Admiral Instruments

Squidstat Operating Manual
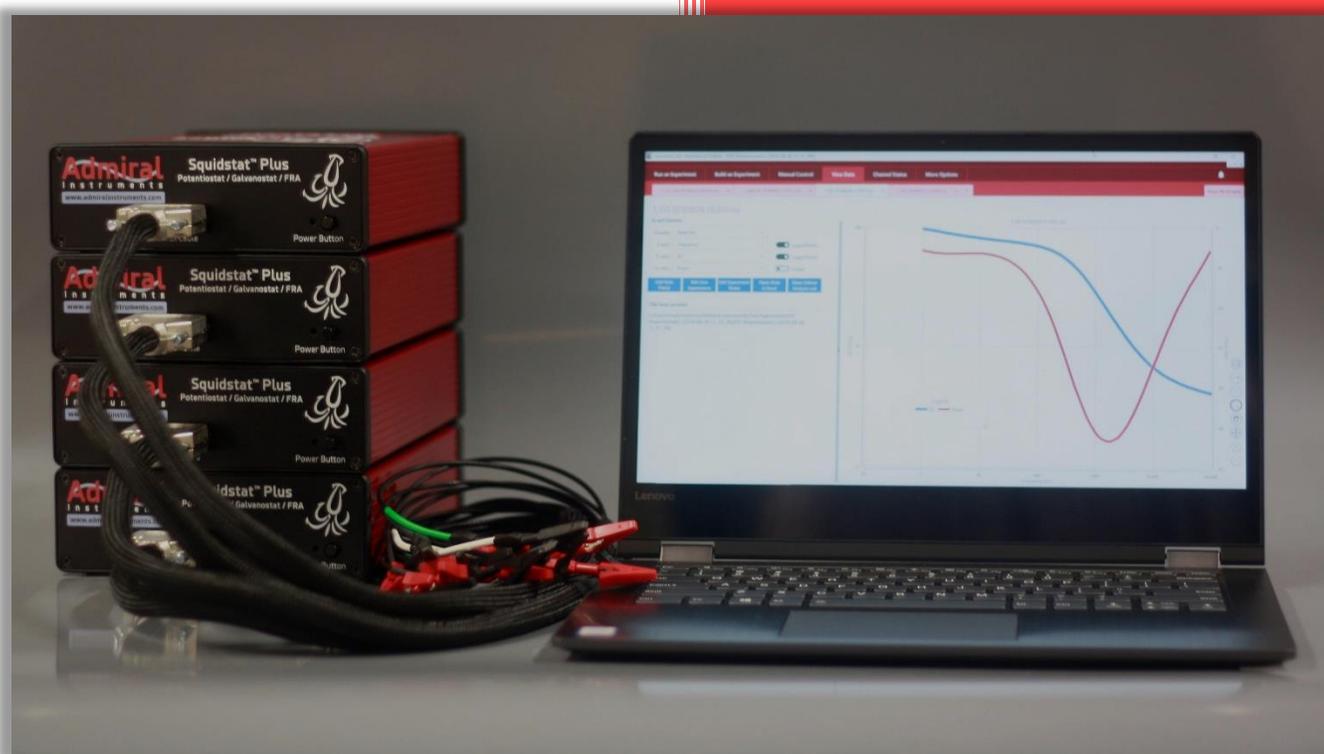
support@admiralinstruments.com

Call us: +1 480 256 8706

Squidstat Plus
Squidstat Ace
Squidstat Prime
Squidstat Solo

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1   Support Information and Warranty Statement

1.1   Support

Please email support@admiralinstruments.com or call **+1 480 256 8706** (calls answered Monday through Friday 9 am to 5 pm GMT/UTC-07:00). Responses to email and phone inquiries are guaranteed within one business day. Technical support services are always provided free of charge.

1.2   Warranty and Disclaimers

Our Warranty covers any defects in material and workmanship under normal use during the two-year period following delivery of products. During this period, Admiral Instruments will repair or replace, at no charge, Squidstat products or parts that prove defective because of improper material or workmanship under normal use and maintenance. Please note that all **Squidstat cabling, cable clips, and resistor assemblies are excluded from these warranty terms**. If replacement cables need to be purchased, please email support@admiralinstruments.com.

Although Admiral Instruments tests hardware and software compatibility and interoperability with PCs running Windows Vista and above or macOS 10.11 and above, we cannot guarantee that our products will work with all customer-supplied PCs or Apple computers.

The content here is presented error-free to the best of our knowledge, but please email support@admiralinstruments.com if you find error in the manual or have suggestions about improving how information is presented here. Information in this document is subject to change without notice.

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2   Safety

Users run the risk of electrical shock and injury if proper safety protocols are not followed. Squidstat products should only be opened by trained professionals or those who have been specifically tasked with performing remote maintenance by Admiral Instruments personnel. Please make sure the Squidstat is disconnected from a power source whenever removing the outer cover and use extreme caution after the cover is removed.

The internal parts do not need servicing by the user, except in specific situations under the guidance of Admiral Instruments personnel. If you suspect your Squidstat needs servicing and repair, please contact us at support@admiralinstruments.com or +1 480 256 8706.

We also recommend disconnecting the cell cables when moving the hardware to a new location because of the chance for the accumulation of static electricity. However, protection from electrostatic discharge is built into all Squidstats (up to 25 kV).

All Squidstat potentiostats are designed for use in standard lab environments. Use in field conditions is suitable, but our instruments are not specifically designed for use in field conditions. We recommend keeping the Squidstat housing away from sources of heat, water, and chemicals.

3   Unpacking

All the necessary hardware and software to operate a Squidstat are supplied by Admiral Instruments. Upon receipt of your Squidstat, please check the accompanying packing list to ensure all the parts on the packing list are present in the box. Please contact Admiral Instruments immediately if any parts are missing or damaged.

Squidstat Plus Package Contents
- Squidstat Plus Potentiostat (Qty. 1)
- Squidstat Power Supply (Qty. 1)
- Potentiostat-to-Power Supply Cable (Qty. 1)
- 6 ft. Power Cord Corresponding to Country of Operation (Qty. 1)
- 3 ft. Primary Cell Cable (Qty. 1)
- Cell Cable Alligator Clips (Black Clips Qty. 3, Red Clips Qty. 2)
- 6 ft. USB cable (Qty. 1)

Squidstat Ace Package Contents
- Squidstat Ace Potentiostat (Qty. 1)
- Squidstat Power Supply (Qty. 1)
- Potentiostat-to-Power Supply Cable (Qty. 1)
- 6 ft. Power Cord Corresponding to Country of Operation (Qty. 1)
- 3 ft. Primary Cell Cable (Qty. 1)
- Cell Cable Alligator Clips (Black Clips Qty. 3, Red Clips Qty. 2)
- 6 ft. USB cable (Qty. 1)

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Squidstat Prime Package Contents
- Squidstat Prime 4-channel Potentiostat (Qty. 1)
- 6 ft. Power Cord Corresponding to Country of Operation (Qty. 1)
- 6 ft. Cell Cable (Qty. 4)
- Cell Cable Alligator Clips (Black Clips Qty. 12, Red Clips Qty. 8)
- 6 ft. USB cable (Qty. 1)

Squidstat Solo Package Contents
- Squidstat Solo Single-channel Potentiostat (Qty. 1)
- 6 ft. Power Cord Corresponding to Country of Operation (Qty. 1)
- 6 ft. Cell Cable (Qty. 1)
- Cell Cable Alligator Clips (Black Clips Qty. 3, Red Clips Qty. 2)
- 6 ft. USB cable (Qty. 1)
- USB Drive Containing Squidstat User Interface software (Qty. 1)

