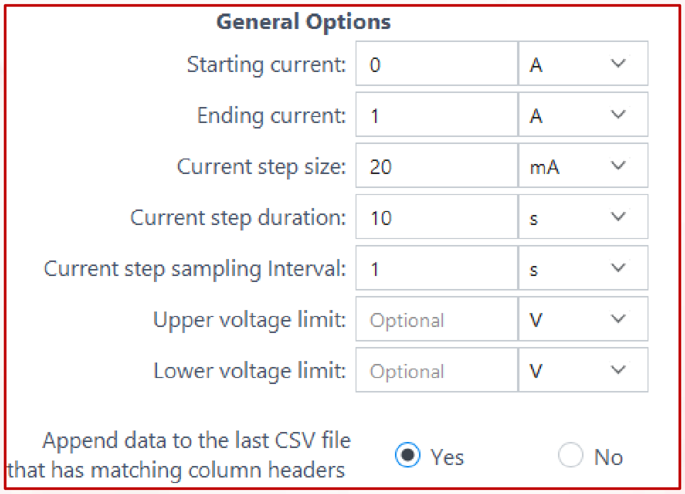
9.23 Stepped Current - Method Tile

This experiment steps the current applied to the working electrode from the starting current to the ending current using a fixed step value and discrete duration for each step. The voltage response at the working electrode is recorded. The graphic to the right shows the current waveform applied to the working electrode over time.



General Options

Starting current: Current applied to the working electrode at the beginning of the scan.



Ending current: The scan will end at this current.

Current step size: The magnitude of the current step. The direction of the scan is determined by the starting and ending currents irrespective of the sign of the current step size.

Current step duration: Time spent at each discrete current step.

Current step sampling interval: The time or voltage difference between two consecutive data points during the experiment.

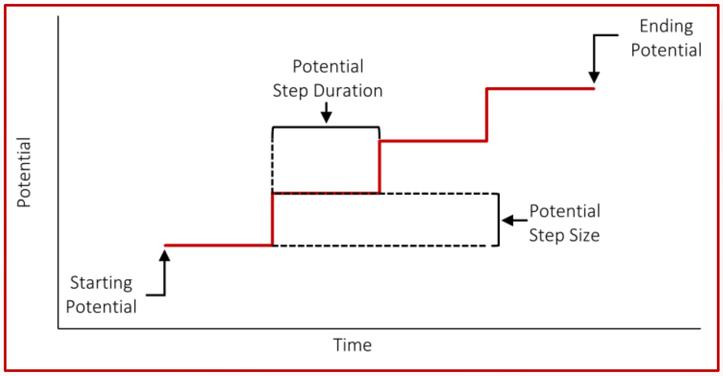
Upper voltage limit: Method tile will end if this voltage value is exceeded.

Lower voltage limit: Method tile will end if the voltage goes below this value.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

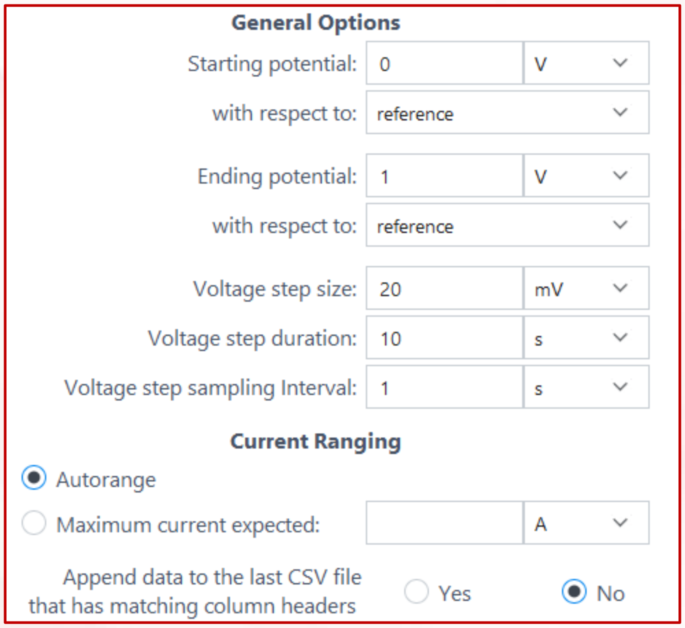
9.24 Stepped Voltage - Method Tile

This experiment steps the voltage applied to the working electrode from the starting potential to the ending potential using a fixed step value and discrete duration for each step. The current response at the working electrode is recorded. The graphic to the right shows the voltage waveform applied to the working electrode over time.



General Options

Starting potential: Voltage applied to the working electrode at the beginning of the scan.



With respect to: Reference/OCP. See general note.

Ending potential: The scan will end at this voltage.

Voltage step size: The magnitude of the voltage step. The direction of the scan is determined by the starting and ending potentials irrespective of the sign of the voltage step size.

Voltage step duration: Time spent at each discrete voltage step.

Voltage step sampling interval: The time difference between two consecutive data points during the experiment.

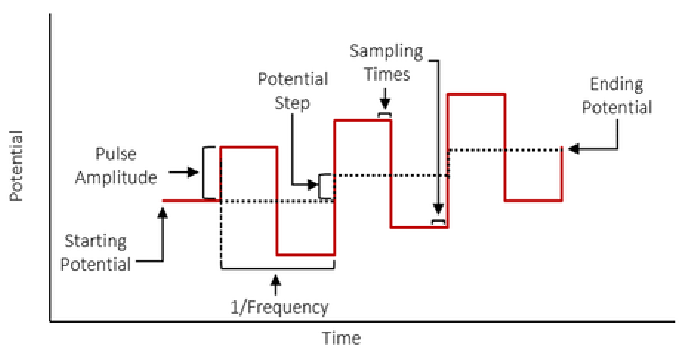
Current Ranging

Autorange: Allow potentiostat to choose current range. See general note.

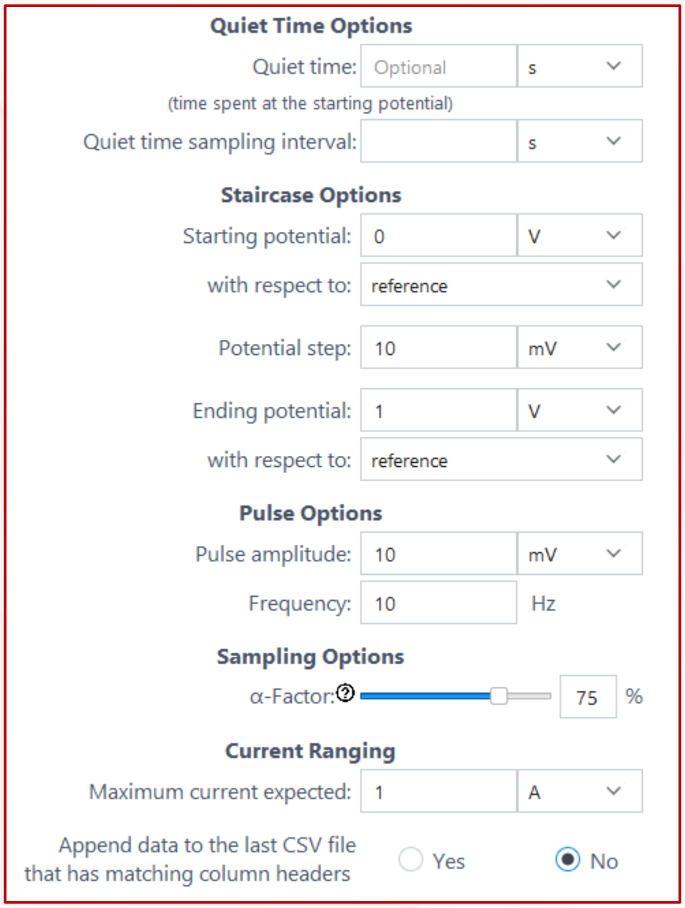
Maximum current expected: Set a fixed current range. See general note.

Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles in the sequence. See general notes.

9.25 Square Wave Voltammetry - Pre-Built Experiment and Method Tile



In this experiment, a series of square pulses are superimposed on a staircase waveform at a defined frequency. Each square pulse consists of a forward pulse and a reverse pulse of equal amplitude but in opposite direction. The current response is sampled at the end of the forward pulse (i_f) and the end of the reverse pulse (i_r). The difference in current at these two points $\left(\Delta i = i_f - i_r\right)$ is plotted against the staircase potential. The graphic shows the waveform of potential applied to the working electrode.



Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during quiet time.

Staircase Options

Starting potential: The Squidstat will apply this potential at the beginning of the scan.

With respect to: Reference/OCP. See general note.

Potential step: The difference between base potentials of consecutive square pulses.

Ending potential. The experiment will end when this potential is reached.

Pulse Options

Pulse amplitude: The magnitude of each pulse.

Frequency. The number of pulses per second.

Sampling Options

α-Factor: The percentage of data averaged during each sampling interval. See general notes.

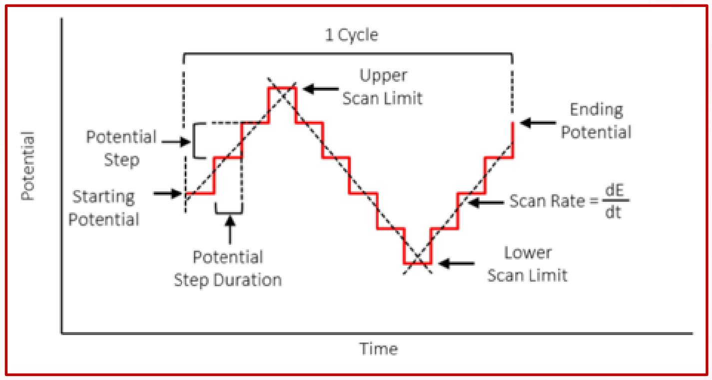
Current ranging

Maximum current expected: Set a fixed current range. See general note.

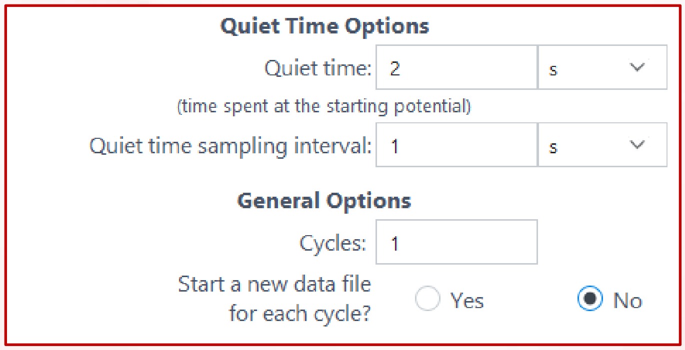
Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles. See general notes.

9.26 Staircase Voltammetry - Pre-Built Experiment

Staircase Potential Voltammetry – Method Tile



This experiment sweeps the potential of the working electrode back and forth between the upper and lower scan limits for a specified number of cycles by changing the potential of the working electrode in steps. The potential step size and potential step duration are fixed throughout the scan. This experiment is similar to Cyclic Voltammetry. However, the goal here is to let the system under test come to a semi-steady state at each potential step. A common application of this method is to obtain performance curves of fuel cell systems. The graphic above shows the waveform of potential applied to the working electrode.



Quiet Time Options

Quiet time: Time spent at the starting potential or starting current before an experiment begins.

Quiet time sampling interval: The time difference between two consecutive data points during the quiet time.

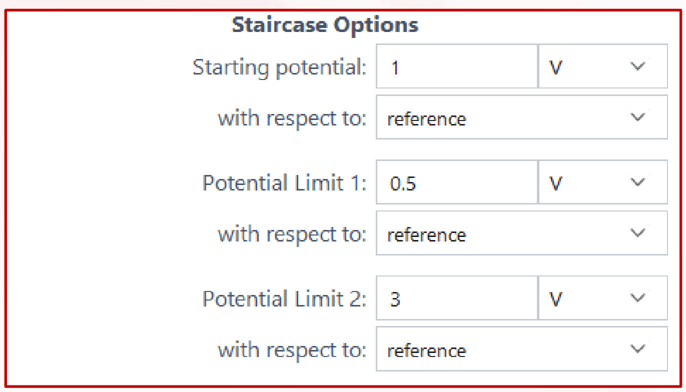
General Options

Cycles: The number of cycles to be performed. The cycling scheme is as follows:

$$Starting\ potential \rightarrow (Scan\ limit\ 1\ \rightarrow\ Scan\ limit\ 2)_n\ \rightarrow Ending\ potential$$

Where "n" is the number of cycles.

Start new CSV file for each cycle: Split data files by cycle. See general note.



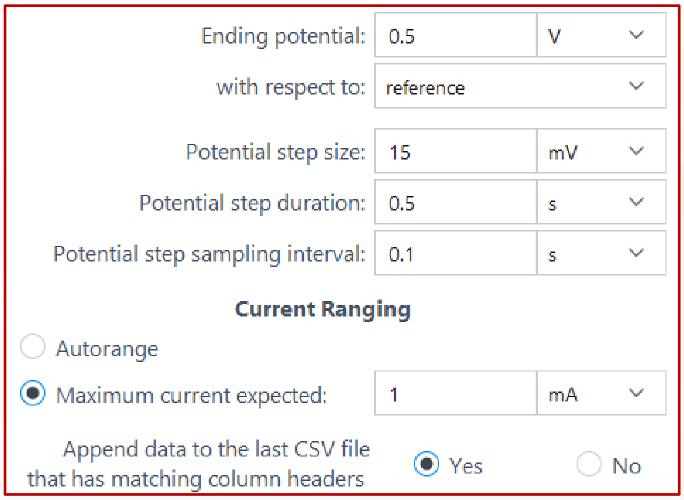
Staircase Options

Starting potential: The Squidstat will apply this potential at the beginning of the scan.

With respect to: Reference/OCP. See general note.

Potential Limit 1: The initial potential limit which the potentiostat will scan too from the starting potential. This value can be either more positive or more negative than the starting potential.

Potential Limit 2: The second potential limit which the potentiostat will scan too from Potential Limit 1.



Ending potential: The scan will end at this potential.

Potential step size: The magnitude of each potential step.

Potential step duration: The Squidstat will apply the potential step for this amount of time.

Potential step sampling interval: The interval between two consecutive data points during a potential step.

<u>Current ranging</u>

Autorange: Allow potentiostat to choose current range. See general note.

Maximum current expected: Set a fixed current range. See general note.

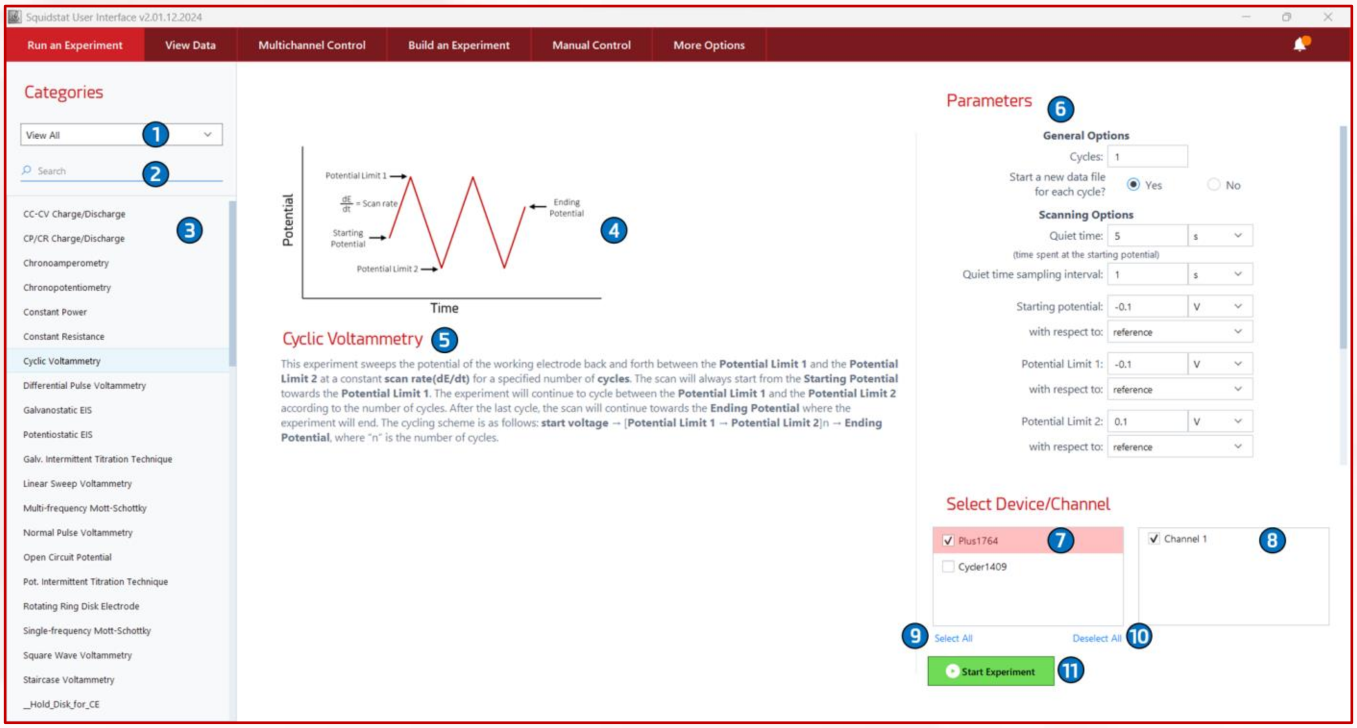
Append data to the last CSV file that has matching column headers: Control if data collected with this tile is placed in a new file or grouped with data from previous method tiles. See general notes.

10 Squidstat User Interface (SUI) Main Screen

Users will find a red bar at the top of SUI containing static tabs: ***Run an Experiment***, ***View Data***, ***Multichannel Control***, ***Build an Experiment***, ***Manual Control***, and ***More Options***. These tabs are used to quickly access our supported features. A notification bell can also be found in the top right-hand corner and is used to quickly find messages that are sent to the SUI log files, including errors, notifications, and general notes.

10.1 Run an Experiment

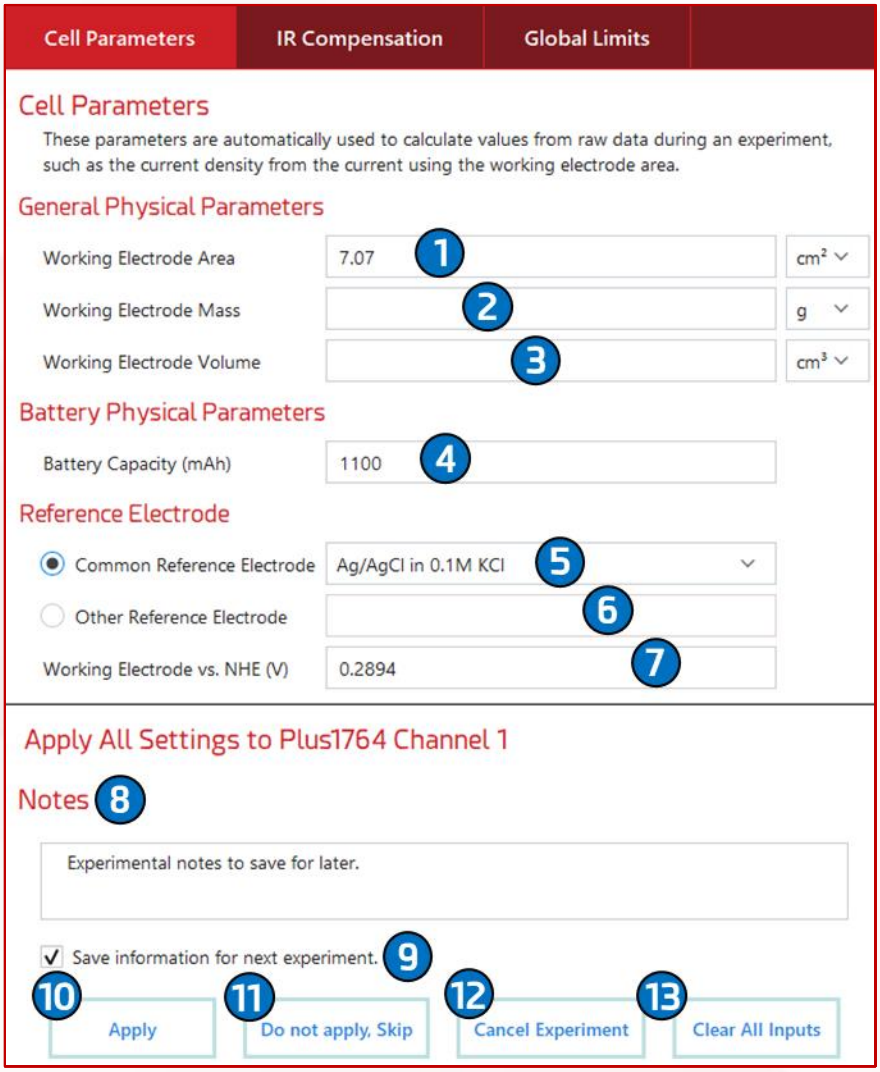
When opening the SUI for the first time, the ***Run an Experiment*** tab is selected by default. Users can modify and run single pre-built electrochemistry experiments from this tab. Custom built experiments will always populate below the pre-built experiments. Users are only able to alter the settings for single experiments for our pre-built experiments. To design detailed custom experiments involving a chain of multiple experiments, see ***Build an Experiment*** in **Section 10.4**.



Label	Description
1	Drop-down menu to select a set of experiments corresponding to an application.
2	Search bar to select experiments by keyword
3	Pre-built and custom experiments can be found in this pane. Select a pre-built experiment to view the description and edit parameters. Custom experiments appear below the list of the pre-built methods. Custom experiments are automatically added to this list once saved. To view only custom experiments, select ***Custom*** in the dropdown box.
4	Illustration of the waveform applied by the Squidstat for the selected experiment.
5	Title and a brief description of the selected experiment
6	Editable parameters for pre-built experiments are displayed here. If a custom-built experiment is selected, two choices are shown: ***Edit Experiment*** and ***Delete Experiment***. ***Edit Experiment*** takes the user to the ***Build an Experiment*** tab to modify the experiment. ***Delete Experiment*** deletes the selected experiment entirely.
7	List of Squidstats that are powered on and connected to the computer. Squidstats unable to run a selected experiment, such as EIS on the Squidstat Solo, will be greyed out.
8	Channels available for the selected Squidstat
9	Selects all inactive channels for all connected devices
10	Deselects all inactive channels for all connected devices.
11	Start an experiment for all channels and devices selected. A window will appear to add custom notes that will be saved in the experiment file, **Section 10.1.1**. After this window a file save window appears to allow users to define where the experiment folder will be saved.

10.1.1 Cell Parameters

This window appears after clicking ***Start Experiment*** and allows users to enter general physical parameters for working electrodes, battery capacity, and parameters for standard reference electrodes and custom reference electrodes. Custom CSV files may use parameters entered here such as working electrode area to calculate current density. Battery capacity must have a value if the user has chosen C-rate as the unit for applied current.

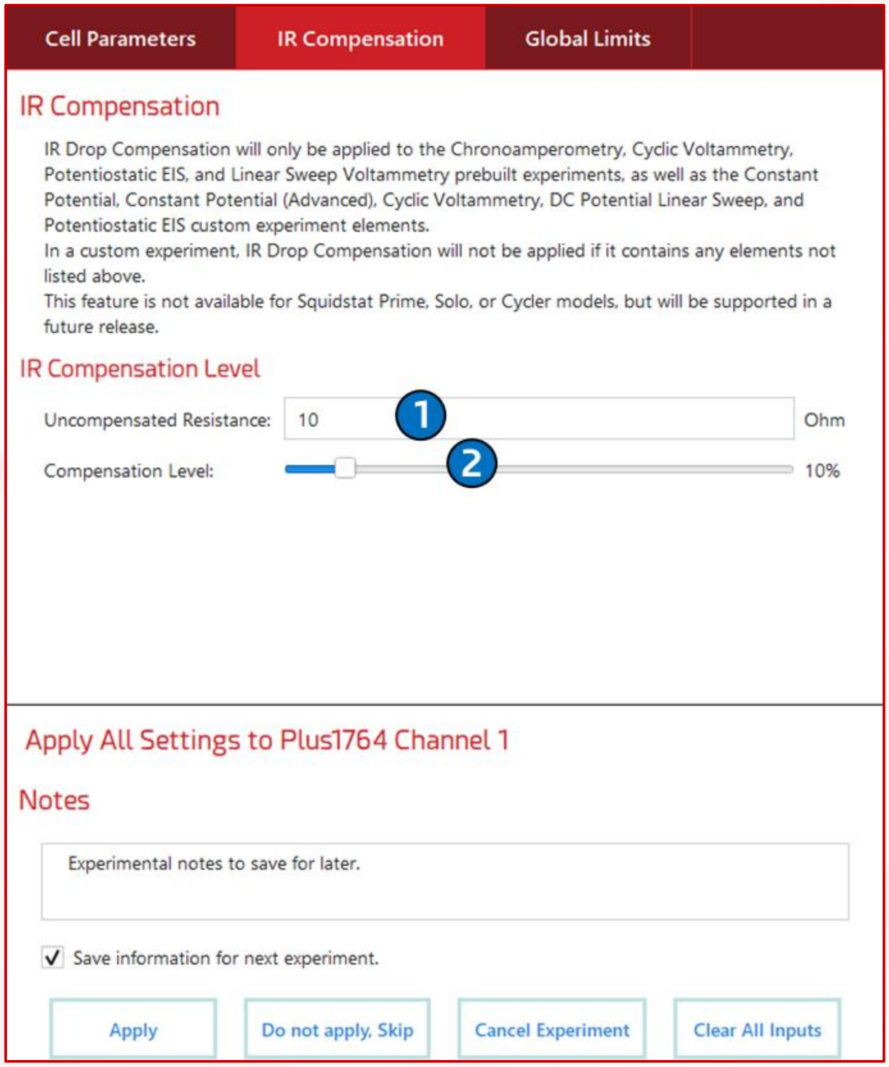


Label	Description
1	Enter the working electrode surface area to automatically calculate current density.
2	Enter the working electrode mass.
3	Enter the working electrode volume.
4	Required value if C-rate was chosen as a unit for current anywhere in the experiment.
5	Drop down menu containing multiple standard reference electrode types. The SUI will automatically provide a potential value versus this reference electrode in the CSV data file.
6	Custom reference electrodes can be entered here.
7	Set a custom value for the reference electrode potential calculation.
8	Notes for this experiment to be saved for later reference.
9	Keeps all entered information, saving users time on the setup of the next experiment.
10	Clicking ***Apply*** adds all information on this page to the experiment settings and notes text file created when the experiments begins.
11	Clicking ***Do not apply, Skip*** results in no information on this page being saved to the experiment settings and notes text file.
12	Clicking ***Cancel Experiment*** will close this window and cancel experiment in queue.
13	Clicking ***Clear All Inputs*** will set all input box values from this window to null.

10.1.2 IR Compensation

Readings from any potentiostat sometimes require building in a correction factor to account for uncompensated resistance. This option allows users to enter this correction factor for IR drop. The value of the uncompensated resistance and the compensation level, expressed as a percentage of the value input for the uncompensated resistance, (0 = no compensation, 100 = full compensation) are configured. The Squidstat applies the corrected voltage accordingly.

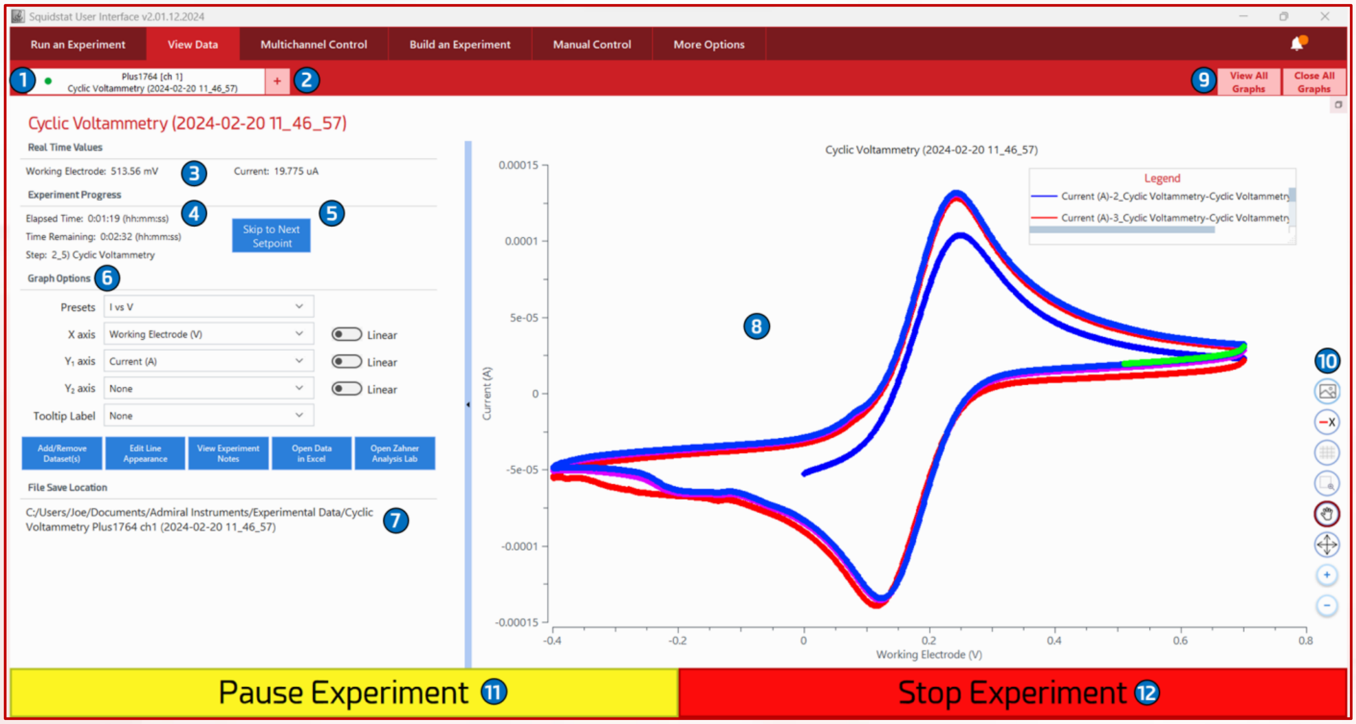
Uncompensated resistances are usually measured using one or more of the following three techniques: positive feedback, current-interrupt technique, and EIS. EIS is only available with the Squidstat Plus, Squidstat Boosted Series, and the Squidstat Cycler. Uncompensated resistance can also be calculated by applying current pulses. The resulting initial jump in the voltage response is due to the uncompensated resistance. This technique can be applied using any Squidstat model.



Label	Description
1	A value must be entered into this box for IR compensation to be applied.
2	Set the IR-compensation level as a percentage. 0 = none, 100 = full.

10.2 View Data

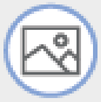
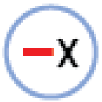
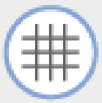
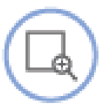
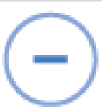
This window has multiple functions depending on the current task. Experiments that are actively collecting data will be shown in this tab and will have a green dot in the graph tab. All graphs generated while running pre-built or custom experiments will populate in this window. Previously collected data can be loaded into this view by using the **+** icon. The ***View All Graphs*** button allows users to show multiple graphs at once. Double click a specific graph to isolate it as a single view.



Label	Description
1	Graph tab. Contains the name of the device, the associated channel, and the name of the experiment. A green dot appears in the tab if an experiment is currently running.
2	Opens a new graph tab to load a new data set allowing the display of previously saved data.
3	Real-time values are populated here during active experiments.
4	Shows the time elapsed since the experiment began, the time remaining for the experiment to end (when applicable), and the active step of the current experiment (when applicable).
5	Skips the current step. For cyclic voltammetry (CV), the Squidstat will jump to the next potential limit and reverse the scan direction. For chrono methods, the ongoing step will end and the next step in the queue will commence. For EIS, this button will skip the measurement at the current frequency and step to the next frequency in the queue.
6	Options to change how the data is displayed on the graph. See **Section 10.2.2 Graph Options** for a detailed description.
7	Path to saved data file. Click on this link to directly go to the folder of the saved file.
8	The data is displayed here.
9	View all open graphs. To maximize a single graph, click the maximize button on the window of that graph. Users can also select individual graphs and drag them around the screen.
10	Function buttons to further refine the graphics of the plot. See **Section 10.2.2 Graph Options** for a detailed description.
11	Pauses and subsequently resumes the experiment.
12	Stops the experiment before its programmed conclusion.

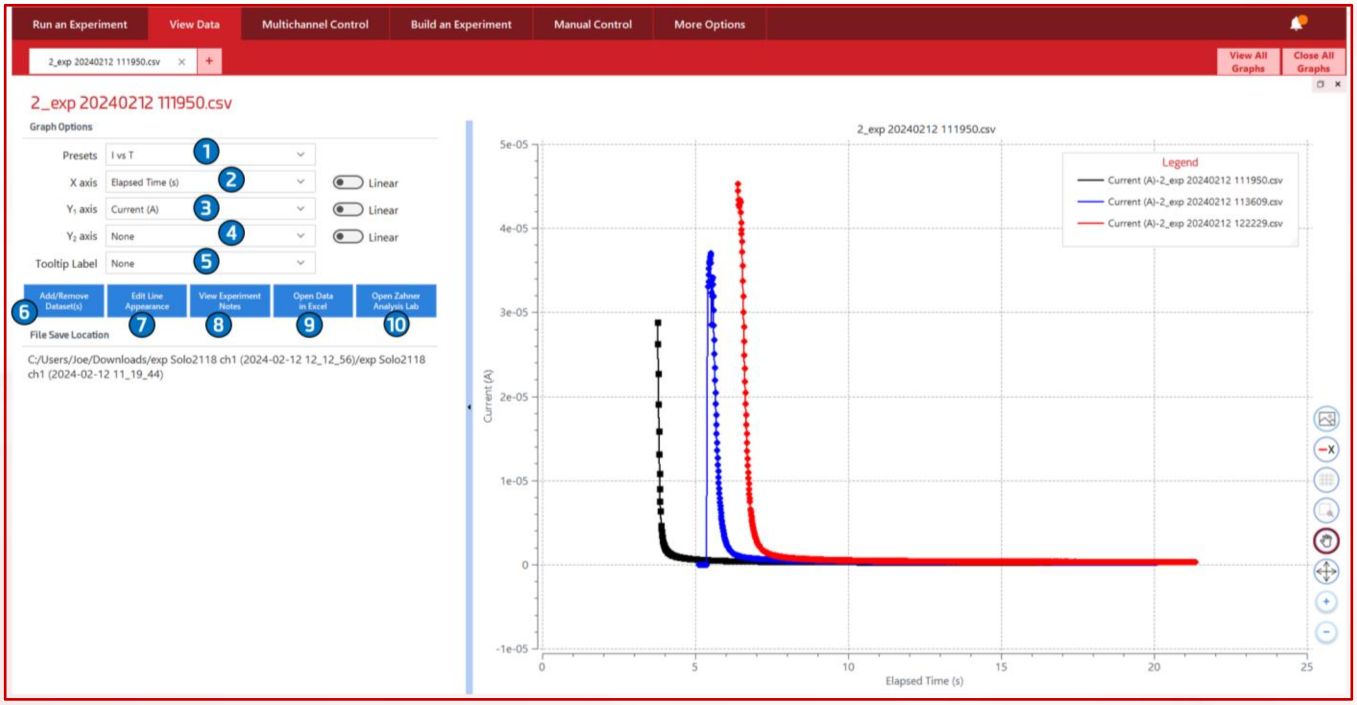
10.2.1 Graphing Tools

Various graphing tools are available to edit the visual aspects of the graphs in the ***Manual Control*** and ***View Data*** windows. These graphing tools also appear by right-clicking with the mouse while placing the cursor on the graph area.

Label	Icon	Description
Save Graph as Image		Saves the graph as a .png image file.
Show/Hide Legend		Display/hide the color-coded data legend.
Show/Hide Gridlines		Add or remove the major gridlines from the graph.
Zoom to Selection		Select any part of the plot to zoom. To zoom in, left-click, hold down the mouse button, and drag the cursor to form a box over the area to zoom into.
Pan View		Shift the plot about the x- and y-axis by left-clicking and holding down the mouse button to freely move the plotted area of the graph.
Reset Zoom		Resets the plot back to its original scale after zoom or pan operations.
Zoom In		Zoom in. Rescales both the x- and y-axis evenly.
Zoom Out		Zoom out. Rescales both the x- and y-axis evenly.