4   Hardware Description

4.1   Squidstat Plus

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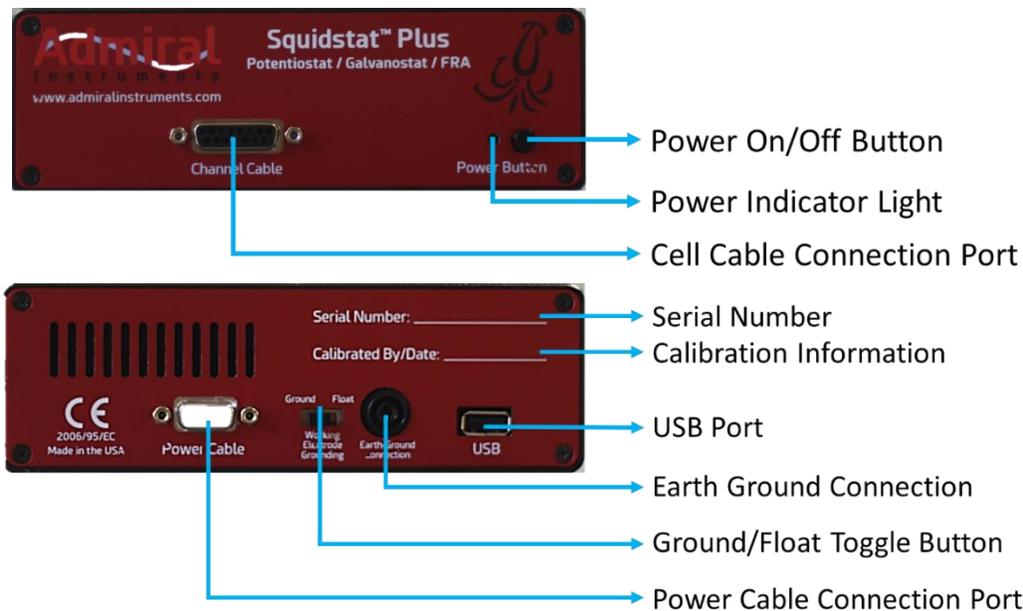
5   Installing the Squidstat User Interface (SUI) v2.0 Beta for the First Time

The Squidstat User Interface (SUI) v2.0 beta version is compatible with Windows Vista/7/8/8.1/10. An Apple-compatible version is currently in development. Please be sure to follow the step-by-step order carefully, otherwise the software may have difficulties recognizing the hardware during installation and installation must be repeated.

Step 1: Visit www.admiralinstruments.com/software and then click on the blue button labeled *Download SUI v2.0*.

Step 2: Clicking the blue button from the previous step brings users to a section titled *Squidstat User Interface Downloads*. From here, click on the folder icon labeled *SUI v2.0 Installation Package* for either Windows or Mac.

Step 3: A popup will appear to download the .zip file containing the installation package. To initiate the download to the location of your choice on your computer, click on *SUI Installation Package.zip*.

Step 4: Navigate to the *SUI Installation Package.zip* filesave location on your computer and then unzip/extract the contents. The contents of the unzipped folder will look like the screenshot to the right.
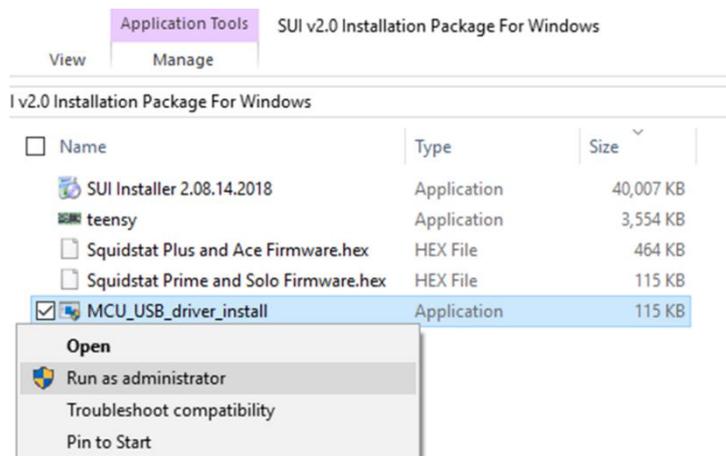
Step 5: Right-click on *MCU_USB_driver_install.exe* then select *Run as administrator*.

Step 6: Follow the on-screen instructions for driver installation. Once completed, you should see a popup stating *Driver INF Installed*.

Application Tools	SUI v2.0 Installation Package For Windows	
View Manage		

I v2.0 Installation Package For Windows

Name	Type	Size
SUI Installer 2.08.14.2018	Application	40,007 KB
teensy	Application	3,554 KB
Squidstat Plus and Ace Firmware.hex	HEX File	464 KB
Squidstat Prime and Solo Firmware.hex	HEX File	115 KB
☑ MCU_USB_driver_install	Application	115 KB

Open
Run as administrator
Troubleshoot compatibility
Pin to Start

Step 7: Go back to the original unzipped folder and run the *SUI Installer .exe* file. The file will have a specific version number corresponding to the release date. Disregard any prompts that may appear about PC protection and follow the on-screen instructions.

Step 8: After SUI installation is complete, launch the SUI from the Start menu or Desktop shortcut.

Step 9: Turn on the Squidstat and plug the USB connection to your computer (but it's okay if you turned it on already before this step). Then look for a notification on the bottom right toolbar about installing a new USB device. It may take a few minutes for the Squidstat to be recognized by the PC.

Step 10: Confirm that the Squidstat is connected to the computer by clicking the notifications icon on the top right and looking for the connection notification. Setup is now complete!

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6   Updating the Squidstat User Interface (SUI) v2.0 Beta to Newer Versions

Our team is routinely adding and refining features and content to the SUI, especially during our beta phase of development. Customers may choose to update the software as often as they wish by checking for new versions on the Admiral Instruments website. If you are experiencing any software issues, the first step we recommend is to install the latest SUI version. To do so, please follow these steps.

Step 1: Uninstall the older version of the SUI by using the *Add or Remove Programs* tool on Windows or the equivalent service on Mac.

Step 2: Visit www.admiralinstruments.com/software and then click on the blue button labeled *Download SUI v2.0*.

Step 3: Clicking the blue button from the previous step brings users to a section titled *Squidstat User Interface Downloads*. From here, click on the folder icon labeled *SUI v2.0 Installation Package* for either Windows or Mac.

Step 4: A popup will appear to download the .zip file containing the installation package. To initiate the download to the location of your choice on your computer, click on *SUI Installation Package.zip*.

Step 5: Navigate to the *SUI Installation Package.zip* filesave location on your computer and then unzip/extract the contents. The contents of the unzipped folder will look like the screenshot to the right.
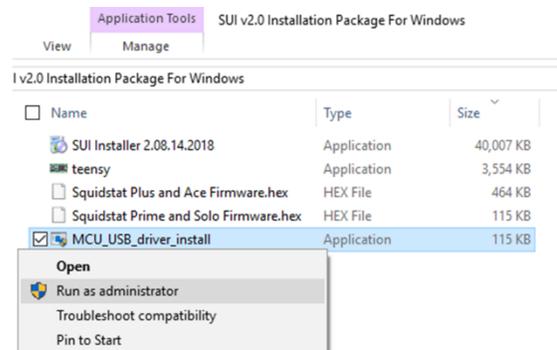
Step 6: Open the sub-folder called *SUI Installer for Windows* and run *SquidstatInstaller.exe* labeled with the most recent date in the filename. Disregard any prompts that may appear about PC protection and follow the on-screen instructions.

Step 7: After SUI installation is complete, launch the SUI from the Start menu or Desktop shortcut.

Step 8: Turn on the Squidstat and plug the USB connection to your computer (but it's okay if you turned it on already before this step).

Step 9: Confirm that the Squidstat is connected to the computer by clicking the notifications icon on the top right and looking for the connection notification.

7   Updating Firmware (Instructions for Squidstat Plus and Squidstat Ace)

When Admiral Instruments releases software updates, customers may also need to update the firmware version installed on their Squidstat(s). Users will know whether a firmware update is required if a notification appears in the SUI, or if directed to do so by Admiral Instruments. When directed to do a firmware update, please follow the instructions below if you are using a Squidstat Plus or Squidstat Ace.