10.2.2 Graph Options

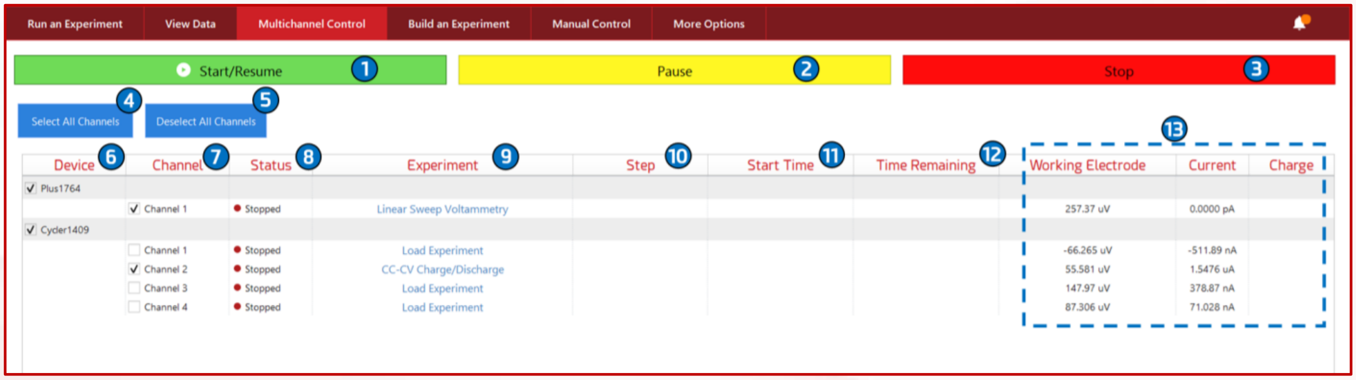
Extended options to format the visual representation of each graph is available under Graph Options. These options are available under both ***Manual Control*** and ***View Data*** control tabs.



Label	Description
1	Choose from predefined plots.
2	List of available parameters to graph on the x-axis. Can be changed from linear to logarithmic by toggling the switch to the right of the drop-down box.
3	List of available parameters to graph on the primary y-axis. Can be changed from linear to logarithmic by toggling the switch to the right of the drop-down box.
4	List of available parameters to graph on the secondary y-axis. Can be changed from linear to logarithmic by toggling the switch to the right of the drop-down box.
5	List of available parameters which can be displayed below X and Y values when the crosshair cursor highlights a data point on a plot.
6	Add previously saved data files to superimpose multiple plots. Removes data files if more than one file is loaded.
7	Open a menu to format the display of the data in the graph.
8	Opens a text editor with the “Experimental Settings and Notes” file from within the directory of the first data set loaded. File contents can be edited and saved within this window.
9	Opens each data set individually in Excel. Superimposed data files will not open in one excel file.
10	Launches the Zahner Analysis Lab, if the software was also installed during the Squidstat User Interface installation process.

10.3 Multichannel Control

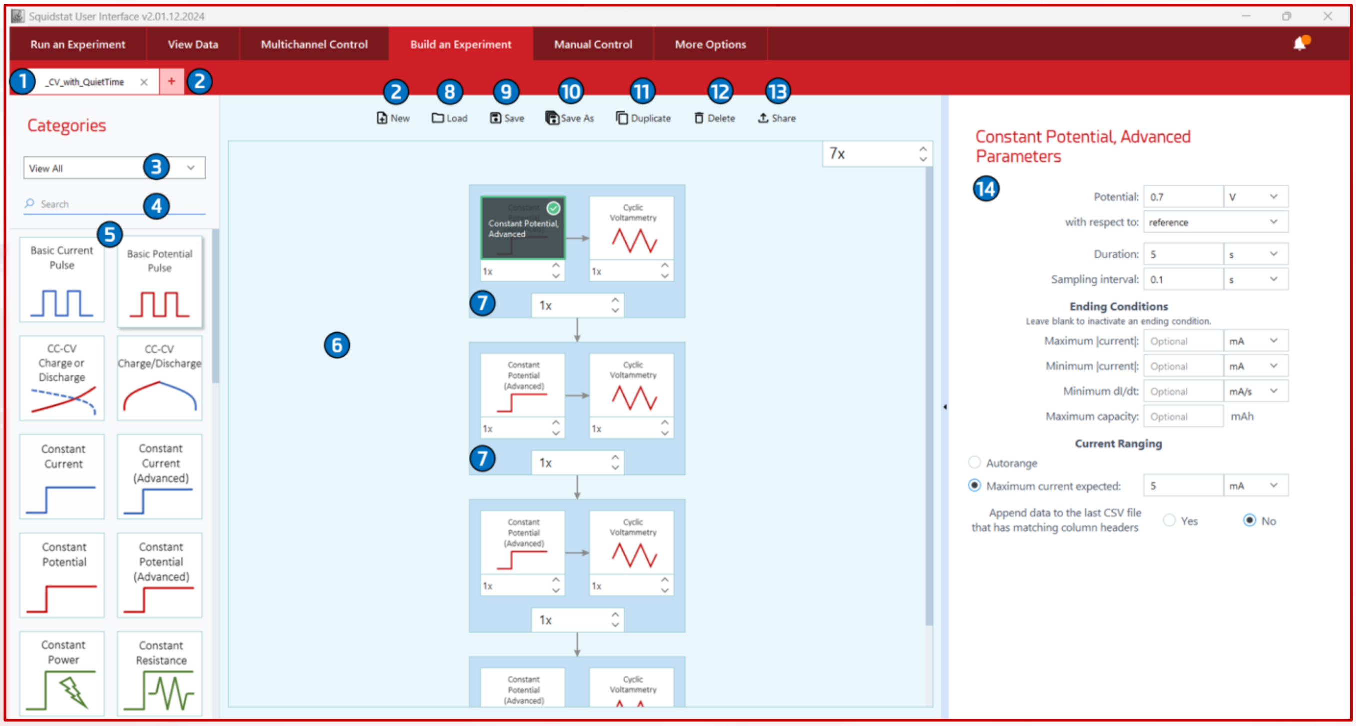
The ***Multichannel Control*** tab allows multiple Squidstats to be controlled and monitored from the same window. Users can start, pause, and stop experiments on multiple Squidstats and Squidstat channels simultaneously. This function allows multiple Squidstats to be utilized together as a single multichannel instrument.



Label	Description
1	Starts (or Resumes) experiments loaded for all activated Squidstat channels.
2	Pauses all experiments on all activated Squidstat channels.
3	Stops all experiments on all activated Squidstat channels.
4	Selects all channels for all Squidstats connected.
5	Deselects all channels for all Squidstats connected.
6	Displays the name of the Squidstat.
7	Displays all channels available for the parent Squidstat.
8	Displays the status of the channel: Active, Paused, or Stopped.
9	Displays the experiment to be loaded to this channel of the Squidstat. Can be pre-built or custom. Only one set of parameters can be loaded at once for pre-built experiments.
10	Displays the active step when an experiment is running.
11	Displays the time the experiment began.
12	Displays the time remaining for the experiment (when applicable).
13	Values related to the working electrode of each channel.

10.4 *Build an Experiment*

The purpose of this tab is to design and construct custom experiments. Users must save custom-built experiments before than can be ran on a Squidstat potentiostat or Squidstat Cycler. To run a custom experiment, click the ***Run an Experiment*** tab followed by one of three actions: 1) scroll to the bottom to find your custom experiment, 2) type the name of your custom experiment into the search bar, 3) use the drop down and select ***Custom*** to display only custom experiments.



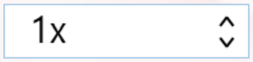
Label	Description
1	Name of the experiment being built or edited. Default name "New experiment" can be changed by saving the experiment under a different name.
2	Opens a new tab to build a new experiment.
3	List of method tiles categorized by their application. Method tiles can appear in multiple categories.
4	Search bar to find method tiles by keyword.
5	List of all available method tiles.
6	Drag and drop method tiles from (5) to this pane. Link method tiles by hovering over a previously dropped method tile. Method tile additions can be made to the left, right, top, or bottom of any tile.
7	The dark blue area contains a sequence of method tiles. Left click to select all of the method tiles in the same row to either delete or duplicate the sequence. A step count box is located below each method tile and the method tile group. Changing the counter from "1x" to "nx" will run this tile/sequence n times before proceeding to the next method tile or sequence of method tiles.
8	Load a previously saved custom experiment to make modifications. A dialogue box will appear with a reminder to save any in-progress experiments. A selection window will then appear with a list of saved experiments. Select the desired experiment and press Open. Press Cancel to go back to the previous page
9	Saves the custom experiment currently under construction.
10	Save the custom experiment as a new file name.
11	Duplicates the selected method tile or tile row.
12	Deletes the highlighted method tile or tile row.
13	Opens the "Custom Experiments" folder containing all saved custom experiments.
14	Parameters for the selected method tile. If multiple method tiles of the same type are in the build window, only the parameters for the selected method tile will be modified.

10.4.1 How to Build Custom Experiments

Building custom experiments within the Squidstat User Interface is like fitting different pieces of a puzzle together. Various method tiles can be arranged as desired to create a practically limitless number of customized experiments.

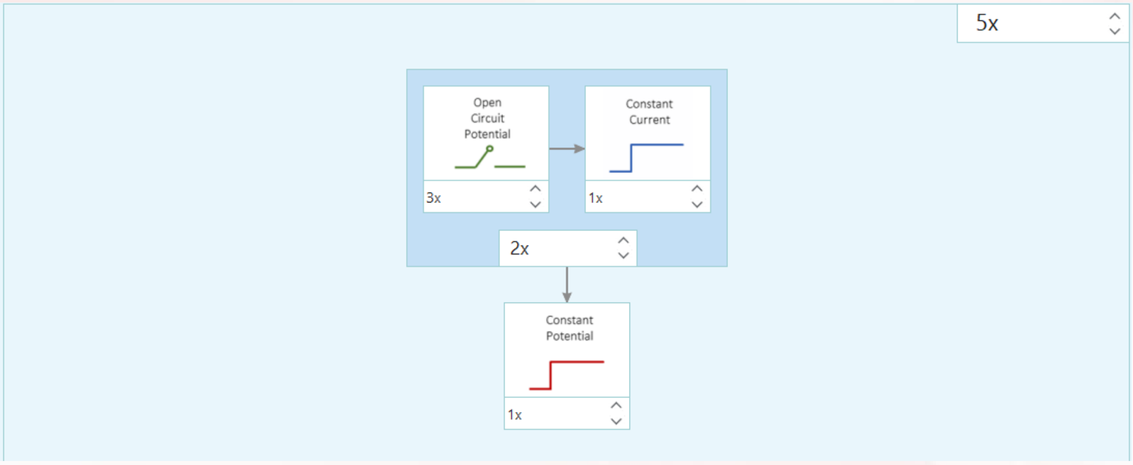
To build an experiment, select a method tile from the category section on the left, then drag and drop it into the **Drag and Drop** area in the middle of the screen with the light blue background. Once the first tile is dropped into place, additional tiles can be added in any direction (up, down, left, or right) by dragging another tile and hovering it near the first tile. The location of the drop will be indicated by a black bar corresponding to the new location. Execution order of the sequence tiles within the experiment follows the arrows from tile to tile left to right, top to bottom.

Individual method tiles, rows of method tiles, and even the complete set of method tiles in the builder can be repeated any number of times before proceeding to the next step of the experiment. To select the number of repeats, simply use the arrows or type in the number of repeats into these boxes:



To change the parameters of each tile, simply click on the tile to open the parameter options on the right side of the screen. For detailed explanations of the parameters for each tile see **Chapter 9**.

Example experiment procedure defined below:



1. The Squidstat will run the **Open Circuit Potential** tile 3 times
2. **Constant Current** tile will run once
3. The Squidstat will again run the **Open Circuit Potential** tile 3 times and the **Constant Current** tile one time, as per the "2x" on the bottom of the first row
4. A **Constant Potential** step will run once
5. After completion of the **Constant Potential** tile, the Squidstat will jump to the **Open Circuit Potential** tile again and repeat the entire sequence four additional times, as per the "5x" on the top right corner.

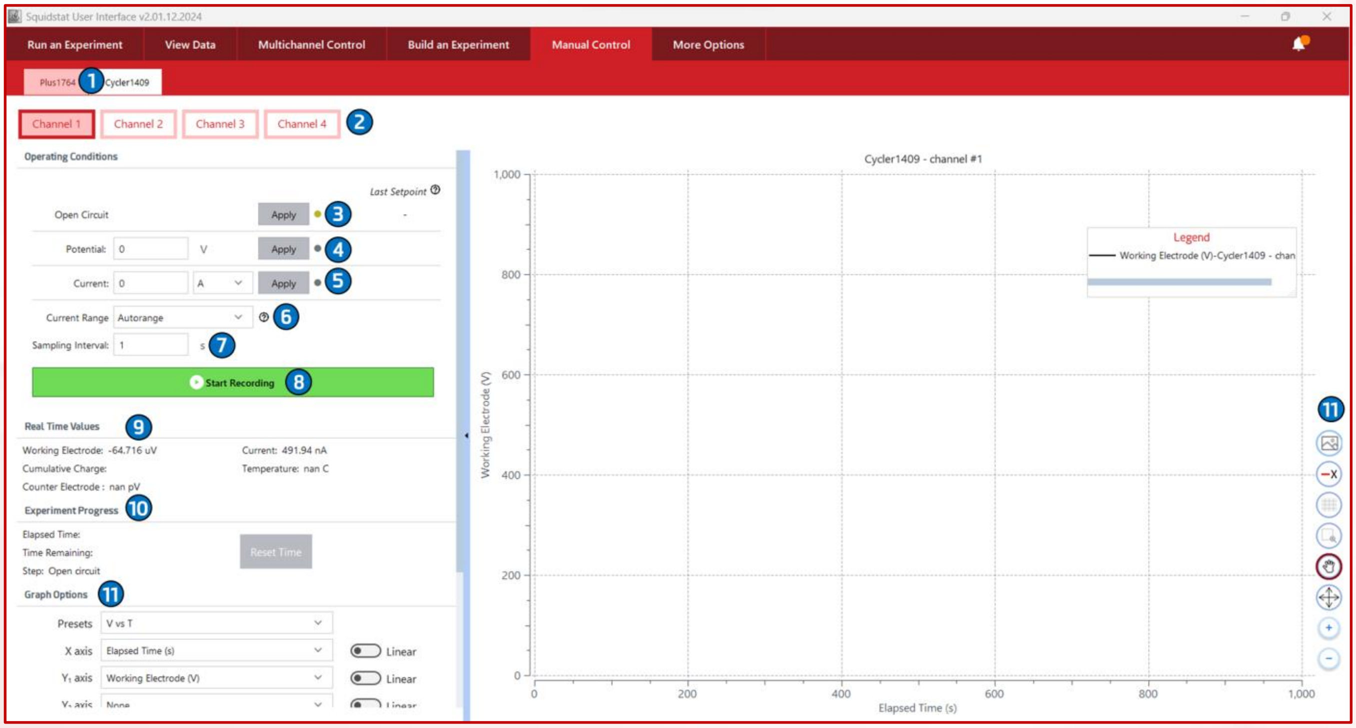
After the method tiles have been arranged and their parameters set, the next step is to save the experiment by clicking the ***Save*** or ***Save As*** button. A popup will appear prompting users to type a name and an accompanying description. Once saved, this custom experiment will appear in the ***Run an Experiment*** tab and can be applied to any applicable channel.

10.4.2 How to Modify or Delete Previous Custom Experiments

To reopen a saved experiment to review its parameters and make modifications, go to the ***Run an Experiment*** tab, select the desired experiment, and click ***Edit Experiment*** on the right-hand side of the window. Alternatively, go to the ***Build an Experiment*** tab and click the ***Load*** button to open a menu of all custom experiments. Users can edit or delete experiments within this menu.

10.5 *Manual Control*

This tab allows users to apply a constant current or constant voltage and change the applied value while the experiment is running.



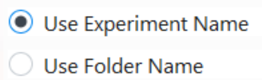
Label	Description
1	Displays the serial number of Squidstats powered on and connected to the computer. A green dot appears beside the name if the Squidstat is actively recording data.
2	Red boxes highlight the Squidstat channel selected to run the manual experiment.
3	Enables an open circuit potential reading.
4	Forces the Squidstat channel to apply a constant potential.
5	Forces the Squidstat channel to apply a constant current.
6	Set the current range for the selected Squidstat channel.
7	Define the sampling time.
8	Brings up a new window to define a save location for the manual experiment data.
9	Real time values from the selected channel on the Squidstat.
10	Time component information for the selected Squidstat channel.
11	All graphing options are identical to those in **Section 10.2.1**.

10.6 *More Options*

This tab enables a higher degree of control over experiments and settings within the SUI. Data output, measurement, font, and channel link options are found on the left-hand panel of the window titled Options. Miscellaneous information such as release notes, device information, and information about Qt are also included within this tab. Each option is described below.

10.6.1 CSV File Names

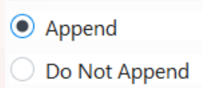
Users have the ability to control the format of the preset CSV file name to include the experiment being conducted to write the file.



Use Experiment Name: Appends the experiment type to the CSV file name.

Use Folder Name: Appends the name of the folder, as defined by the user, to the CSV filename.

The two options below refer specifically to adding the experiment name to the CSV file. Options below are useful when "Use Folder Name" above is chosen.



Append: Adds the experiment name to the CSV file name. Note that the experiment name will be added twice if "Use Experiment Name" is enabled above.

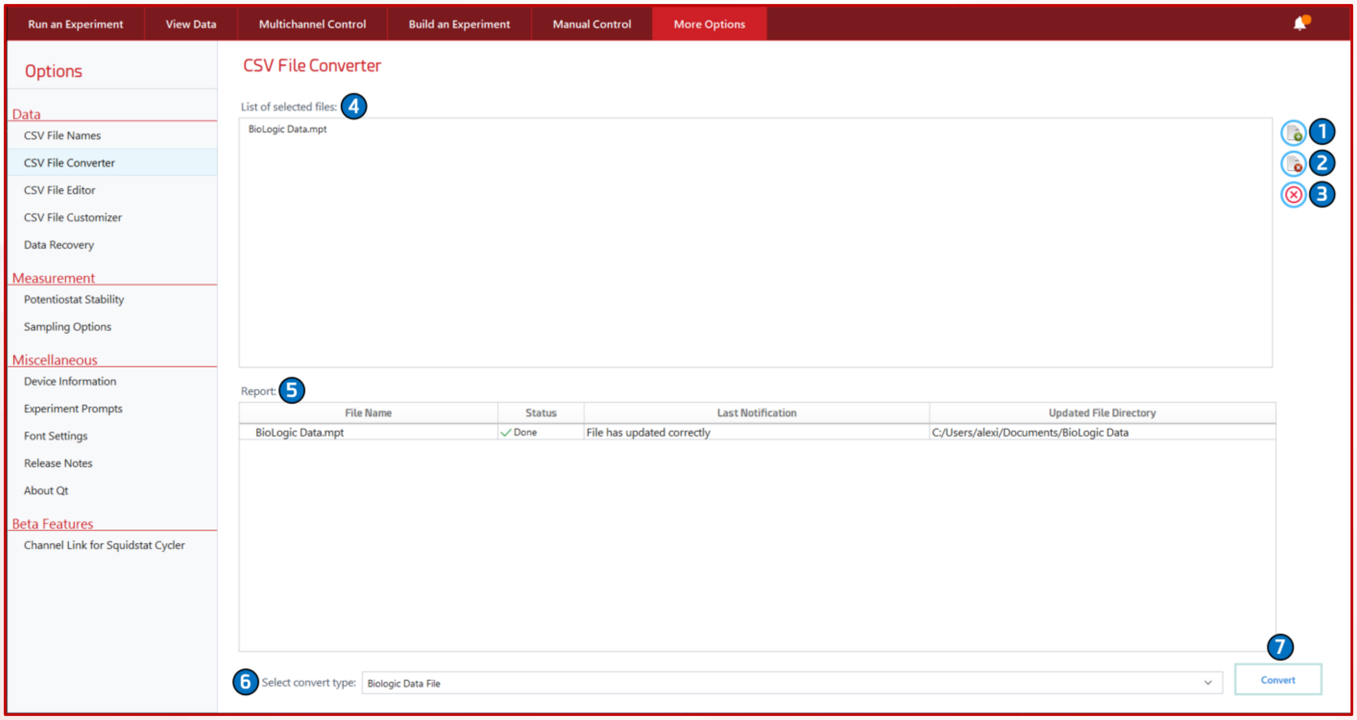
Do Not Append: Will not add the experiment name to the CSV file. If "Use Experiment Name" is selected above the experiment name will only be added once to the CSV filename.

10.6.2 CSV File Converter

This option allows users to convert data files collected by Gamry and BioLogic brand potentiostats to CSV files so they can be viewed in the Squidstat User Interface software. The two file conversions are:

1) Convert Gamry .DTA extension to Squidstat .CSV

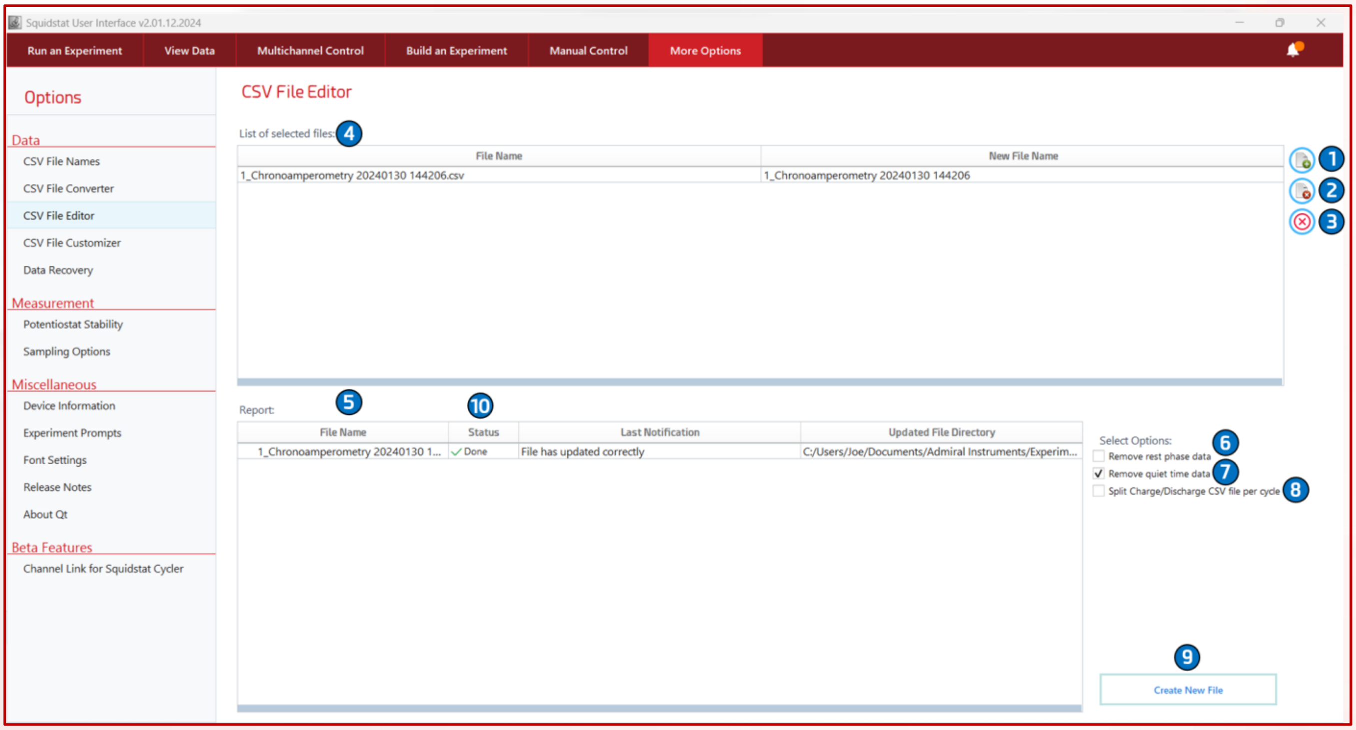
2) Convert BioLogic files with .mpt extension to Squidstat .CSV



Label	Description
1	Choose files to be converted.
2	Remove selected file.
3	Removes all files.
4	List of the files added.
5	Status of file conversion: complete/successful/error. Also shows the path to the folder where the converted file was saved.
6	List of available formats to convert to.
7	Initiates file conversion.

10.6.3 CSV File Editor

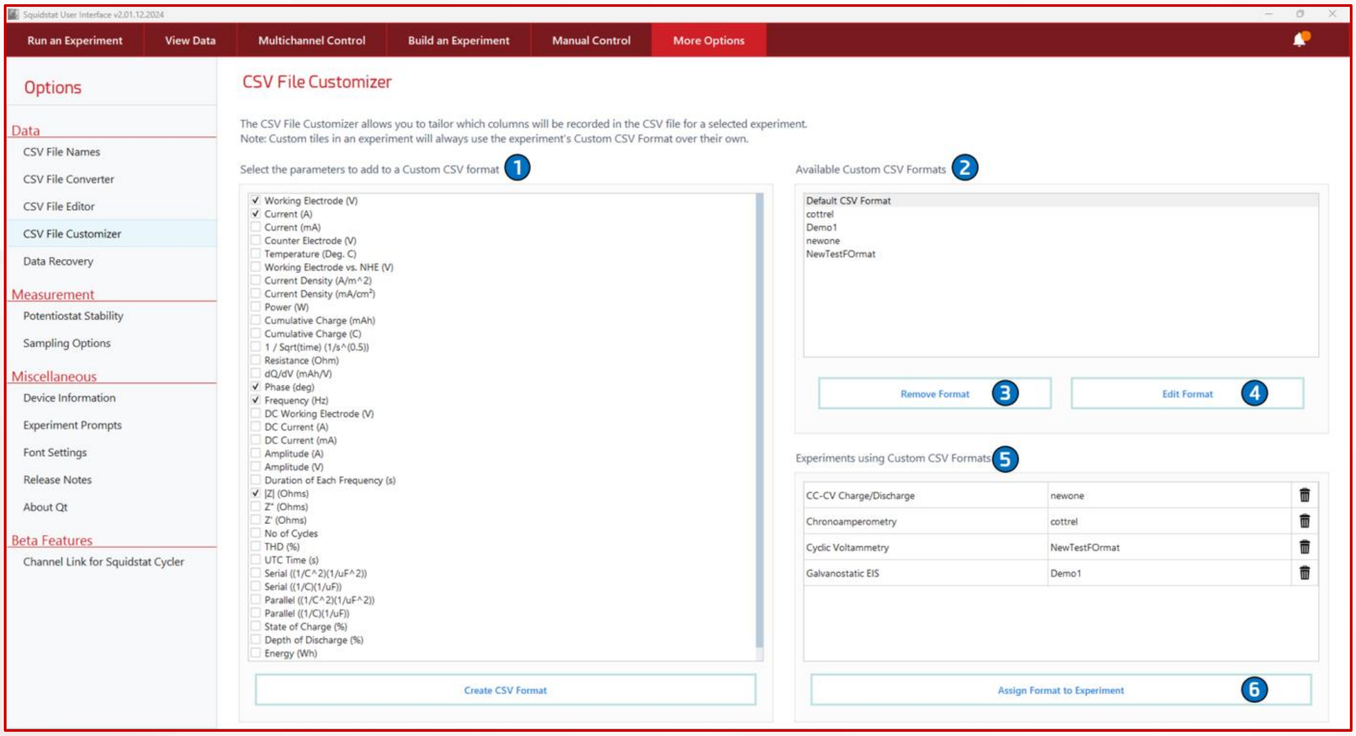
This option can remove rest phase data, remove quiet time data, and split a multi-cycle CSV file into one CSV file per cycle. These operations are only applicable to CSV files from Charge/Discharge experiments in the ***Run an Experiment*** tab.



Label	Description
1	Choose files to be edited.
2	Remove selected file.
3	Removes all files.
4	List of the files added.
5	Name of the new file created after editing. Double-click the file under this column to provide a different name. Otherwise, the new edited CSV file will have "_1" added to the end of the old filename.
6	Removes rest phase data from CSV files from Charge/Discharge experiments.
7	Removes quiet time data from CSV files.
8	Split CSV files from Charge/Discharge experiments into one CSV file per cycle. i.e. 20 cycles results in 40 files.
9	Initiates file editing.
10	Status showing complete/successful/error. Also shows the folder where the new file is saved.

10.6.4 CSV File Customizer

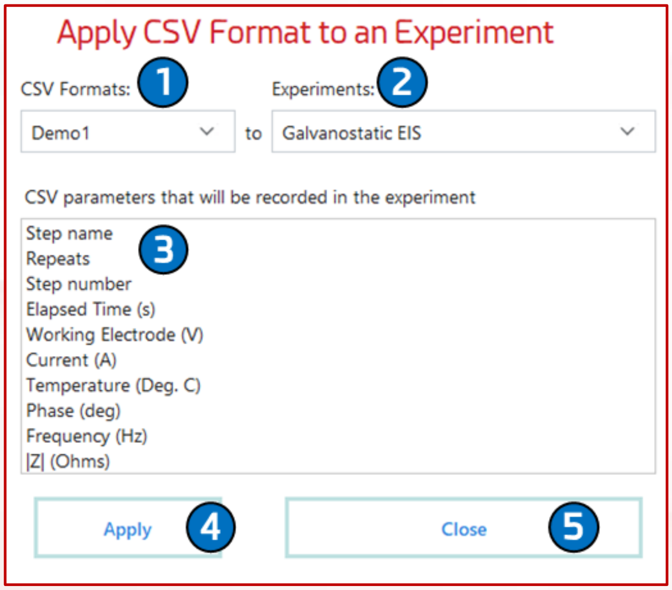
Users have the ability to define exactly how their data will be written to a file. Any parameter with a checkmark in the box will be included as a header in the custom CSV file. Users must first define the headers for the CSV file and ***Create CSV Format***. After saving the custom format the name will appear under (2). Then users can ***Assign Format to Experiment***. Once the assignment has been completed the custom CSV format name will appear in the column on the right of (5) and the experiment name on the left.



Label	Description
1	Parameters available to write to a csv file.
2	Default csv parameters are found here. Custom formats will populate here once created.
3	Deletes the custom format entirely.
4	Edit the selected custom format.
5	Shows which experiments will be using a custom csv format.
6	Opens a new menu. Options are defined below.

10.6.5 Apply Custom CSV to Experiment Window

Using the CSV Format dropdown menu, choose an available custom CSV format to apply to the experiment where a custom CSV format is desired.



Label	Description
1	List of available custom CSV formats
2	List of experiments to apply the custom CSV format to.
3	Displays all parameters that will be written to a file using the custom CSV format.
4	Links the custom CSV format to that specific experiment.
5	Close the window. Does not save changes.

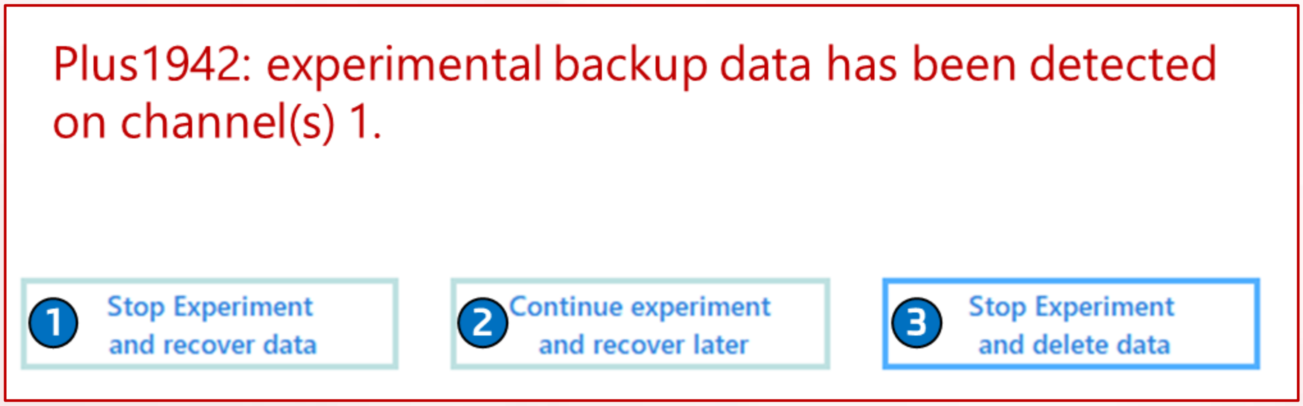
10.6.6 Data Recovery

Data recovery is available on the following models: Squidstat Prime with serial numbers >1100. Squidstat Solo with serial numbers >1100. Squidstat Plus with serial numbers >1100. Squidstat Ace with serial numbers between 1100 and 1699. All Squidstat Penta, Squidstat Decka, and Squidstat Venta.

Entire experiments are uploaded to the Squidstat when starting an experiment. If data collection is disrupted during an experiment, the Squidstat hardware is capable of storing 16 GB of raw data.

This option is used when there is backed-up data in the hardware and the user decides to recover it. The device and channel from where data is to be recovered and the folder where recovered data is to be stored can be chosen.

If another experiment is initiated on a Squidstat before the raw data is recovered, the data will be lost permanently.



Label	Description
1	Stop the experiment currently running on the Squidstat but not showing data collected in ***View Data***
2	Continue running experiment loaded onto Squidstat. Do not start an experiment before going to ***More Options*** > **Data Recovery**
3	Stops the active experiment on the Squidstat and deletes any data collected for that run.

10.6.7 Potentiostat Stability

Although it is rare, any potentiostat can be unstable. This happens when the bandwidth of the potentiostat is incorrect for the experiment. In simple terms, bandwidth is a measure of how quickly a potentiostat can react to the changing conditions of an experiment. If a potentiostat underreacts or overreacts, the potentiostat along with the device under test will be unstable. Therefore, both the applied and measured signals are unreliable. When such conditions arise, the stability range of a potentiostat should be modified.

The SUI remembers the last stability range used when it is restarted. Hence, for normal experiments, the stability range should be reset to the default value by clicking ***Reset to Default*** settings box. If the stability range is not in the default setting, the following message will appear at the bottom of the ***Run an Experiment*** window:

> "One or more default parameters in ***More Options*** tab has been changed for [Mode][Serial Number]. Please go to ***More Options*** if you want to reset them to default."

10.6.8 Sampling Options

Show data sampled during experiment "quiet times"

If users choose not to show data sampled during experiment quiet times, the real-time data recorded during the quiet times in methods like cyclic voltammetry and EIS will be not be shown in the ***View Data*** tab. However, the quiet time data will still be available in a separate CSV file in the raw data folder. This function does not apply to non-real-time data.

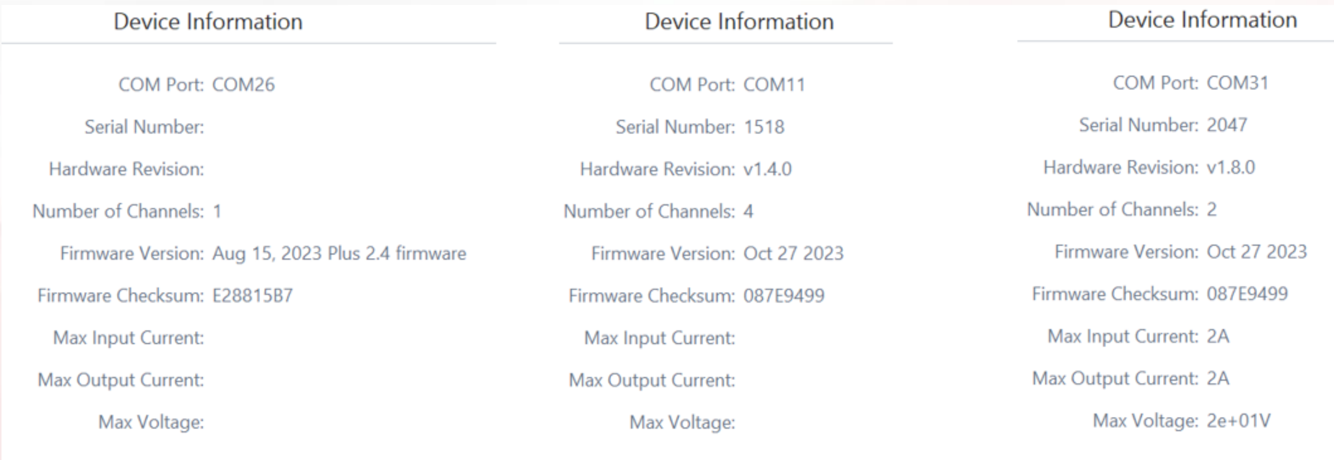
Show data sampled during experiment "rest time"

If users choose not to show data sampled during experiment rest time, the real-time data recorded during the rest times in Charge/Discharge experiments will not be shown in the ***View Data*** tab. However, the rest time data will still be available in the raw data file of the experiments. This function does not apply to non-real-time data.