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Step 1: Open the SUI and click on the *More Options* tab at the top. On the main tab panel at the top left, click More Options and select *Update firmware* from the list on the left side of the screen.

Step 2: Under the *Choose which device you want to update* menu, select the Squidstat unit to be updated.
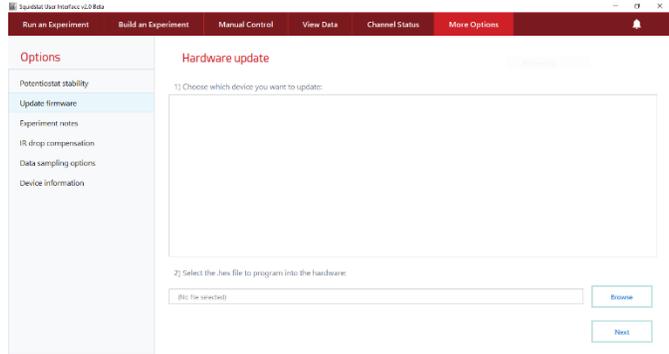
Step 3: Under the *Select the .hex file to program into the hardware* menu, click *Browse* and navigate to the folder containing the newest version of the SUI Installation Package downloaded from www.admiralinstruments.com/software.

Step 4: Select *Squidstat Plus and Ace Firmware.hex* file inside the folder labeled with the most recent date. Then click *Open*.

Step 5: Back to the SUI *More Options* menu, click "Next" on the bottom right of the screen and then wait for the progress bar to indicate that the update is complete. It is very important that no interruptions occur during the few seconds required for the update to finish.

Step 6: Confirm that the update was successful by checking for a notification on the notifications icon on the top right corner of the screen.

Step 7: Sometimes a notification will not appear even though the firmware updated successfully. In such cases, please try restarting the SUI and Squidstat.

8   Updating Firmware (Instructions for Squidstat Prime & Squidstat Solo)

When Admiral Instruments releases software updates, customers may also need to update the firmware version installed on their Squidstat(s). Users will know whether a firmware update is required if a notification appears in the SUI, or if directed to do so by Admiral Instruments. When directed to do a firmware update, please follow the instructions below if you are using a Squidstat Prime or Squidstat Solo.

Step 1: Find a screwdriver that will fit the screws on the Squidstat enclosure cover. With the Squidstat powered off, unscrew the screws and remove the top cover. Then, turn on the Squidstat and ensure it is connected to the computer via USB.

Step 2: Navigate to the folder containing the newest version of the SUI Installation Package downloaded from www.admiralinstruments.com/software.

Step 3: Run the application named *teensy.exe* contained in this folder.

Step 4: In the Teensy program window that appears, click *File->Open HEX File*, then navigate to the SUI Installation Package folder and select *Squidstat Prime and Solo Firmware.hex*. Then click *Open*.

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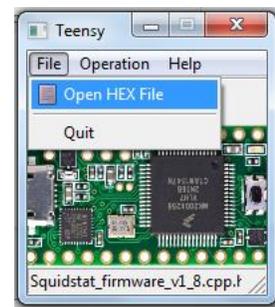
Step 5: Locate the microcontroller mounted on one of the circuit boards inside the Squidstat. It will have a micro USB cord plugged into it. Now press the white button on the microcontroller. A single light press should be all that is necessary. There's no need to hold this button down for more than a second.
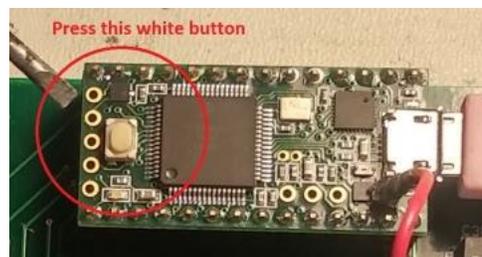
Step 6: After pressing the button on the microcontroller inside the Squidstat, a window should appear titled "Driver Software Installation" which will say device driver software is being found for an Unidentified Device. Wait for this window to show that the USB Input Device is ready to use, which may take several minutes. If such a window does not appear, the device driver software may already be installed so continue to the next step.
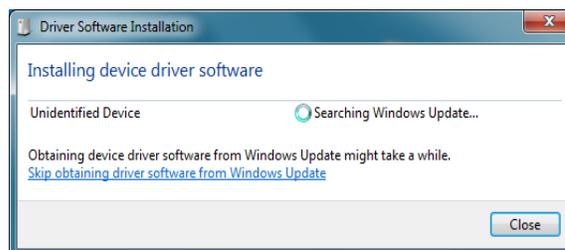
Step 7: Return to the Teensy program and click Operation->Program on the Teensy window. A progress bar should then briefly appear (for about 1 second) to indicate that the firmware has uploaded successfully.
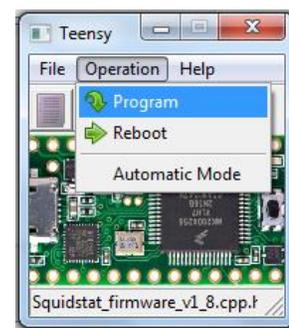
Step 8: The firmware is now updated, so exit out of the Teensy window and reattach the screws on the Squidstat cover.

Step 9: Confirm that the update was successful by checking for a notification on the notifications icon on the top right corner of the SUI program.

Step 10: Sometimes a notification will not appear even though the firmware updated successfully. In such cases, please try restarting the SUI and Squidstat.

9   Cell Connections

Any standard electrochemical system has at least three parts which are generally referred to as the 1) Working Electrode (WE), 2) Counter or Auxiliary Electrode (CE) and 3) an electrolyte. The electrons, or electronic current, flow between the WE and CE through an external circuit consisting of a potentiostat/galvanostat. Ions, or ionic current, flow between the WE and CE through the electrolyte.

Typically, voltage and current at the WE are of primary interest to apply and/or measure. The potentiostat/galvanostat ensures that the voltage and current applied at the WE are set according to the user's specification to the highest degree of accuracy possible. The CE completes the external circuit with the WE. Voltage and current are not applied at the CE.

To control the voltage at the WE independent of current, the Reference Electrode (RE) is introduced to the electrochemical system. Little or no current flows through the RE.

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All Squidstat cell cabling follows the wire and clip color coding scheme below. A color-coded legend is printed onto the top or front of each Squidstat for easy access. These electrode connections may be used individually by connecting the alligator clips, or numerous electrode connections can plug into each other by plugging the gold banana pins into the holes on the back of each red or black banana pin housing.
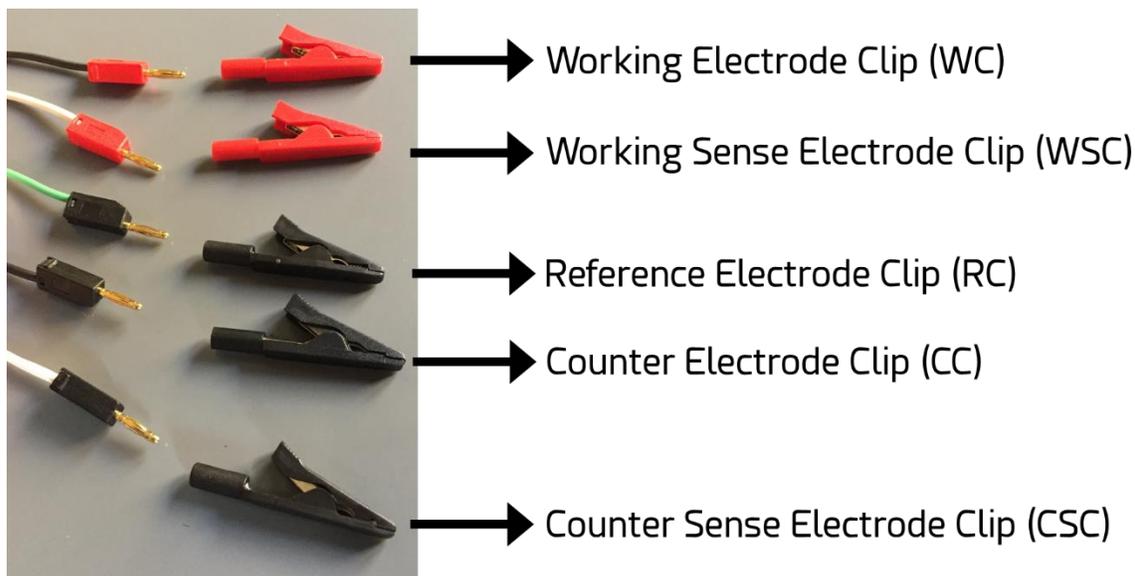
Red Clip / Black Wire: Working Electrode Clip (WC)
This is a current carrying connection. The WC is connected to the working electrode (WE).