10.6.9 Device Information

The Device Information page displays key details for each connected Squidstat, including the COM port, serial number, hardware revision, number of channels, firmware version, firmware checksum, maximum input current, maximum output current, and maximum voltage. The specific information shown varies depending on the Squidstat model. The figure below provides an example of the information displayed for each model.

All Potentiostat Models **Squidstat Cycler Base Model** **Customized Squidstat Cycler**



10.6.10 Experiment Prompts

Experiment Notes Prompt

After clicking ***Run Experiment***, a pop-up appears allowing users the option of adding notes about their experiment for future reference. These notes are accessible in the ***View Data*** window and in the text file that accompanies the raw data in each experiment folder. Users that do not wish to see this pop-up before starting every experiment can turn this option off here.

One Folder Prompt when Starting an Experiment on Multiple Channels

Before starting an experiment, users have the option of saving their channel data individually into folders they name and create themselves. Users who wish to create only a single folder where all channels will save data can turn off this option here.

Stop, Pause, or Resume Experiment Prompt

If users wish to be prompted before stopping, pausing, or resuming an experiment they can turn on this feature here. This is helpful if users wish to avoid unexpected interruptions to the desired potentiostat state.

10.6.11 Font Settings

This option sets the font of axis labels and the title of the graph in the ***View Data*** and ***Manual Control*** tabs.

10.6.12 Release Notes

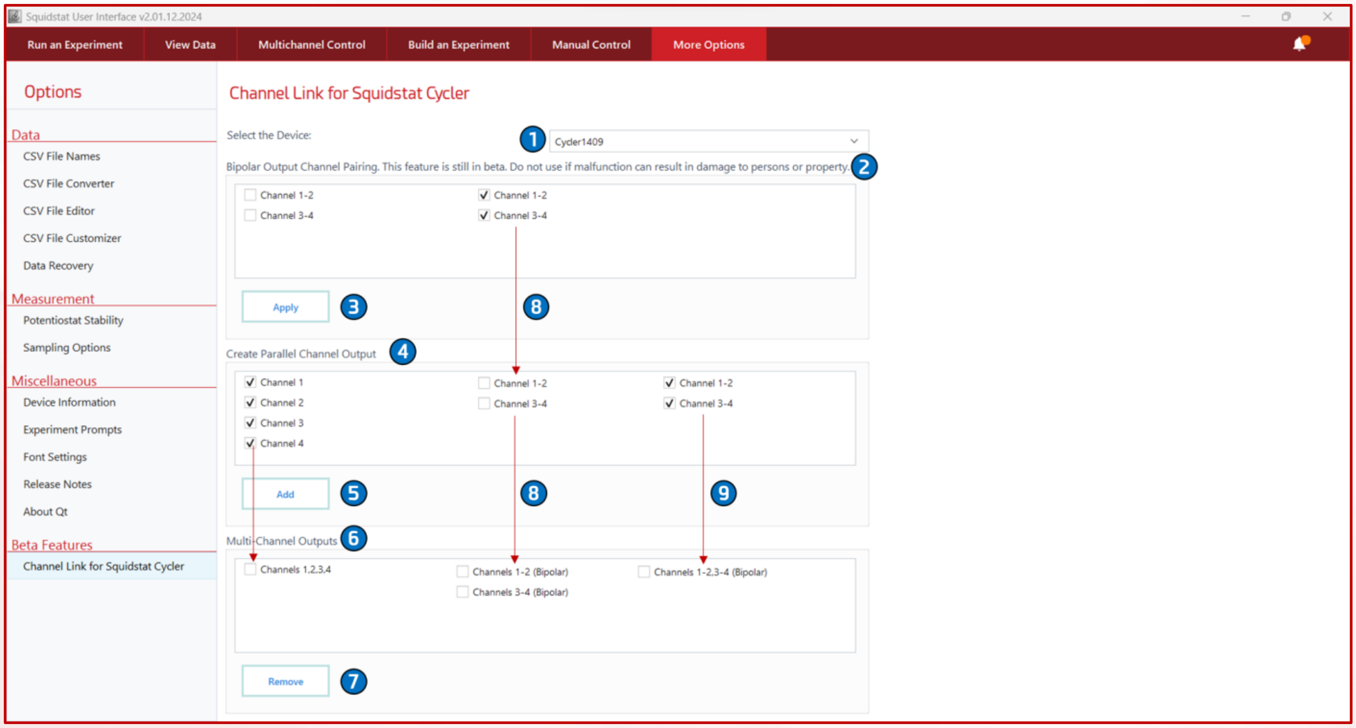
A history of new features, bug fixes, and miscellaneous changes made to the Squidstat User Interface are listed here.

10.6.13 About Qt

Basic information about the Qt version and public license are listed here.

10.6.14 Channel Link for Squidstat Cycler

The Squidstat Cycler is capable of multi-channel operation in two configurations: parallel mode, which increases the maximum current limit, and bipolar mode, which extends the voltage range to include negative voltages. No more than two channels can be combined in bipolar mode. However, multiple bipolar linked channels can be combined in parallel.



Label	Description
1	Drop-down menu to select the device to enable bipolar or parallel modes on.
2	Channel pairs available to link in bipolar mode are shown here.
3	Click ***Apply*** to select which channels should be linked. Successful linkages will populate in (4) and (6).
4	Channel pairs available to link in parallel mode are shown here.
5	Click ***Add*** to select which channels should be linked. Successful linkages will populate in (6).
6	Linked channels are shown here. Bipolar linkages are defined with a "-" and (Bipolar). Parallel linkages are defined by a ",".
7	Linkages can be removed by activating the check box and clicking ***Remove***.
8	Once a bipolar channel linkage is made the option to use this linkage in parallel mode is still available and the linkage will populate in (4) and (6) after clicking ***Apply***.
9	Linking two bipolar mode channels in parallel will result in a four-quadrant system with twice the available current. Ex. 0-5 V, +/-5 A default configuration → +/-5 V, +/-10 A.

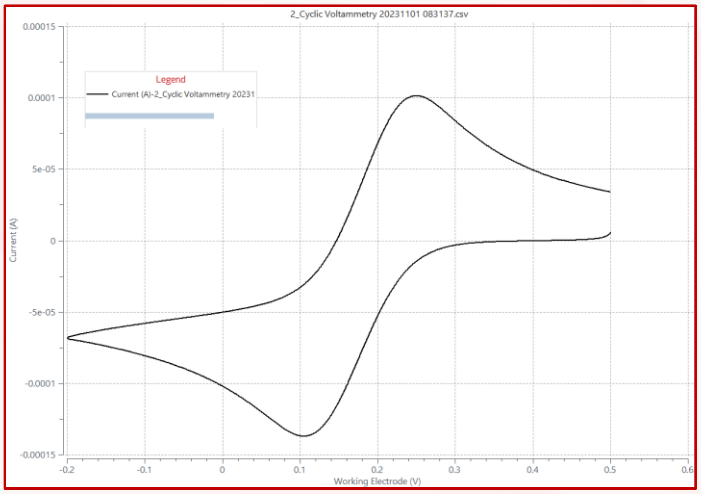
11 Preset Graphs

11.1 General

Several graphing presets are included in the SUI to represent data in common configurations when using the ***View Data*** tab. ***Manual Control*** contains basic presets for plotting DC experiments and excludes preset graph types. Some preset graphs are referred to by common names such as Bode Plot, Nyquist Plot, etc. Brief descriptions are provided below for the preset graphing options included in the SUI.

11.2 Voltammogram (i vs. E)

Voltammograms plot current (i) or current density (j) on the y-axis and the working electrode potential (V) on the x-axis. Voltammetry is a quantitative method to extract information about the electrochemical processes involved in the system. Examples include 1) the number of electrons involved in redox reactions, 2) the degree of reversibility of a redox reaction, 3) capacitance effects, 4) electrocatalytic rate constants, etc. In fuel cell research, voltammograms are often used to find the electrochemically active surface area of an electrode and hydrogen crossover effects. In supercapacitors, voltammograms can be used to calculate capacitance. In batteries, they can be used to elucidate effects of the solid-electrolyte interphase (SEI). Reaction mechanisms can be inferred by modelling data resulting from voltammograms.



11.3 Tafel Plot

The Tafel plot is a common tool for evaluating electrochemical kinetics. In this plot, the log of current i (A) is plotted on the y-axis, and overpotential (η), or working electrode potential E (V), is plotted on the x-axis.

Overpotential is defined as:

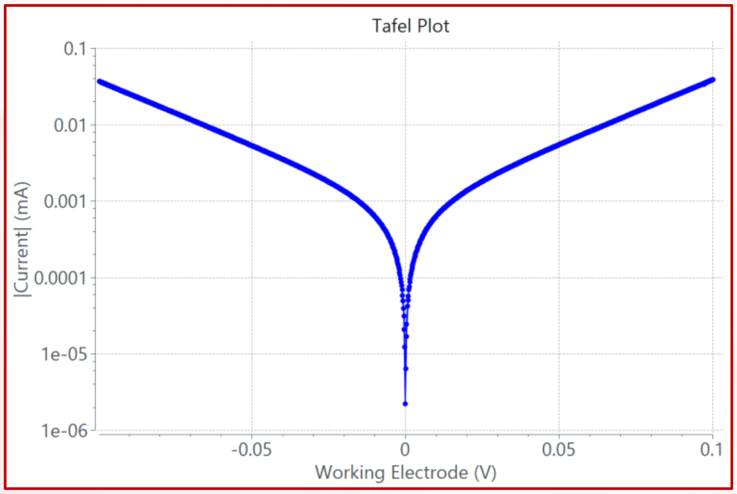
$$\eta = E - E_{eq}$$

Where E is the potential measured at the working electrode and E_{eq} is the equilibrium potential at the working electrode given by the Nernst Equation. In practice, E_{eq} can be considered steady-state potential at open-circuit, where the net flow of current is 0. The profile of the Tafel plot does not change if plotted against either η or E.

The Tafel plot is perhaps the most famous plot in corrosion-based electrochemistry. The relation between log(i) and η are given by the Tafel equation:

$$\eta = a + b \log(i)$$

Where a and b are Tafel constants. In short, the linear regions of the plot log i vs. E are fitted with a linear regression. The current intercept (i.e. where η = 0) of this linear regression gives log i_o, the exchange current, and the slope is the Tafel slope. If it is a reduction reaction, the Tafel slope is cathodic and if it is an oxidation reaction, the Tafel slope is anodic. The exchange current i_o is the current at the equilibrium potential E_{eq} when the sum of the anodic and cathodic currents is equal to zero (a zero-net current). This is a fundamental kinetic parameter of an electrochemical reaction, which can be substituted for the equilibrium rate constant k^0 in kinetic equations in certain conditions. It is a measure of how facile the reaction kinetics are: the higher the i_o, the faster the reaction rate.



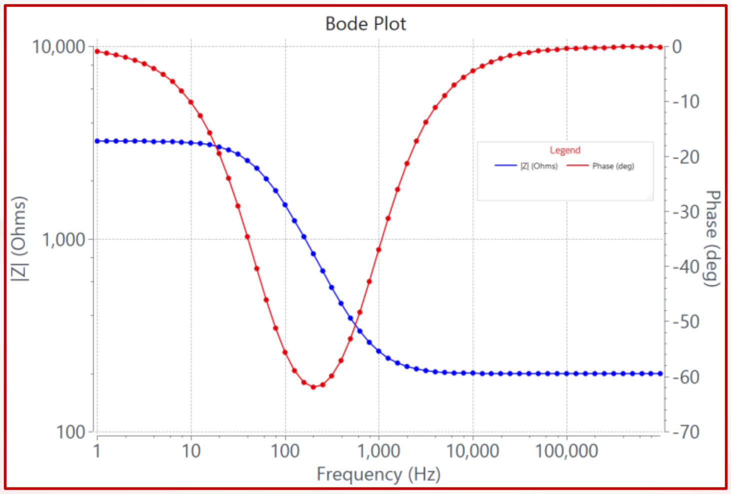
In the field of corrosion, i_o is also called the corrosion current (i_{corr}) from which the one-dimensional planar corrosion rate can be calculated using Faraday's law of electrolysis:

$$\frac{m}{t} = \frac{(i_{corr} * M)}{nF}$$

Where m is the mass, t is the total time, M is the molecular weight, F is the Faraday constant and n is the stoichiometric number of electrons.

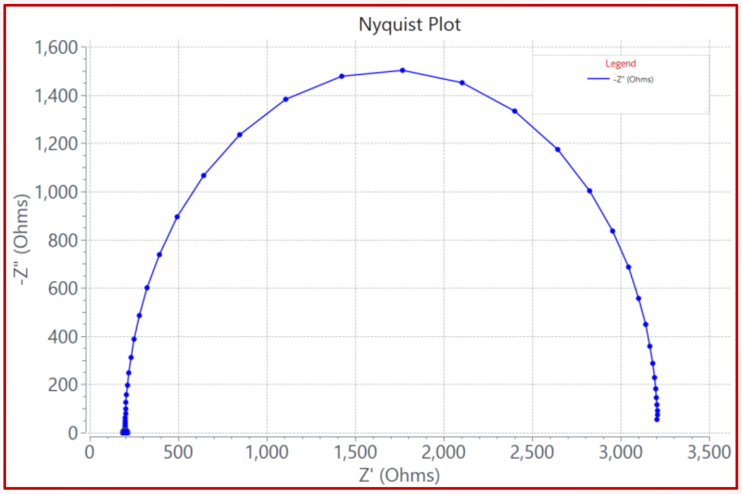
11.4 Bode Plot (|Z| and Phase vs. Frequency)

A Bode plot is one graphical representation of EIS data. The plot presents the logarithmically scaled impedance modulus (log |Z|) against logarithmically scaled frequency (log f) on the primary y-axis, with the phase angle (φ) displayed on the secondary y-axis. This representation allows for the identification of frequency-dependent phenomena and differentiation between capacitive, resistive, and inductive behaviors based on the phase angle.



11.5 Nyquist Plot ($-Z_{im}$ vs. Z_{re})

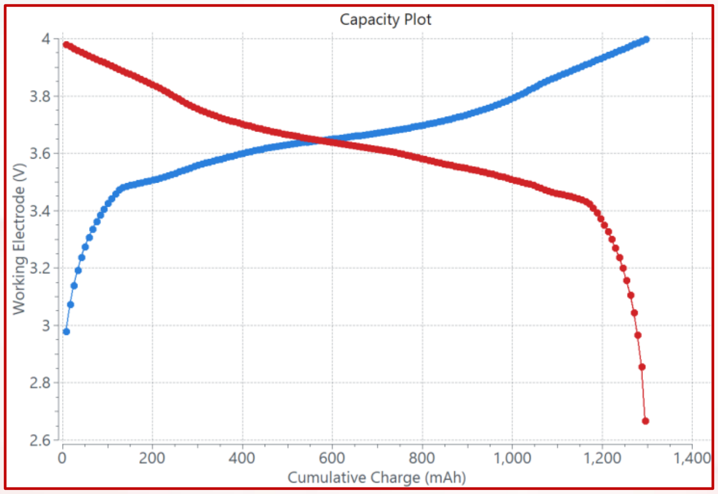
A Nyquist plot is another graphical representation of EIS data. The negative imaginary impedance ($-Z_{im}$ or -Z") is plotted against the real impedance (Z_{re} or Z′) in a complex plane. While frequency is not explicitly displayed, low frequency data is located on the right side of the plot while higher frequencies are on the left.



Nyquist plots are highly popular as the shape provides valuable insights regarding electrochemical processes occurring at the interface under investigation. For example, the x-intercepts of the semicircular portion at high frequencies can be used to calculate the solution resistance and the charge-transfer resistance. Mechanistic details about charge-transfer can be gleaned from the diameter of the semi-circle. For more detailed information on conducting and analyzing EIS experiments, refer to the application note Introduction to Electrochemical Impedance Spectroscopy.

11.6 Charge/Discharge or Capacity Plot (V vs C)

A capacity plot is a graphical representation of galvanostatic charge/discharge data. The working electrode potential (V) is plotted on the primary y-axis, and capacity or cumulative charge (mAh) during each half-cycle (i.e. charge or discharge) is plotted on the x-axis. During the discharge phase, the potential decreases while cumulative charge increases; during the charging phase, both the potential and cumulative charge increase. The application of this plot is mostly in battery research.



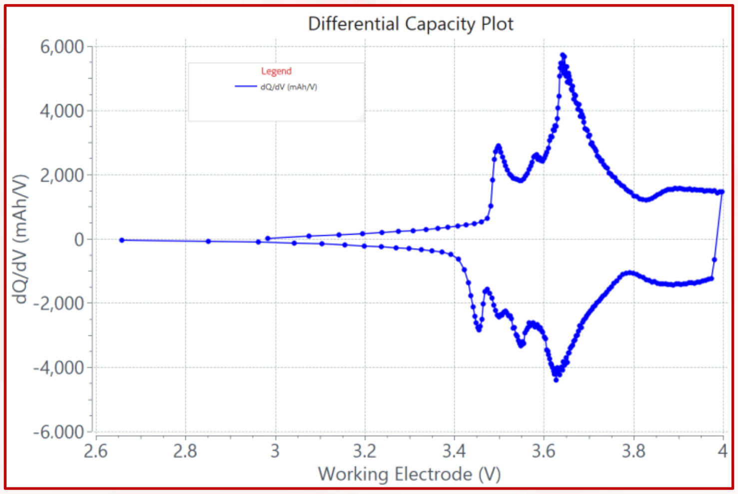
Because a capacity plot gives a direct measure of the capacity of a battery, it is the most popular plot used in battery research to analyze and characterize performance. For example, the battery capacity, or total amount of charge that the battery can store, is typically indicated by the maximum charge capacity reached during the charge cycle. The capacity of a battery at a given discharge rate can also be directly determined from the cumulative charge at a given lower voltage limit. Additionally, cycling performance can be characterized by changes in capacity and voltage profile over multiple charge and discharge cycles, which can indicate the degradation or aging of the battery over time. Other insights such as battery efficiency and state of health can be determined from this plot.

11.7 Differential Capacity Plot ($\frac{dQ}{dV}$ vs V)

A differential capacity plot is a graphical representation of galvanostatic charge/discharge data. The derivative or differential of capacity or cumulative charge (mAh) with respect to working electrode potential (V) is plotted against the working electrode potential (V). The equation used to calculate $\frac{dQ}{dV}$ is given below.

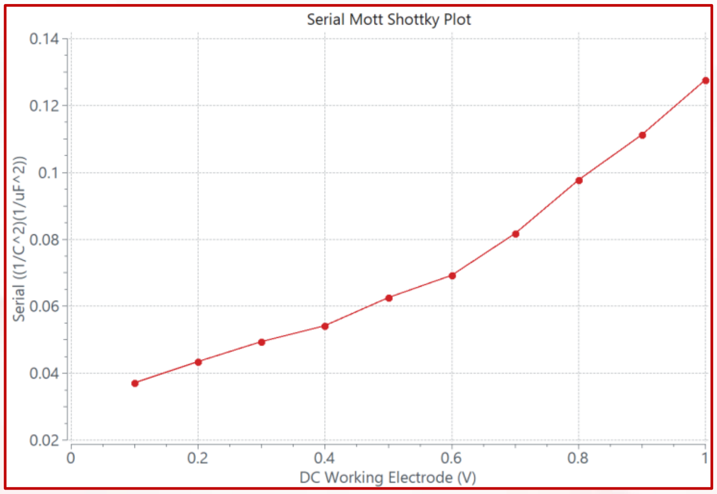
$$\frac{(\boldsymbol{Cumulative\ Charge})_n - (\boldsymbol{Cumulative\ Charge})_{n-1}}{(\boldsymbol{Working\ Electrode\ Potential})_n - (\boldsymbol{Working\ Electrode\ Potential})_{n-1}}$$

Differential capacity plots show voltage-dependent peaks that can be correlated to reactions inside a battery and can be used for battery diagnostics. The peaks correspond to significant changes in capacity per change in voltage. The position and shape of the peaks give insight into the specific electrochemical processes, including redox reactions, phase transitions, and adsorption/desorption processes, among others. This plot has numerous applications in battery diagnostics including lifetime prediction, failure analysis, and degradation analysis.



11.8 Mott-Schottky Plots [Serial ($1/F^2$ vs V) and Parallel ($1/F^2$ vs V)]

In a Mott-Schottky plot, the reciprocal of the square of capacitance $\frac{1}{C^2}\left(\frac{1}{\mu F^2}\right)$ is plotted against the working electrode potential (V). The capacitance is calculated from the impedance data using an equivalent circuit model where the capacitor is either in series with (Serial) or in parallel with (Parallel) a resistor.



The equations used for both preset calculations are given below, in units of $\left(\frac{1}{\mu F^2}\right)$.

Serial Capacitance:

$$\frac{1}{C^2} = \left(2\pi f Z'' \times 10^6\right)^2$$

Where *f* is the frequency (Hz) and Z" is the imaginary impedance (Ohms).

Parallel Capacitance:

$$\frac{1}{C^2} = \left(\frac{2\pi f |Z|^2}{Z''} \times 10^6\right)^2$$

Where |Z| is the modulus of the impedance (Ohms).

This plot is commonly used to calculate flat band potential of a semiconductor/electrolyte interface. It is one of the key parameters used in the study of semiconductor electrochemistry.

12 Troubleshooting Squidstats and Common Questions

12.1 General Troubleshooting

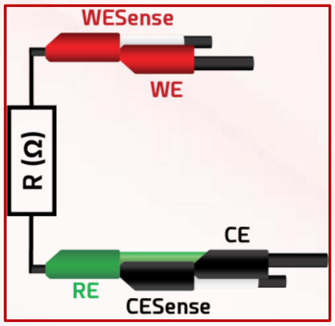
If a Squidstat is not behaving as expected, the problem might be with the Squidstat or with the experimental setup. Below is a list of common issues and solutions. If further support is needed, contact Admiral Instruments by e-mail at support@admiralinstruments.com or by phone at +1 (480) 256-8706 (calls are answered Monday through Friday from 9:00 A.M. to 5:00 P.M. GMT/UTC-07:00).

To initiate the troubleshooting process, please email support@admiralinstruments.com with the following information:

1. The raw data .csv file(s) associated with the issue
2. "Experiment settings and notes.txt" file associated with the raw data .csv file(s) above
3. The log file(s) generated during the same timeframe this experiment was initiated (the log files are by default located in the documents folder: Documents -> Admiral Instruments -> Logs)
4. A picture of the experimental setup, if possible

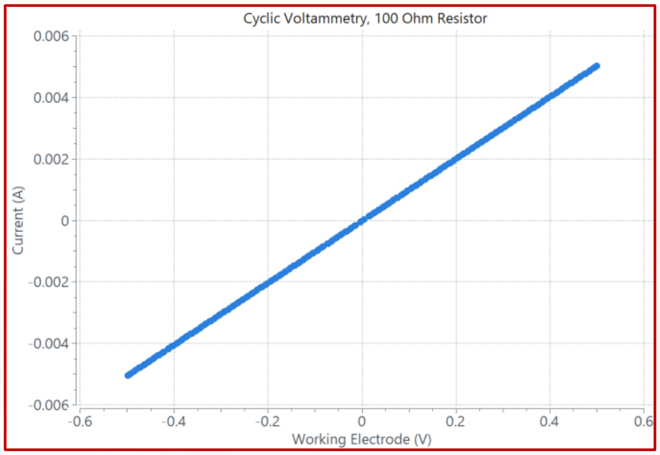
This information will be the best starting point to determine a solution for any specific issue.

12.1.1 Collected data is not within expectations



If collected data is not within expectations, there may be an issue with the Squidstat, the Squidstat cable, or the electrochemical cell. First, double check that all leads are connected properly, and that all electrical connections are secure. To verify the Squidstat cable is the issue, please follow the instructions outlined in the application note Testing the Integrity of a Squidstat Potentiostat Cable.

To check if the Squidstat is the issue, disconnect the leads from the electrochemical cell and connect to a test object of known value. This can be a dummy cell such as the InkBoard provided by Admiral Instruments with all potentiostat purchases (see **Chapter 14**) or a discrete resistor. If using a discrete resistor, connect the working electrode clip and working electrode sense clip to one side of the resistor. Next, connect the reference electrode clip, counter electrode clip, and counter electrode sense clip to the other side of the resistor, as demonstrated in the lead connection diagram shown above. Chronopotentiometry or chronoamperometry are experiments to complete with this setup. The voltage or current response should equal the expected value calculated from Ohms law ($V=iR$). For example, chronopotentiometry of a 100 Ω resistor with the current setpoint equal to +5 mA should return a voltage response of +0.5 V. For chronoamperometry of a 100 Ω resistor, a voltage of +0.5 V should return a current response of +5 mA.



Cyclic voltammetry (CV) can also be performed on a test object of known value to check for issues with instrumentation. Choose parameters that do not exceed the instrument limits or the safety limits of the test object. The resulting current vs voltage graph should be linear, and the forward and reverse sweeps should overlap each other perfectly, as shown in the plot to the left. The maximum current should agree with the value calculated from Ohms law ($V = iR$) within the accuracy of the current range. The maximum current is independent of scan rate. The resistance can be calculated from the slope of the curve and should be within the accuracy of the resistor. The example CV in the figure to the left was conducted on a 100 Ω resistor with the potential limits equal to -0.5 and 0.5 V.

If the response is not as expected, repeat the experiment with a different resistor or test object. If the problem persists, repeat the experiment with a different cable (if available). If the problem goes away when using a different cable, the cell cable needs to be replaced. After receiving a new cell cable, the Squidstat Support Utility software (see **Section 13**), will be required to reprogram the calibration constants for select models. If the problem persists when using a spare cable, the Squidstat may need to be serviced or replaced.

12.1.2 Squidstat is plugged in but does not power on

The source of this issue can vary between models. In some cases, following the protocol to reset the instrument, **Section 6.2** (also below), can resolve the issue. A firmware update will take place if this is successful.

12.1.3 The real-time values displayed in the *Manual Control* tab are nonsensical

It is normal to see widely varying voltages within the compliance range when the leads are not connected to anything. If they are not within the ±12 V compliance range of the Squidstat Plus, or the ±10 V compliance range of the Squidstat Prime and Squidstat Solo, the Squidstat likely needs the calibration constants reprogrammed into the unit. Please see **Section 13** on how to recalibrate a Squidstat using the Squidstat Support Utility.

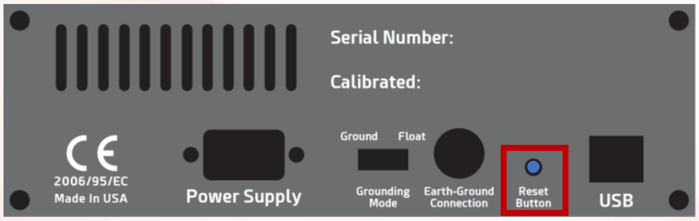
12.1.4 The SUI is frozen or crashes after starting an experiment

The most common reason the software freezes or crashes is a result of too many datapoints on the graph and the computer does not have enough RAM. The minimum amount of RAM recommended is 4 GB. If your computer has less than 4 GB, lower the sampling rate of the experiment to generate fewer datapoints, or switch to a computer with more RAM.

12.1.5 Squidstat hardware and software won't connect or sync

This issue can be resolved by resetting the Squidstat. Instructions for resetting a Squidstat: Solo, Prime, Plus, Penta, Decka, or Venta are below. Squidstat Cyclers and Customized Squidstat Cyclers do not include a reset button as these models will never require a reset. Unlike the firmware for Squidstat potentiostats, the Cycler firmware prevents the instrument from booting with corrupted or incomplete firmware, or booting without permission from the software, eliminating all potential causes which may require a manual reset.

1. Power off the Squidstat.
2. Ensure the power supply is plugged in, the USB connection is plugged in, and the Squidstat software is open on the computer.
3. Press and hold the blue reset button located on the back of the instrument (outlined in red in the diagram below), and while continuing to hold down the reset button, press the front power button to turn on Squidstat.



4. If successful, a firmware update will begin automatically in the software. Release the reset button.

12.1.6 There is unexpected noise observed on one or more channels

Noise can be caused by a diverse number of issues. Below are some of the most common causes of noise.

Check if there is a good, continuous electrical connection between the cable leads, the electrodes, and the electrolyte. Loose wires, poorly soldered connections, or corroded alligator clips can lead to noise. If using an alligator clip, very gently tug on the clip. If it slides, adjust the position of the clip. Sandpaper can be used to freshen up the connection points on old alligator clips or remove corrosion.

Check to see if other nearby electronic devices may be causing the noise, especially those plugged into the same outlet or power strip as the Squidstat (for example a lamp or other lab instruments). Try unplugging the device suspected of causing interference to see if the noise disappears. If the device causing the interference cannot be moved elsewhere, plug the Squidstat into a different power outlet.

If possible, use a Faraday cage to shield the cell from outside noise. Both the cell and the connections between the cable leads and the electrodes should be inside the Faraday cage. This is especially important when measuring very low currents or voltages or high impedances.

12.1.7 The SUI stops immediately after starting an experiment

This can be caused by several issues.

- This might be due to improper connection of the cell cables. Please make sure that the electrode clips are connected properly and securely to their respective electrodes.
- The Squidstat will immediately stop if the current and/or voltage are out of the specified range or instrument limits. This can happen due to several reasons:

- Entrapment of air or gas bubbles during the experiment. Find the bubbles and remove them.
- The electrodes are not properly immersed in the electrolyte solution.
- The electrode clips are not properly engaging the electrode termini. Make sure the clips are corrosion-free and secure.
- The response of the electrochemical device under test is outside of the setpoint or measurement range of the Squidstat. The ***Manual Control*** tab can be used to apply various currents and voltages in real time to determine the response of an unknown cell. If the cell response to an applied potential or current is out of range, please adjust the experimental settings accordingly or use a potentiostat with a higher current and/or voltage limit. Detailed hardware specifications for all Squidstats can be found on the Admiral Instruments website.

12.1.8 Current spikes are observed while running voltammetry experiments

This is most likely due to auto-ranging. In the parameters list under the **Current Ranging** section, select the option for *Maximum current expected* and enter the maximum current expected during the experiment. For more information on current ranging, see **Section 9.1**.

12.1.9 Voltage and current plateaus during a voltammetry experiment

If the voltage plateaus before reaching the potential limit during a voltammetry experiment, the compliance voltage of the instrument is most likely being exceeded. The compliance voltage is the maximum allowable voltage output, combining both working and counter electrode voltages as well as output current. To confirm, check if the counter electrode potential is at or near the ±12 V compliance range of the Squidstat Plus or the ±10 V compliance range of the Squidstat Prime and Squidstat Solo. Compliance voltage issues can only be resolved by changing the design of the electrochemical cell. Here are some common suggestions:

1. If the electrolyte is particularly resistive, the potentiostat must apply a higher voltage to the counter electrode to overcome the *i*R drop of the solution between the WE and the CE. Reduce the distance between the working and counter electrode to reduce the *i*R drop.
2. Increase the conductivity of the electrolyte.
3. Increase the size of the counter electrode. As a general rule, the counter electrode area should be at least 3x larger than the working electrode area.
4. Reduce the size of the working electrode.

12.1.10 Data collection appears to have stopped but the SUI can still be interacted with

There are a few possible causes for this issue:

- The computer fell asleep. In this circumstance, the pause/resume function no longer works and data is no longer being collected. Check that the computer Power and Sleep settings are set to never fall asleep and that screensavers are disabled.
- The connection between the computer and the data file save location was severed for any period of time. Even if the connection is lost and subsequently re-established, data collection will **not** resume. All experiment files should always be saved to the local main hard drive, as opposed to an external hard drive/USB or a network cloud drive requiring an internet connection.

- EIS at low frequencies can take a long time to complete, which can make it seem like the software stopped collecting data when the experiment is still active. The last recorded frequency is always displayed under the "Real Time Values" header to the left of the graph in the ***View Data*** window. If the last reported frequency is lower than 1 Hz and the LED power indicator on the Squidstat is blinking, the experiment is still active.

12.1.12 Adjusting computer sleep settings for long-duration experiments

To ensure the connection is never lost between the Squidstat and the SUI, the computer Power and Sleep settings should be configured to never put the hard drive to sleep, and screensavers should be disabled.

12.2 Frequently Asked Questions

The following information below has been compiled to assist in common questions that arise as users begin to gather data and want to compare results between runs. General protocols for working with the graphing features are also added as a reference.

12.2.1 How can I see frequency data in a Nyquist Plot?

Select the ***Frequency (Hz)*** parameter from the drop-down menu of the ***Tooltip Label*** under Graph Options in the ***View Data*** control tab. Drag the crosshair cursor to a data point. The frequency corresponding to the data point will appear as Label below X and Y value in a yellow box:

12.2.2 How can I graph data from an old data file?

To open an old data file, click the **+** in the ***View Data*** control screen or click ***Add/Remove Dataset(s)*** under Graph Options to superimpose additional data files on a current dataset.

12.2.3 How can I overlay numerous datasets on top of each other within the same graph?

To overlay data files, choose the blue ***Add/Remove Dataset(s)*** box under Graph Options in the ***View Data*** control tab, click ***Add Dataset***, browse to the folder where the data file to be overlaid is located, select the data file, and open the file. The new overlaid data will appear in a different color than the previous one. It should be kept in mind that files from DC experiments cannot be overlaid over files from AC experiments. Data files can be superimposed on a plot that is showing real-time data of an ongoing experiment.

12.2.4 How do I remove unwanted traces from a plot?

Unwanted traces can be removed from a plot by clicking the blue ***Add/Remove Dataset(s)*** box under Graph Options in the ***View Data*** control tab, selecting the data file corresponding to the trace, and pressing ***Remove Dataset***. Multiple data files can be selected and removed at once. However, at least one data file needs to remain in the Loaded Datasets window that pops-up after clicking the ***Add/Remove Dataset(s)*** box. Data files can be added to a plot showing real time data. However, the real-time data cannot be removed from an active experiment plot.

12.2.5 How do I hide a trace or line from a graph?

A line or trace can be hidden from a graph by clicking the blue ***Edit Line Appearance*** box under Graph Options in the ***View Data*** control tab. Next, select the file to be hidden from the Dataset List by turning Curve Visibility off, and clicking ***Apply***. A trace showing real-time data can also be hidden. In this way, a trace can be temporarily removed from a graph without deleting the corresponding data file.

12.2.6 How can I zoom in/out on the graph?

To zoom in on the entire plot, press the circular button with the **+** sign at the right side of the graph. To zoom out the entire plot, press the circular button with the **–** sign on the right side. The plot can also be zoomed in and out by using the scroll wheel on the mouse. To bring the plot to its original size, press the circular button with crossed arrows on the right side of the graph.

12.2.7 How do I change the min and max values shown on each axis?

To change the maximum, minimum, and major unit of an axis, double left-click on the axis. A box will open where these values can be entered. The step size can also be changed in this menu. To apply the changes, hit the Enter key or click elsewhere on the screen.

12.2.8 How can I change the labels of an axis?

Unfortunately, the label of the axes cannot be changed in the current version of the SUI. For now, only the legend of the plot can be changed by clicking on the blue ***Edit Line Appearance*** button under Graph Options.

12.2.9 How do I change the title of a graph?

Double left-click on the default title of the graph and rename it.

12.2.10 How do I change the font size of the title and label of the axes?

Go to the ***More Options*** control tab, click Software Settings from the Options, and change the value of the Set Axis Label Font box.

12.2.11 How can I save a graph as an image file?

To save a graph as an image, press the circular button with a picture icon on the right side of the graph. The image will be saved as a PNG file.