Red Clip / White Wire: Working Sense Electrode Clip (WSC)
This is a voltage sensing connection. It is an integral part of the feedback loop. It can be connected anywhere along the path between the reference electrode and the working electrode, though normally it is connected directly to the working electrode using the stackable banana pins.

Black Clip / Green Wire: Reference Electrode Clip (RC)
This is a voltage sensing connection. It is the "zero" potential against which the working and counter electrode potentials are compared.

Black Clip/Black Wire: Counter Electrode Clip (CC)
This is a current carrying connection. CC is connected to the counter electrode (CE). The current flows only between WC and this electrode, CC.

Black Clip/White Wire: Counter Sense Electrode Clip (CSC)
This is a voltage sensing connection. It is connected anywhere along the path between the reference electrode and the counter electrode. Usually, it is connected directly to the counter electrode using the stackable banana plugs. However, since it is not a part of the feedback loop, it may be placed anywhere a voltage measurement point is needed.

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Users are welcome to label each individual wire with its corresponding electrode clip designation, or simply refer to the color-coded legend printed onto the front or top of each Squidstat enclosure.

9.1 Electrode Configurations

There are several ways to connect an electrochemical system to a potentiostat, depending on the number of electrodes involved in the experiment. Common configurations are described below.

9.1.1 Two-electrode Configuration

In this configuration, the WC and WSC are combined and connected to the WE assigned to the experiment. The CE, CSE, and RC are combined and connected to the CE assigned to the experiment. Since the potential of the WE is set with respect to the CE, the changes in the CE during the course of experiment affects the potential at the WE. However, this is also the simplest electrode configuration.
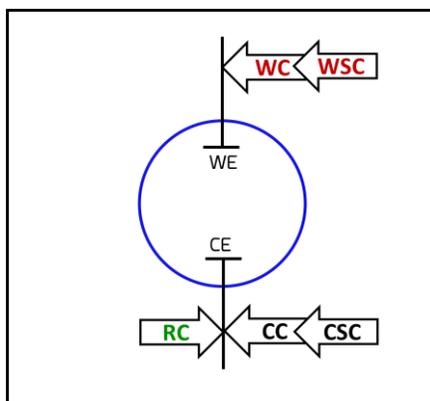
9.1.2 Three-electrode Configuration

This is the most popular configuration in physical and analytical electrochemistry. In this configuration, the WC and WSC are connected to the WE assigned to the experiment. The CC and CSC are connected to the CE assigned to the experiment. The RC is connected to the RE. Since the potential of the WE is applied or measured with respect to an independent RE, changes in the CE during the course of the experiment do not affect the potential at the WE.

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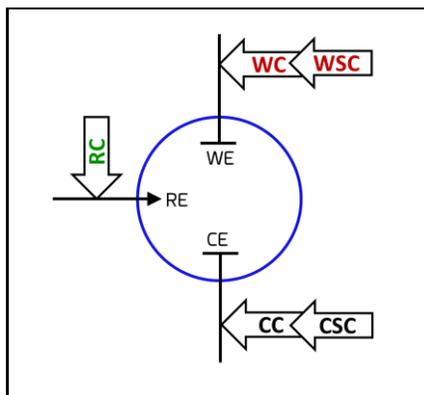
9.1.3   Four-electrode Configuration

In this configuration, the WC is connected to the WE, the CC and CSC are connected to the CE, the RC is connected to the first RE, and the WSC is connected to a second RE. This configuration is useful if a user wants to connect the WSC at a different point other than the WE. One reason to separate the WC and the WSC is to avoid inductive interference from the current flowing in the WC during alternating current measurements, for example electrochemical impedance spectroscopy (EIS). Another reason is to conduct a 'four-points resistance measurement'. It is important to remember that the potential measured at the WSC is fed into the potentiostat to control potential at the WE.
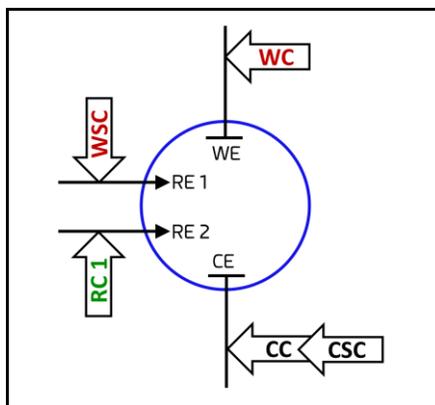
9.1.4   Five-electrode Configuration

In this rare configuration, the WC is connected to the WE, the CC is connected to the WE, the RC is connected to the RE, the WSC is connected to a second RE, and the CSC is connected to a third RE. Here, the WSC and CSC are attached independently from the WC and CC, respectively. This is useful if a user wants to connect WSC and CSC at different reference points. It is important to remember that the potential measured at the WSC is fed into the potentiostat to control the potential at the WE. However, the potential measured at the CSE is not fed into the potentiostat to set the potential at the WE or the CE. Therefore, the CSC is an extra sensing clip that can be attached to any arbitrary points to measure voltage. For example, a pressure transducer can be attached to the RC or to a ground and the CSC to monitor pressure in the electrochemical cell during an experiment.

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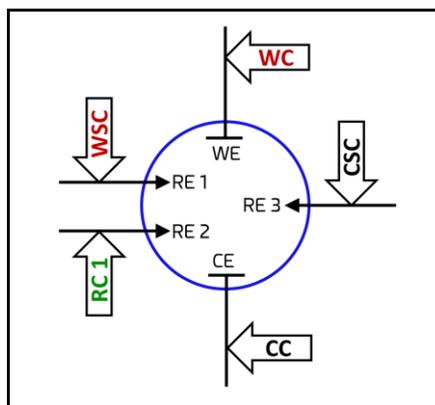
10 Squidstat User Interface (SUI) Main Screens

The top of the SUI consists of a red bar containing a collection of tabs to access various features and a notification log that appears on the right-hand corner.

10.1 Control Tabs

10.1.1 Run an Experiment

When opening the SUI for the first time, the *Run an Experiment* tab is selected by default. The purpose of this tab is to select and run pre-programmed electrochemistry experiments. Please remember that only parameters for an experiment can be entered, as the procedures for any of the experiments in the list cannot be modified in this tab.

Label	Description
1	List of experiments to choose from (both pre-programmed and custom user-designed)
2	Drop-down menu to select a set of experiments corresponding to a particular application
3	Search bar to select experiments by keyword
4	Select an experiment to view its description and edit the experimental parameters. After saving custom-built experiments created in the *Build and Experiment* tab, the newly-built custom experiment appears below the list of the pre-programmed common electrochemical methods. Such custom-built experiments are automatically added to the custom group and can be viewed as a group by clicking "**Custom**" in the search box.
5	Illustration of the waveform applied by the Squidstat for the selected experiment
6	Title and brief description of what the selected experiment does
7	Parameters for the procedures of an experiment are edited here (except custom experiments)
8	Parameters of the selected pre-programmed experiment that can be edited are displayed here. If a custom-built experiment is selected, two choices appear: "Edit Experiment" and "Delete

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	Experiment." "Edit Experiment" will take you to the screen in the Build an Experiment tab where you can modify the experiment. "Delete Experiment" deletes the selected experiment.
9	Lists the Squidstats, and their respective channels, available to run the selected experiment
10	List of Squidstats that are powered on and connected to the computer
11	Channels available on the connected Squidstats
12	Button to start the experiment. To run an experiment, select the device and the corresponding channel of the device to run the experiment under the Select Device/Channel on the bottom right corner of the screen and press Start the Experiment button. If an experiment is running, Pause Experiment and Stop Experiment buttons appear beside the related device and channel. The buttons can be used to pause/resume or stop experiments.