12.2.12 How do I change the color and size of the series plotted on a graph?

To change the appearance of a dataset graphed on the plot, press the blue ***Edit Line Appearance*** box. Select the dataset to be changed in the Dataset List box. Under the box, various plot options are available to change the appearance of the plot. After changing the plot appearance, click ***Apply*** to confirm those changes or ***Cancel*** to reject the changes.

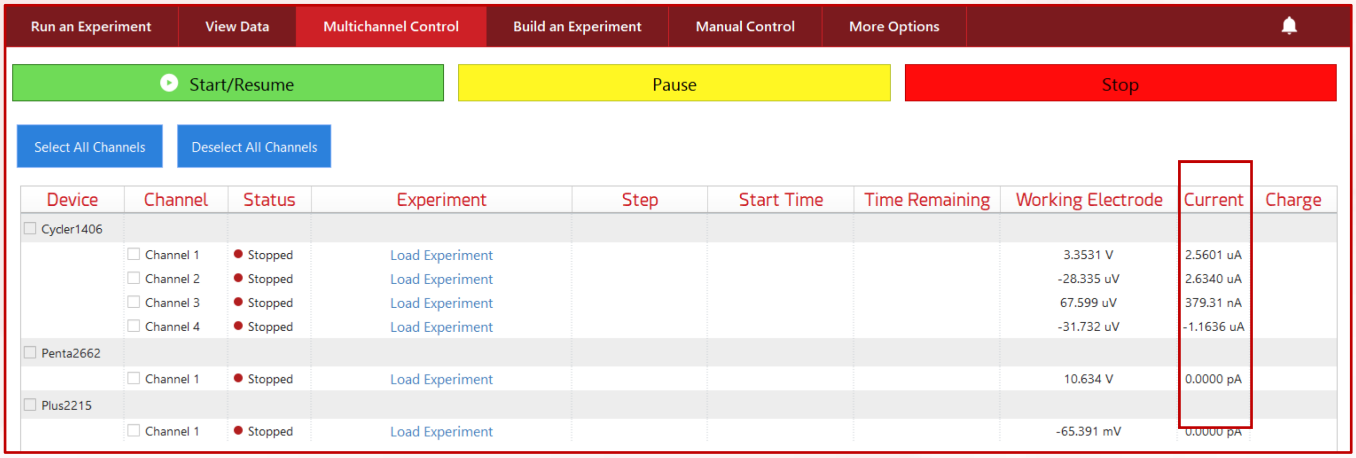
12.2.13 How can I view multiple graphs side-by-side?

To view multiple graphs side-by-side in the same window, click on the ***View All Graphs*** button on the top right corner of the ***View Data*** window. Although all the graphs are included in the same window, the size of the graphs is fixed. Therefore, a user must scroll down using the slide bar if there are more than 8 graphs. To view the graphs back in their windows, click on the maximize button on any of the graphs.

12.2.14 Why is the open circuit current different between Squidstat potentiostats and Cyclers?

In the ***Multichannel Control*** and ***Manual Mode*** windows, the real-time values for current and voltage are always displayed. When a channel is inactive or running an open circuit potential tile, Squidstat potentiostat models will always display "0 pA" for the current value, while the Squidstat Cycler will display a fluctuating value, typically no more than 10 µA. The open circuit current for the Cycler could be higher or lower depending on what is connected to that channel.

All potentiostats and Cyclers have a certain amount of leakage current flowing through the cables at open circuit. For simplicity, this value is set to display as 0 pA for the potentiostat models, because the leakage current is on a scale lower than the pA range and is therefore negligible. The leakage current for the Cycler is higher than the potentiostat models, at approximately 6 µA, whch may not be considered negligible for some users. Therefore, a value in the µA range is displayed for the Cycler instead of the value 0.



12.2.15 How often should my Squidstat be recalibrated?

Recalibration is generally unnecessary within the 2-year warranty period. Please refer to section 1.2 for a comprehensive overview of how to evaluate if your Squidstat requires recalibration, and the two types of recalibration services available to you.

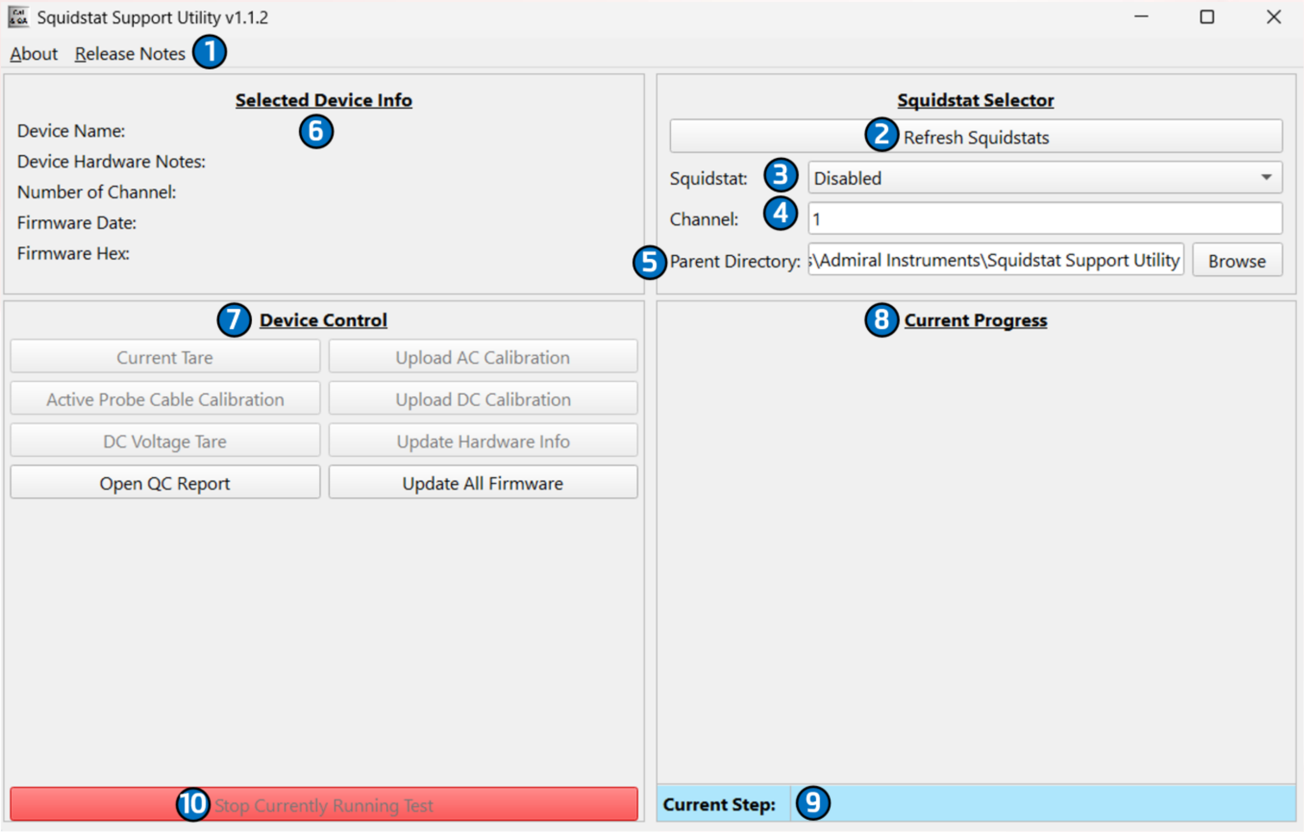
13 Squidstat Support Utility

The Squidstat Support Utility is a comprehensive tool designed to facilitate diagnostics, firmware updates, remote partial recalibrations, and hardware information management for Squidstat potentiostats. This tool does not support Squidstat Cyclers. It can be downloaded from the software page of our website. This manual outlines the supported functions and provides detailed instructions for utilizing each feature effectively.

13.1 Supported Functions:

1. Update firmware on the Squidstat if an automatic update is unsuccessful or if the firmware has been corrupted.
2. Perform a voltage and current tare to self-correct minor measurement offsets in calibration accuracy and perform a diagnostic quality check.
3. Re-upload original calibration files in the event that the calibration files have been deleted from the device due to malfunction.
4. Update hardware information programmed into the Squidstat (i.e. serial number, model number, etc.)
5. Perform a recalibration when replacing an old channel cable with a new cable (only applicable to the Squidstat Plus with serial numbers 1700 and above).

13.2 User Interface



Label	Description
1	File Menus. The **About** menu contains a User Guide for the support utility, and licensing information for Qt. The **Release Notes** menu contains release notes for all Support Utility releases.
2	Updates the list of all Squidstats connected to the PC running the Support Utility.
3	Dropdown menu listing the serial number of each connected Squidstat. Choose the desired unit from this list.
4	Text input for the channel number of the Squidstat chosen in the dropdown menu. Enter 1 for all single channel models.
5	All files generated while using the Support Utility will be written to this folder path. The default is `Documents\Admiral Instruments\Squidstat Support Utility`. To change the parent directory, click **Browse.**
6	Lists the Device Name, Device Hardware Notes, Number of Channels, Firmware Date, and Firmware Hex file of the unit selected in the Squidstat Dropdown Menu.
7	This menu lists all available functions. When a unit is selected, some or all functions will become available. Functions which cannot be used by the selected model will remain greyed out. A detailed description of each function is outlined in the following section.
8	When a function is initiated, messages indicating the status of the function will appear in the Current Progress section.
9	Displays the name of the currently active step during a test.
10	Stops active tests before their programmed conclusion.

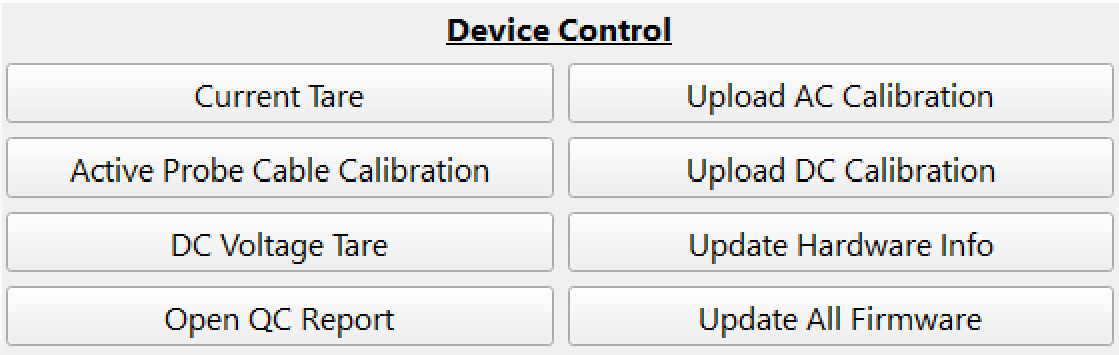
13.3 How to Use the Support Utility

Connect a Squidstat and Update Firmware

1. Ensure the instrument is powered on and connected to the computer via USB.
2. Click the **Refresh Squidstats** button in the Squidstat Selector section.
3. If the firmware is out of date, you will be prompted to update it. The firmware update process will begin automatically.
4. Once the instrument is connected, select it from the dropdown in the **Squidstat Selector** section.
5. Before running any calibrations, ensure you have the correct channel entered in the textbox labeled **Channel**.
6. Verify that the specified path for the calibration files is correct by clicking the **Browse** button and selecting the appropriate folder. This folder will be used to save any calibration files for the instrument.

Note: If the instrument is not detected, it is possible the firmware is corrupted. If this is the case, you will need to update the firmware manually via the **Update All Firmware** option in the **Device Control** section. If the firmware is still not updating when pressing **Update All Firmware**, power off the Squidstat, hold the reset button on the back or bottom of the instrument, and while still holding the reset button down then press the power button, see **Section 6.2**. Then press the **'Update All Firmware'** button again. This should allow the firmware to update.

13.4 Device Control Menu



13.4.1 Current Tare

This function is an offset correction tool, used to correct instances where measured DC current accuracy is beyond advertised specifications. It may not correct instances where the applied current deviates from the intended setpoint or where the measured current offset exceeds 3% of the current range. In those cases, please contact Admiral Instruments customer support.

This function consists of two steps, performed automatically:

1. Current Tare: A current tare will be performed and new calibration constants will be uploaded to the instrument.
2. Quality Check: This will check if the current tare function resolved the issue. Instruments will pass if the measured current deviates from zero by less than 0.1% of the range maximum when no load is connected. If it was successful, then the calibration report generated will list all current ranges as passing.

To perform this test, select the 'Run Current Tare' option in the Device Control Menu. When performing this procedure, you will be prompted to switch the load which is connected to the device. Please follow the instructions in the on-screen prompts to complete the diagnostic.

13.4.2 DC Voltage Tare

This function is an offset correction tool, used to correct instances where measured DC voltage accuracy is beyond advertised specifications. It may not correct instances where the applied voltage deviates from the intended setpoint or where the measured voltage offset exceeds 3% of the voltage range. In those cases, please contact Admiral Instruments customer support.

This function consists of two steps, performed automatically:

1. DC Voltage Tare: A voltage tare will be performed and new calibration constants will be uploaded to the instrument.

2. Quality Check: This will check if the voltage tare resolved the issue. Instruments will pass if the measured voltage reading deviates from zero by less than 1 mV when the working/working sense/reference are shorted together. If it was successful, then the calibration report generated will list all voltage ranges as passing.

To perform this test, select the **Run DC Voltage Tare** option in the Device Control Menu. When performing this procedure, you will be prompted to switch the load which is connected to the device. Please follow the instructions in the on-screen prompts to complete the diagnostic.

13.4.3 Active Probe Cable Calibration

This function is used to recalibrate any Squidstat Plus with serial numbers 1700 after receiving a channel cable replacement. These models require a calibration of the probe circuitry embedded within the ends of the sense leads in the cable. Other Squidstat models that do not have embedded circuitry at the ends of the cables do not need recalibration when receiving a new cable. **Do not use this function to recalibrate original cabling unless explicitly directed by Admiral Instruments.**

There are four major steps in this function, which run automatically:

1. AC Calibration with the following loads: 10 Ω, 100 Ω, 100 kΩ, no load (Open Circuit).
2. DC Voltage Tare with a 100 Ω load
3. AC Quality Checks with the following loads: 10 Ω, 100 Ω, and 100 kΩ.
4. DC Voltage Tare Quality Check with a 100 Ω load.

When connecting the leads to the 100 kΩ resistor load, make sure to attach the grounding cable (provided with all shipments) from the female 4 mm banana socket on the back of the instrument to the GND terminal on the InkBoard for proper shielding!

To perform the new cable calibration procedure, select the **Run Cable Calibration** option in the **Device Control** section. When performing this test, you will be prompted to switch the load which is connected to the device. Please follow the instructions in the on-screen prompts to complete the diagnostic. For a more detailed explanation of this function, please read the application note Calibrating a Replacement Squidstat Active Probe Cable.

13.4.4 Open QC Report

This function opens the Quality Control (QC) report generated after performing a Current Tare, DC Voltage Tare, or Active Probe Cable Calibration.

13.4.5 Uploading DC Calibration Data

This will upload DC Calibration files to any Squidstat potentiostat. Please contact Admiral Instruments for access to these files before beginning this procedure. To upload the DC data, click **Upload DC Calibration**. Navigate to the folder containing the calibration data and select the file "cal.csv". You should get confirmation that the DC data was uploaded in the **Current Progress** section of the Support Utility window.

13.4.6 Uploading AC Calibration Data

This will upload AC Calibration files to any Squidstat potentiostat. This only applies to the Squidstat Plus, Penta, Decka, and Venta models. Please contact Admiral Instruments for access to these files before beginning this procedure. To upload the AC data, click **Upload AC Calibration**. Navigate to the folder containing the calibration data and select the folder itself - do not select individual files. You should get confirmation that the AC data was uploaded in the **Current Progress** section of the Support Utility window.

13.4.7 Update Hardware Information

The following data can be updated with this function:

1. Serial number. Please note that the proper prefix (Plus, Prime, Solo, etc.) must be included in the new Serial Number along with the 4-digit model number. Examples include Plus1234 or Prime5678. Any deviation from this format may cause malfunctions.
2. Hardware model designation. The hardware model can be updated using the dropdown box Labeled 'Hardware Model' in the 'Update Hardware Information' menu. This function will only need to be used in very rare circumstances. Improperly updating the hardware model may cause malfunctions.


Unless instructed by Admiral Instruments, do not update the hardware information of your instrument. If done improperly, malfunctions could occur.

13.5 The Quality Check Report

An example QC report for a Squidstat Plus is shown on the following page. The rightmost column indicates whether the unit passed or failed a specific test.

DC Voltage Tare Accuracy reports the zero-offset measurement error as a voltage and as a percent error of the voltage range. The percent error is calculated according to the following equation:

$$Zero\text{-}Offset\ Measurement\ \%\ Error = \frac{|Zero\text{-}Offset\ Measurement\ Error\ (V)|}{V_{max}\ Range\ (V)} \times 100\%$$

The V_{max} Range is the maximum voltage in that range. For example, V_{max} for the 5 V range would be 5 V. If the unit supports Active Probe Cable Calibration, these results are included in that section of the report.

AC Current Accuracy Calibration reports Pass/Fail for each calibration and quality check test.

DC Current Tare Accuracy reports the zero-offset measurement error as a current and as a percent error of the current range. The percent error is calculated according to the following equation:

$$Zero\text{-}Offset\ Measurement\ \%\ Error = \frac{|Zero\text{-}Offset\ Measurement\ Error\ (A)|}{I_{max}\ Range\ (A)} \times 100\%$$

The I_{max} Range is the maximum current in that range. For example, I_{max} for the 100 mA range would be 0.1 A.

Recalibration Report

Model:	Squidstat Plus 2.4
Serial Number:	Plus1894
Recalibration Date:	08/17/2023

Channel 1

Active Probe Cable Calibration

DC Voltage Tare Accuracy

Range	Zero-Offset Measurement % Error	Zero-Offset Measurement Error (V)	Pass/Fail?
10V	0.00065%	0.000065	Pass
5V	0.00055%	0.000027	Pass
1V	0.00070%	-0.000007	Pass
500mV	0.0015%	-0.0000076	Pass

AC Current Accuracy

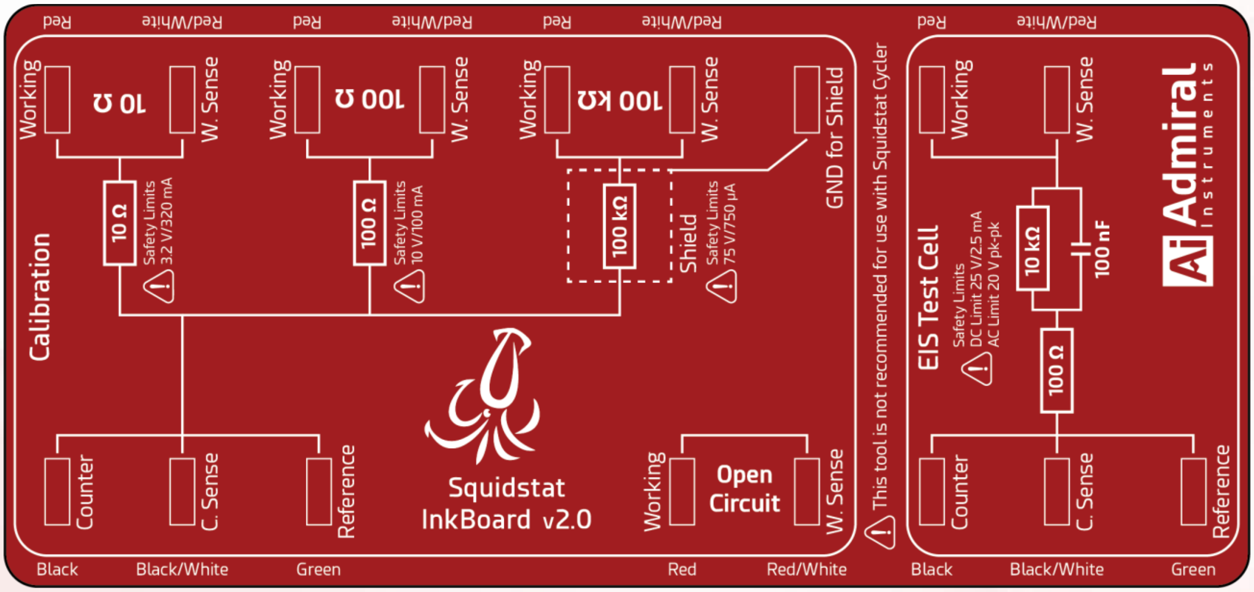
Sweep	Pass/Fail?
Calibration	
Open Leads	Pass
10 Ohm	Pass
100 Ohm	Pass
100 kOhm	Pass
Quality Check	
1A	Pass
100mA	Pass
5mA	Pass
1mA	Pass
100µA	Pass
10µA	Pass
1µA	Pass
100nA	Pass

DC Current Tare Accuracy

Range	Zero-Offset Measurement % Error	Zero-Offset Measurement Error (A)	Pass/Fail?
1A	0.0011%	0.000011	Pass
100mA	0.000033%	-0.000000033	Pass
5mA	0.00079%	0.000000039	Pass
1mA	0.0021%	0.000000021	Pass
100µA	0.0023%	2.3E-09	Pass
10µA	0.0021%	2.1E-10	Pass
1µA	0.0042%	4.2E-11	Pass
100nA	0.0047%	4.7E-12	Pass

14 Squidstat InkBoard

The Squidstat InkBoard is a Printed Circuit Board (PCB) used for calibration and troubleshooting Squidstat potentiostats. A diagram of the board is shown below.



There are five total circuits on the InkBoard, each with five connection terminals. The connection terminals are labeled with the name and colors of each lead on all Squidstat potentiostat cabling. The Inkboard is not intended to be used with the Squidstat Cycler. The AC current response from the EIS test circuit falls well below the accuracy specifications of the Cycler's lowest current range. However, the 10 Ohm and 100 Ohm resistors can be used for testing the Cycler. To connect the Cycler to the discrete resistors on the InkBoard, connect the equivalent leads outlined in **Section 7.3** to the appropriate terminals. Note that the Cycler has no equivalent to the counter sense lead, so no lead should be connected to that terminal.

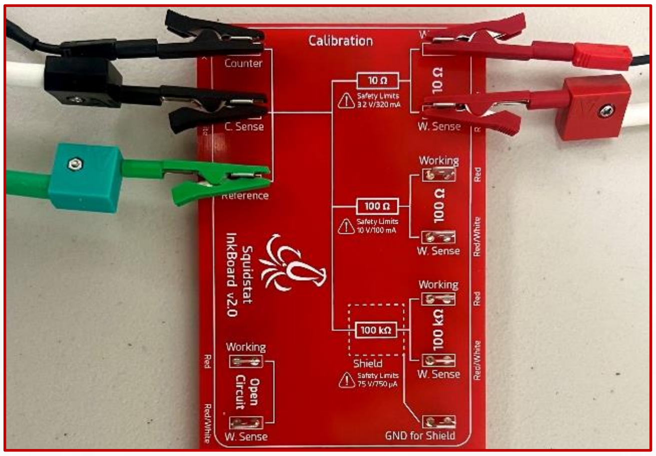
There are two sections on the InkBoard: A calibration section containing four circuits, and an EIS Test Cell containing one simplified Randles circuit. Both are described in the next two sections.

> The current and voltage safety limits are provided next to each circuit. Do not exceed these limits, or you could permanently damage the components!

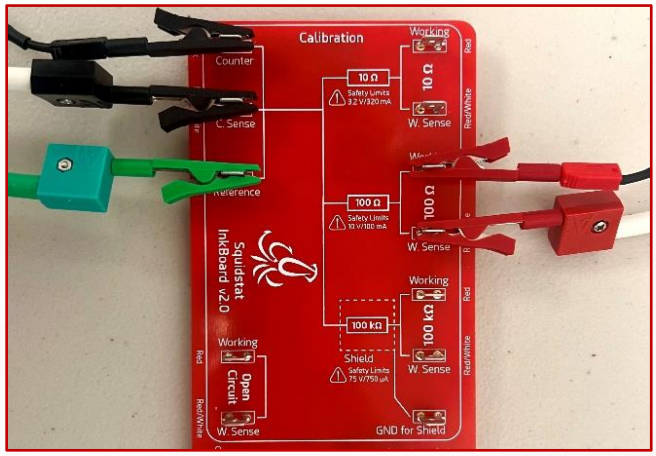
14.1 Calibration

There are four total circuits on the calibration section of the InkBoard: a 10 Ω resistor, a 100 Ω resistor, a 100 kΩ resistor, and an open circuit (no load) configuration.

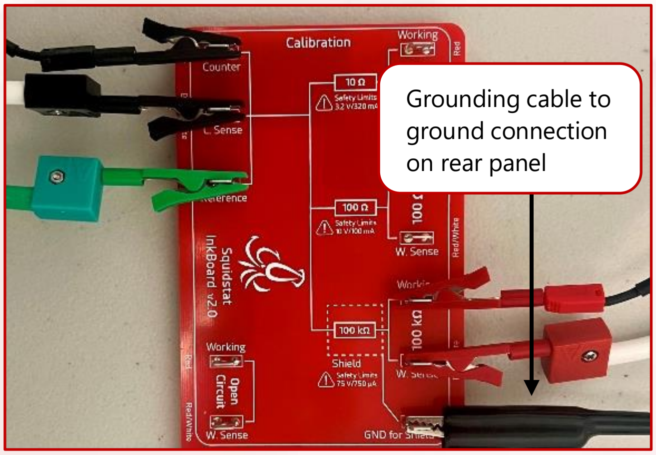
To switch between circuit configurations, move the working (red) and working sense (red/white) leads to the appropriate terminals while keeping the counter (black), counter sense (black/white), and reference (green) leads in the same position. The figure below shows the lead connections for each resistor and the open circuit (no load) configuration:



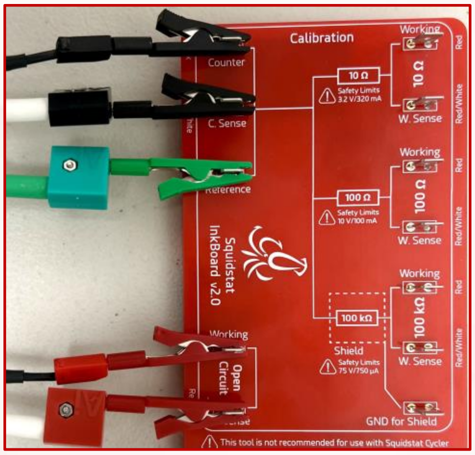
10 Ω



100 Ω



100 kΩ



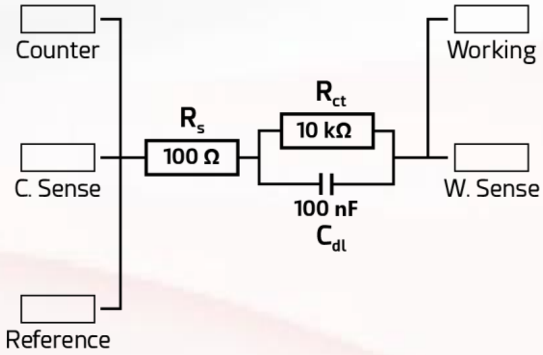
Open Leads

The resistor circuits can also be used to test the operation of any Squidstat. The current and voltage response to an applied voltage or current, respectively, should follow Ohms Law, V = *i*R. See **Section 12.1.1** for more information about checking Squidstat operation with resistors.

Each resistor has a tolerance of 1%.

14.2 EIS Test Cell

The EIS Test Cell is a simple Randles circuit, an R(RC) equivalent circuit for a basic electrochemical system. The circuit consists of the solution resistance (**R_s**) in series with the charge-transfer resistance (**R_{ct}**) and the double-layer capacitance (**C_{dl}**). The figure below shows the circuit and the values of each component, as well as the connection points of each lead.



Recording a potentiostatic EIS spectrum is one way to check the EIS performance of a Squidstat potentiostat. If a unit has issues, the results will be dramatically different than the example data provided on the following page, or the experiment will not run at all.

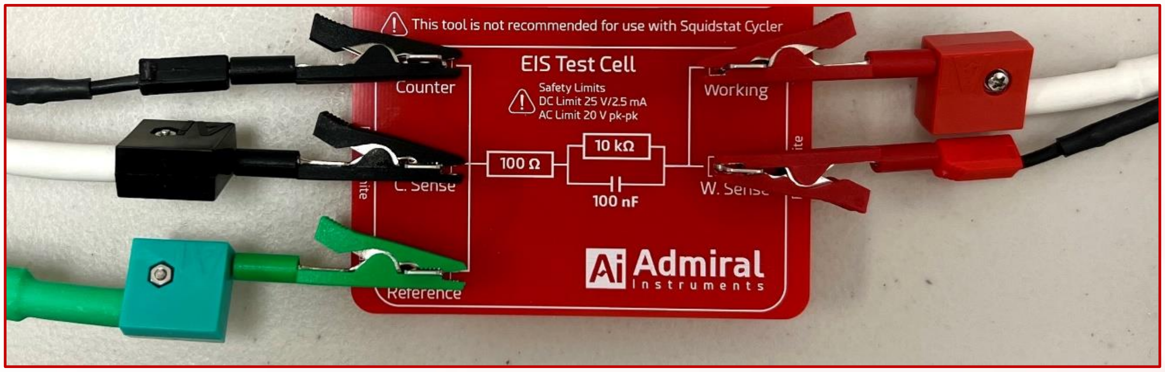
While visual inspection is usually sufficient to check if the system is working properly, equivalent circuit modeling can also be performed. The tolerance of all components is 1%, so the calculated values for the fit should fall between the following:

R_s – between 99 Ω and 101 Ω

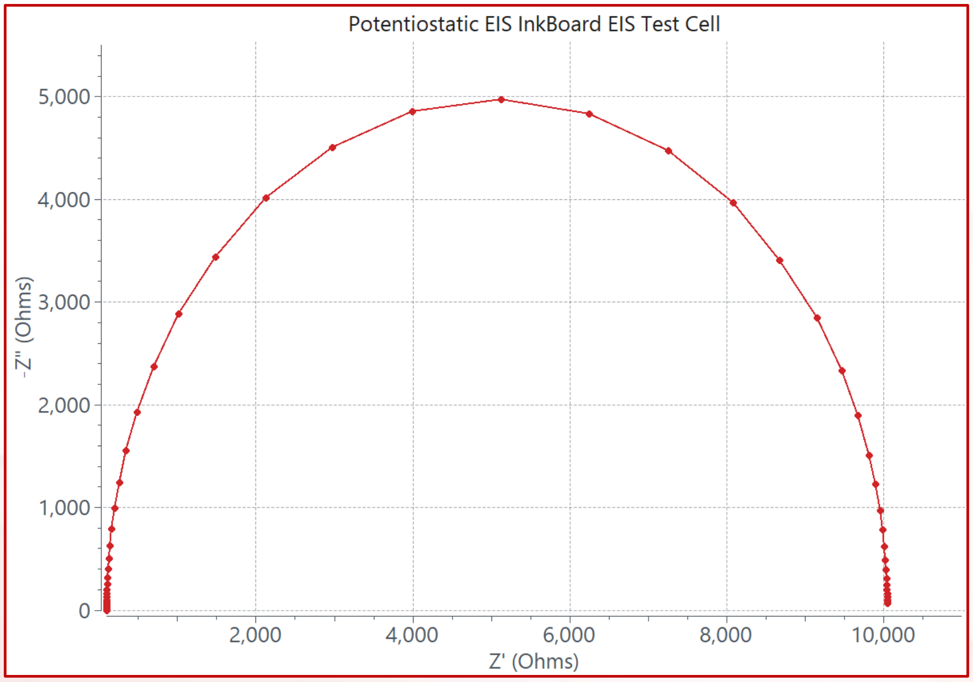
R_{ct} – between 9.9 kΩ and 10.1 kΩ

C_{dl} – between 99 nF and 101 nF

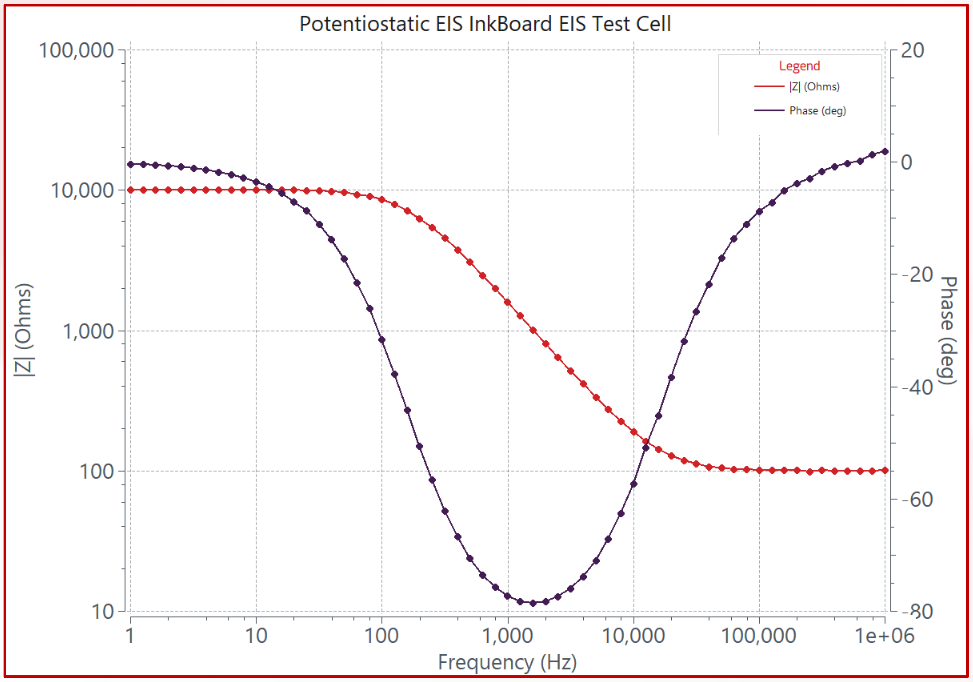
The figure below shows how to connect a Squidstat Potentiostat to the EIS Test Cell on the InkBoard.



14.2.1 Example Potentiostatic EIS Data



Nyquist Plot



Bode Plot

Sampling Parameters

Starting Frequency: 1 MHz

Ending Frequency: 1 Hz

Steps per decade: 10

AC Amplitude: 10 mV

DC Bias: 0 V
with respect to: Reference

Appendices

A. Common Error Messages

#	Error Message	Definition	Solution
1	Error loading calibration data	Calibration data was corrupted or erased from the device.	Please contact us if you encounter this error.
2	The current has exceeded instrument limits!	The measured current has exceeded the limit of the instrument or fixed current range, causing the channel to shut off.	Ensure that the current response is within the instrument's specified limits or increase the maximum current expected.
3	*deviceName* Working Electrode is floating with respect to earth ground	The ground/float switch on the rear panel is in the "float" position. The working electrode is now grounded to the chassis instead of through earth ground.	Verify the rear panel switch position and adjust based on user needs.
4	Recovery data on the device was deleted because it was collected in a previous SUI version	This message appears after updating between certain software versions. In most cases, no actual data is being deleted.	No action is required.
5	Error while loading CSV: File name does not end in .csv	The file path exceeds the maximum path length allowed on Windows (260 characters).	Move or rename the file to reduce the path length and try loading the data again.
6	AC+DC Current Limit Exceeded	The combined AC and DC current has exceeded the limit of the Cycler channel, causing the channel to shut off.	Ensure the measured current remains within the cycler's specified limits, or place two Cycler channels in parallel to increase the maximum available current.
7	AC+DC Voltage Limit Exceeded	The voltage has exceeded the limit of the Cycler, causing the channel to shut off.	Ensure the measured voltage remains within the Cycler's specified limits.
8	Maximum Number of Experiment Nodes Exceeded Limit = Value	The custom experiment exceeds the instrument's allowable node capacity, listed below by model: Cycler: 160. Prime: 512. All other models: 2048.	Reduce the number of nodes in the custom experiment.

9	OCP Fault	The voltage of the device connected to the cycler exceeds the instrument's voltage limits.	Disconnect the device immediately to prevent damage.
10	Control Fault: Unable to maintain setpoint	Cycler is unable to maintain a current or voltage setpoint.	Check for and secure any loose connections in the setup.
11	Connection Fault: Compliance Voltage Reached w/ No Output Change	Cycler has reached the maximum allowable compliance voltage before reaching the desired current or potential setpoint output.	Check for and secure any loose connections in the setup.