10.1.2 Build an Experiment

The purpose of this tab is to build a custom experiment. Please keep in mind that to run the custom-built experiments, first save them and then go to the Run an Experiment screen where they will appear below the list of the pre-programmed experiments in the manner they are saved.
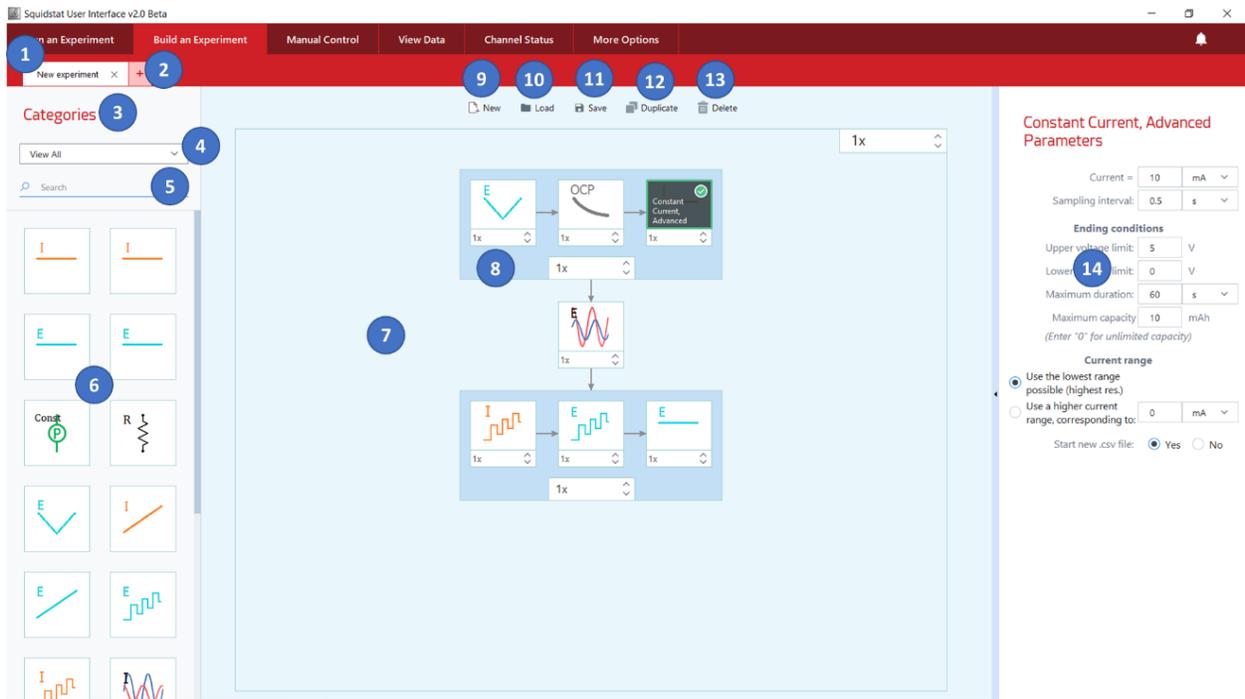
| Label	Description
1	Name of the experiment being built or edited. The default name "New experiment" can be changed by saving the experiment under a different name.
2	Open a new page to build a new experiment
3	Area for basic method tiles to be chosen to build an experiment
4	Drop-down menu of basic method tiles categorized based on their application. The same method tile can appear in more than one category.
5	Search bar to select method tiles by keyword

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6	List of all available method tiles
7	Drag, drop, and link method tiles from the Category panel to build custom experiments
8	The dark blue area forms the background of a row of method tiles. Left-click to highlight all of the method tiles of the same row, either to delete or duplicate the entire row.
9	Open a new page to build a new experiment
10	Button to reopen an old custom experiment to make modifications. On clicking, a dialogue box will appear with a reminder to save the experiment. A selection window will then appear with a list of old experiments. Highlight the experiment to be opened and press Open. You can also delete the experiment by pressing Delete. Press Cancel to go back to the previous page.
11	Button to save the custom-built experiment. On clicking, a new window appears where the name for the experiment and description for the experiment can be given.
12	Button to duplicate a method tile. This especially comes in handy when one wants to add a new tile while keeping same parameters of the old method tile. If a row has only one method tile, a new step tile will be duplicated in the next row below. If a row has more than method step tile, a new tile will be duplicated beside the existing step tile in the same row. An entire row can also be duplicated by highlighting the darker blue area.
13	Button to delete a method tile. Highlight the method tile or row of tiles before pressing delete.
14	The parameters related to a method tile are displayed here after a method tile that has been dragged and dropped into the blue area has been selected. The selected method tile will be highlighted in black.

10.1.3 How to Build Custom Experiments

The process of building custom experiments within the Squidstat User Interface is like fitting different pieces of a puzzle together. Different 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. Experiment tiles proceed consecutively from left to right, then top to bottom. To follow the sequence tiles within the experiment, simply follow the arrows from tile to tile.

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 parameters options on the right side of the screen. Detailed explanations of the parameters for each tile are available in later chapters of this manual.

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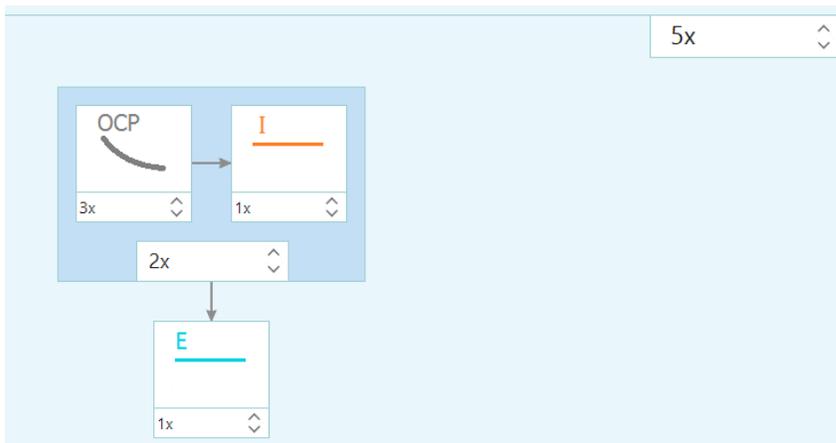
Example:

In the above example, the following experiment has been created:

1. The Squidstat will run the "OCP" tile 3 times
2. A constant current step will run once
3. The first two steps will run a total of two times, as per the "2x" on the bottom of the first row
4. A constant voltage step will run once
5. After completion of constant voltage (the "E" tile), the Squidstat will jump to "OCP" tile again and repeat the first four steps four more times, as per the "5x" on the top right corner

After all the method tiles have been arranged, and after all their parameters have been set, the next step is to save the experiment by clicking the "Save" button at the top. A popup will appear prompting users to type a name and accompanying description. Once saved, this custom experiment will appear in the "Run an Experiment" tab and can run on any applicable channel.

10.1.4 How to Modify or Delete Previous Custom Experiments

To reopen an old 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. To delete the experiment, click the **Delete Experiment** button in the same area. Alternatively, go to **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.

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10.1.5 Manual Control

In this screen, users can turn on any connected channel and toggle between open circuit mode and voltage or current setpoints that can be changed in real-time and run for indefinite periods.

Label	Description
1	Tab displaying the name and serial number of the Squidstat powered on and connected to the computer. A green dot appears beside the name if the Squidstat is actively recording data.
2	Red boxes to select channels of a Squidstat to run the manual experiment
3	Toggle button to switch between potentiostatic and galvanostatic modes. Corresponding parameters to set potential or current values, sampling interval, and current ranges appear on the right according to the mode selected. In potentiostatic mode, options to select auto range or custom current ranges are available. A user can enter new voltage or current set points without stopping the experiment.
4	Toggle button to switch open circuit mode on and off. To apply the set potential or current, switch the open circuit mode to the off position.
5	Real time values display. These values are displayed even the channel does not have an experiment running on it.
6	Clock showing the amount of time elapsed after the start of the manual experiment. This clock will pause if the user chooses to pause the channel in manual mode.
7	This section contains various options to manipulate the data displayed on the graph. Please see the sub-chapter about graphing options for the detailed description of the blue buttons.
8	Location of the saved data file. Click on this link to directly go to the folder of the saved file.
9	Data display from the selected channel running in Manual Control
10	Function buttons to further manipulate graphics of the plot. Please see the sub-chapter about graphing options for the detailed description of these buttons.

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10.2 View Data

This screen contains all of the graphs generated while running pre-built or custom experiments.

Label	Description
1	Tab containing the name of the device, the associated channel, and the name of the experiment. A green dot appears in the tab while the experiment is currently running.
2	Open a new graph tab to display previously saved data
3	Real time values display
4	Clock showing the elapsed time since the start of the experiment, the step of the experiment currently being run (when applicable), and the time elapsed during the step
5	Button to skip the step currently running. If a cyclic voltammetry (CV) experiment is running, by pressing this button, the Squidstat will reverse the scan direction and start the next scan from the potential limit of the current scan. If it is a chrono method, this button will end the step and start the next step in the queue. If it is an EIS method, this button will skip the measurement at current frequency and skip to the next frequency in queue.
6	This section contains various options to manipulate the data displayed on the graph. Please see the sub-chapter about graphing options for the detailed description of the blue buttons.
7	Location of the saved data file. Click on this link to directly go to the folder of the saved file.
8	Data display from the selected channel running the experiment
9	Button to view all graphs that are open in the tabs in the View Data window. Up to eight graphs can be viewed simultaneously. 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 manipulate graphics of the plot. Please see the sub-chapter about graphing options for the detailed description of these buttons.
11	Button to pause and subsequently resume the experiment
12	Button to stop the experiment before its programmed conclusion

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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		Description
Save graph image		Saves the graph image as a .png file
Show/hide legend		Display/hide the color-coded data legend
Show/hide gridlines		Add or remove the major gridlines to the graph
Zoom to selection		When this mode is selected, any part of the plot can be zoomed into. To zoom in, left-click and hold down the mouse button and drag the cursor to form a box over the area to zoom into.
Pan View		When this mode is selected, users can left-click and hold down the mouse button to freely move the plotted area of the graph. Users can also use the mouse scroll wheel to zoom in or out of the plot.
Reset Zoom		Resets the plot back to its original size after being zoomed or panned
Zoom in		Zoom in
Zoom out		Zoom out

10.2.2 Graph Options

More options to format graphs are available under **Graph Options**. These options are available under both **Manual Control** and **View Data** control tabs.

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Labels	Description
1	Drop-down menu to choose from various types of premade graph options.
2	Drop-down menu of available parameters to graph on the X axis. The axis can be changed from linear to logarithmic by toggling the switch to the right of the drop-down arrow.
3	Drop-down menu of available parameters to graph on the primary Y axis. The axis can be changed from linear to logarithmic by toggling the switch to the right of the drop-down arrow.
4	Drop-down menu of available parameters to graph on the secondary Y axis. The axis can be changed from linear to logarithmic by toggling the switch to the right of the drop-down arrow.
5	Button to add previously-saved data files to overlay multiple plots on top of one another
6	Button to open options to format the data displayed on the graph
7	Button to view the content of the "Experimental Settings and Notes" file where notes and parameters of the experiment are automatically saved at the beginning of the experiment. The contents of the file can be edited and saved within this window.
8	Button to open a data file as a spreadsheet in Excel
9	Button to open a data file in Zahner Analysis Lab for further analysis. This is an add-on program that is purchased separately.

10.2.3 Common Graphing Questions

How can I graph data from an old data file?
To open an old data file, click the tab with the plus sign in the **View Data** control screen or click the blue **Add Data Files** box under Graph Options.

How can I overlay numerous datasets on top of each other within the same graph?
To overlay data files, choose the blue **Add Data Files** box under **Graph Options** in the **View Data** control tab. The new overlaid data will appear in a different color than the previous one.

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 at the right side. The plot can be zoomed in and out also by just using the scroll wheel on the mouse connected to the PC. To bring the plot to its original size, press the circular button with crossed arrows on the right side of the graph.

How do I change the min and max values shown on each axis?
To change the maximum, minimum and major unit of an axis, 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.

How can I change the labels of an axis?
Unfortunately, the label of the axes cannot be changed in current version of the SUI. We plan to enable this feature in the future. For now, only the legend of the plot can be changed by clicking on the blue Edit Line Appearance button under Graph Options.

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How can I save a graph as an image file?

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

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.

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 are 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 individual windows, click on the maximize button on any of the graph.

10.3 Saving and Exporting Raw Data

All data collected by the Squidstat potentiostats is saved automatically, and in real-time, to a Comma Separated Values (.CSV) file after starting any experiment. An example of how the data is displayed in the .CSV file format is below. The variable being saved is labeled in Row A. There are two types of data formats, one for DC measurements and one for AC measurements.

The first two columns (Step number and Step name) are same for both data formats. The difference between the two data formats is the parameters reported. For AC measurements, the parameters reported are: frequency, impedance modulus, phase, real impedance, imaginary impedance and total harmonic distortion (THD).

A1			$\times$	$\checkmark$	f_x	Step number		

	A	B	C	D	E	F	G	H
1	Step number	Step name	Frequency (Hz)	|Z| (Ohms)	Phase (deg)	Z' (Ohms)	Z" (Ohms)	THD(%)
2	2_1	EIS	9999.98	21.5755	-75.455	5.41851	-20.8841	0.193808
3	2_1	EIS	7943.25	26.0116	-76.3673	6.13086	-25.2788	0.365807
4	2_1	EIS	6309.57	33.0312	-76.4506	7.73869	-32.1119	0.253146
5	2_1	EIS	5011.87	40.6824	-76.5384	9.47061	-39.5647	0.280955
6	2_1	EIS	3981.05	50.1199	-76.3442	11.8327	-48.7031	0.344029
7	2_1	EIS	3162.26	61.5183	-75.6117	15.2868	-59.5887	0.215493

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For DC measurements, the parameters reported are: elapsed time, working electrode potential, current, absolute current, counter electrode potential and cumulative charge.

	A	B	C	D	E	F	G	H	I
1	Step number	Step name	Elapsed time (hr)	Elapsed time (s)	Working electrode (V)	Current (mA)	|Current| (mA)	Counter electrode (V)	Cumulative charge (mAh)
2	3_1	CV scan	0.00303948	10.9421	-0.497693	-0.0573611	0.0573611	-0.4914	0
3	3_1	CV scan	0.0030436	10.9569	-0.497998	-0.0593173	0.0593173	-5.60092	-2.40E-07
4	3_1	CV scan	0.00304762	10.9714	-0.497387	-0.0562608	0.0562608	8.24907	-4.73E-07
5	3_1	CV scan	0.00305164	10.9859	-0.497082	-0.0566887	0.0566887	-9.93776	-7.00E-07
6	3_1	CV scan	0.00305566	11.0004	-0.496166	-0.0579419	0.0579419	-12.5063	-9.30E-07

When running experiments that have both DC and AC methods, the .CSV file for AC methods are created separately. This includes DC data collected during the "quiet time" that users may include in EIS experiments.

Users do not need to manually save raw data after a channel has started, as long as they remember the filesave location they have selected. To make it easy to access this filesave location, it is always displayed on the bottom left of the View Data window. The folder and the name of the data folder is selected after activating an experiment and adding any optional experiment notes in the corresponding popup menu, unless **Experimental Note Visibility** has been turned off in the **More Options** menu.

Every raw data folder includes a text file called "Experiment Settings and Notes" in addition to the .CSV raw data file(s). The parameters, method steps, and notes made during the experiment are noted in this text file. The content of this file can be edited by clicking the blue **Edit Experiment Notes** box under the **Graph Options** in the **View Data** control tab. The notes can be added initially after pressing green **Start Experiment** box in the **Run an Experiment** control tab.
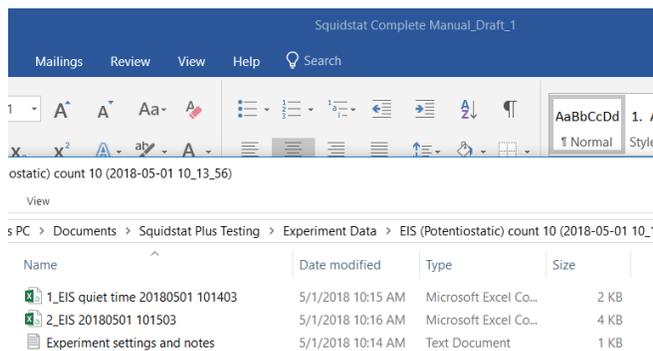
The number of .CSV files in the folder depends on the data saving settings chosen when building the experiment. Users have the option to separate the data into different .CSV files when changing to different methods or appending data from different methods onto a single .CSV.

The .CSV file format is a universally-recognized file format that is readily exportable into practically any third-party software. Specific instructions about how to import .CSV files into third-party software is provided by the third-parties themselves. Examples of analysis software compatible with Squidstat raw data includes Z-View from Scribner Associates and EC-Lab from Biologic Scientific Instruments.

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10.3.1 Channel Status

The purpose of this window is to quickly display the status of one or more Squidstats being controlled on a single computer. Users using many Squidstats in a multichannel configuration will find this window to be particularly useful.
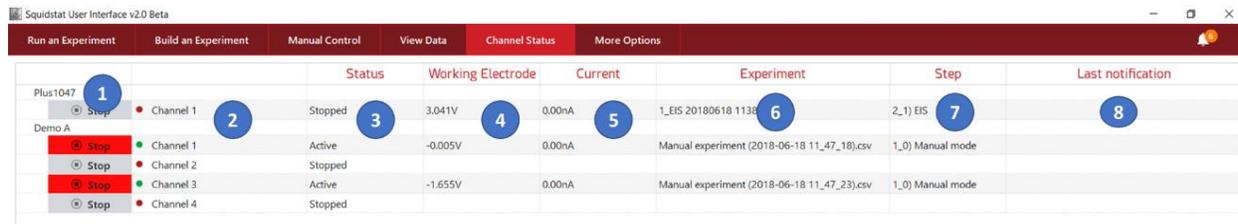
| Label	Description
1	Column displaying the name of the Squidstats connected to the computer. If a channel is active, the experiment running on the channel can be stopped by clicking the red Stop button.
2	Column displaying the channels associated with the Squidstats. The active channels are highlighted by green dots and the inactive channels are highlighted by red dots.
3	Column showing the operating status of each instrument and its corresponding channel(s)
4	Column showing the voltages of the working electrodes from the associated channels in real time. If an experiment is stopped, the last voltage recorded is what is displayed.
5	Column showing the current of the working electrodes from the associated channels in real time. If an experiment is stopped, the last current recorded is what is displayed.
6	Column showing the name of the experiment being run on the corresponding channel
7	Column showing the step of the experiment currently running on the channel
8	Column showing any notifications associated with the channel

10.3.2 More Options

This control tab is for more advanced control. Six options for more advanced control are provided on the left-hand panel of the window. Each option is described below:

10.3.2.1 Potentiostat Stability

Operational amplifiers (op-amps) are the circuit elements of a potentiostat that ensures the user-specified potential or current is applied at the working electrode clip. In a typical feedback operation, op-amps check voltage/current output, calculate the amount of modulation needed, and adjust the voltage/current. The op-amp takes some time to complete this operation, usually on the microsecond scale. If an experiment is comparatively slower than this operation, the op-amp has enough time to properly modulate and send out a stable voltage/current response.

Users can select the stability range for any Squidstat channel. Four ranges are available. Each range represents the bandwidth of signal frequency the op-amp can respond to without going into oscillation. The default mode is Range 1 (high stability, low speed). Range 1 is recommended for normal use, because it is the most

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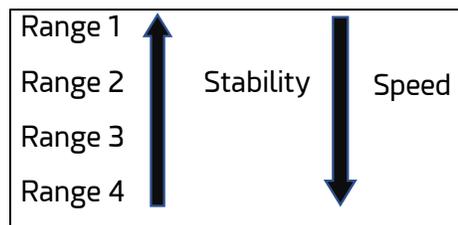
stable mode that can respond to a wide range of signal frequencies without showing any oscillations or ringing effects. When carrying out fast experiments, such as cyclic or linear voltammetry with high scan rates, or chrono experiments with very short pulses, switching to higher ranges might be needed. This is because the op-amp in Range 1 does not have enough time to send out a stable voltage/current response. Users can determine if they would benefit from switching to a higher range if they observe ringing or oscillations in the data.

10.3.2.2 Update firmware

Some software updates also require an accompanying firmware update to fully enable new features developed in later versions of the SUI. The Squidstat Plus is designed for firmware updates to be carried out using this option. Squidstat Prime and Squidstat Solo users must follow a separate set of instructions.

10.3.2.3 Experiment Notes

Before starting any experiment, users have 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 use this feature before starting every experiment can turn this option off here.

10.3.2.4 IR drop 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. After selecting this option, a user can choose the device and channel in which this correction is to be applied. The value of the uncompensated resistance and the compensation level (0 = no compensation to 100 = full compensation) can be entered in the next two boxes. A correction to the voltage is then made accordingly.

Uncompensated resistances are usually measured using one or more of the following three techniques: positive feedback mechanism, current-interrupt technique, and EIS. Of the three, EIS is available with the Squidstat Plus. 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.

10.3.2.5 Data sampling options

During experiments, data is recorded by the Squidstats at time intervals as short as 10 µs. However, not all of this data is saved by default, as doing so would quickly result in datafiles that are too large to manage. Therefore, data saved by the Squidstat are averaged values over selectable time periods. This options menu allows users to select how the recorded data are averaged and saved into the .csv file for the experiment.

Constant Potential/Current Open Circuit

Maximum data averaging period: | 0.001 | seconds

Potential/Current Sweep

α - Factor: | 1%

EIS

Minimum number of cycles sampled: | 2

Show data sampled during experiment "quiet times"

◯ Yes   ⦿ No

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Constant Potential/Current open Circuit

In the Maximum data averaging period for Constant Potential/Current and Open Circuit, the value entered in seconds specifies the period within which the data recorded every few microseconds are averaged. If a user specified the sample interval less than this value while setting up constant potential/current and open circuit methods, the time interval over which recorded data is averaged will be the specified sample interval.

Potential/Current Sweep

Due to the digital nature of modern potentiostats, it cannot apply an edge-free analog voltage or current ramp. The ramping is done in very small steps. For example, during a voltage ramp, potential is increased by a small step and held at that potential for a certain amount of
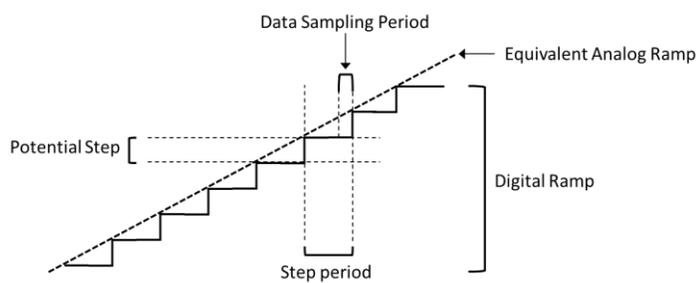
time or step period. A user can choose how to sample data during the step period by varying the **α-Factor**, which is the ratio of the data sampling period to the step period. If the α-Factor is 25%, the last 25% of the recorded data are averaged and tabulated in the .csv file. If the α-Factor is 100%, 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 a voltammetry experiment. Depending on the electrochemical reactions in play, one can obtain a response close to that from a true analog ramp by varying the α-Factor.

EIS

During EIS, users can choose the frequencies to be applied on an electrochemical device by entering the starting frequency, ending frequency, and steps per decade. With this option, users can enter the number of times each frequency is applied. The responses are averaged for that frequency. Users might choose to apply each frequency more than one time to get more noise-free response. However, this will increase the time taken to complete the EIS experiment and the data will have error if the electrochemical system is drifting during the experiment.

Show data sampled during experiment "quiet times"

If users choose not to show data sampled during experiment quiet times, the data recorded during the quiet times in methods like cyclic voltammetry and EIS will be not be shown in the **View Data** control tab. However, the quiet time data will still be available in .CSV format in the raw data folder.

10.3.2.6 Device information

This option shows users a list of all the different Squidstats connected to their computer. Clicking on any individual device will display its corresponding hardware information. This information is useful when communicating with Admiral Instruments technical support.

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11   Basic Method Tiles

The method tiles within the *Build an Experiment* tab are described in this section. Each method tile has a series of steps which need to be defined by the user.

11.1   Constant Current (Advanced)

This method tile runs chronopotentiometry, galvanostatic-coulometry, and constant-current charge and discharge experiments. In this method, a constant current is applied for a certain amount of time. The graphic in the tile shows a profile of *current (i) vs. time (t)* during a constant current method step.
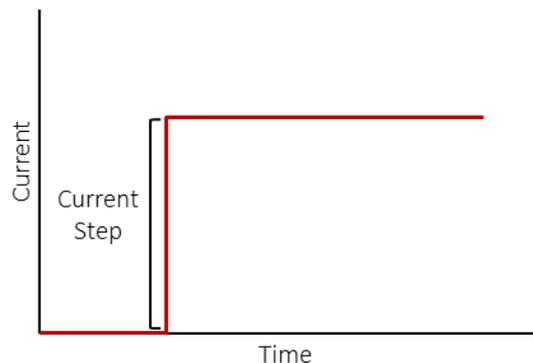
Current: This is the constant current that is applied. It can be a positive or a negative value.

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

Ending Conditions: This method will end whenever the first of any of the following conditions are met. Users can choose to fill in each condition or leave one or more conditions blank. But, at least one must be filled in for the experiment to work properly.

  Upper voltage limit: The method will end if the voltage response becomes higher than this value.

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

Maximum duration: The Squidstat will apply current for this amount of time, if no other ending conditions are met before this amount of time has elapsed.

Maximum capacity: The step will end if the capacity (Current × time) is higher than this value. To make this option ineffective, enter zero.

Current range: More than one current range is available to measure a wide scale of current magnitudes accurately**.** Although users can let the SUI automatically choose the current ranges used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when these two options should be used.

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Use the lowest range possible: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

Use a higher current range, corresponding to: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when the Squidstat switches between current ranges.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.2 Constant Potential (Advanced)

This method tile runs chronoamperometry, potentiostatic-coulometry and constant-voltage charge and discharge experiments. In this method, a constant voltage is applied for a certain amount of time. The graphic in the tile shows a profile of voltage (E) vs. time (t) during a constant voltage method step.
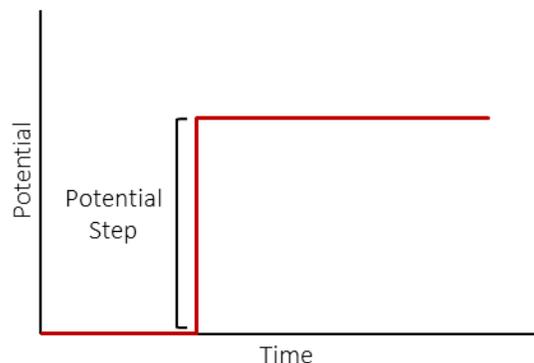
Potential: This is the constant potential that is applied. It can be a positive or a negative value.

With respect to: The reference potentials against which the working electrode potentials are set. Users can select either "open circuit" or "reference". Open circuit is the open circuit potential, which is fixed throughout the experiment. The reference is the potential of the electrode where the reference electrode clip is attached. This reference potential can change during the experiment depending on the electrode.

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

Ending Conditions: This method will end whenever the first of any of the following conditions are met. Users can choose to fill in each condition or leave one or more conditions blank. But, at least one must be filled in for the experiment to work properly.

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Maximum duration: The Squidstat will apply potential for this amount of time if no other ending conditions are met before this amount of time has elapsed.

Maximum current: The method will end if the current response becomes higher than this value.

Minimum current: The method will end if the current response becomes lower than this value.

Minimum dI/dt: The method will end if the rate of change of the current response with respect to time is less than this value.

Maximum capacity: The method will end if the capacity (current × time) is higher than this value. To remove this ending condition, enter zero.

Current range: More than one current range is available to measure a wide scale of current magnitudes accurately**.** Although users can let the SUI automatically choose the current ranges used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when these two options should be used.

Use the lowest range possible: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

Use a higher current range, corresponding to: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when the Squidstat switches between current ranges.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.3 Cyclic Voltammetry

This method tile runs a cyclic voltammetry experiment. In this method, potential is varied back and forth between two points or vertices at a fixed rate with respect to time. The graphic in the tile shows a profile of voltage (E) vs. time (t) during a cyclic voltammetry step. **Scanning direction**: Select "scan to the upper vertex first" for a positive scan or select "scan to the lower vertex first" for a negative scan.

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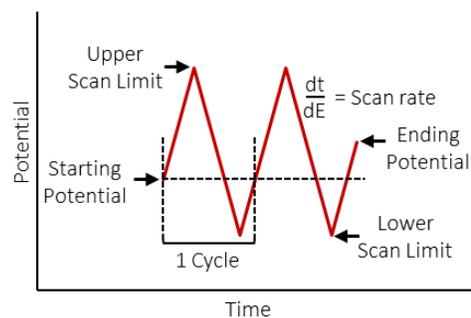
Starting potential: This potential will be applied for a specified amount of time before scanning starts. This is also the starting point for the scan.

With respect to: The reference potentials against which the working electrode potentials are set. Users can select either "open circuit" or "reference". Open circuit is the open circuit potential, which is fixed throughout the experiment. The reference is the potential of the electrode where the reference electrode clip is attached. This reference potential can change during the experiment depending on the electrode.

Quiet time: The starting potential will be constantly applied for this duration before scanning starts.

Upper scan limit: The more positive of the two potential limits/vertices. The scanning direction will change from a positive to negative scan at this potential.

Lower scan limit: The more negative of the two potential limits/vertices. The scanning direction will change from a negative to positive direction at this potential.

Scanning direction:	Scan to upper vertex first	
Starting potential:	0	V
with respect to	open circuit	
Quiet time:	5	seconds

(time spent at the starting potential)

Upper scan limit:	0.1	V
with respect to	open circuit	
Lower scan limit:	-0.1	V
with respect to	open circuit	
Scan rate (dE/dt):	10	mV/s
Sample at intervals of:	0.1	mV
Repeats:	3	

Current ranging
- ◉ Autorange
- ○ Approx. max current: 100 mA

Scan rate: The rate of change of voltage with time. The maximum scan rate of the Squidstat Plus is 180 V/s. The maximum scan rate of the Squidstat Prime and Solo is 30 V/s.

Sample at intervals of: The interval between two consecutive data points. It can be chosen based on an interval of time (seconds) or voltage (mV). Care should be taken at high scan rates because at very high scan rates, the Squidstat will not be able to record data at very small intervals. This is also true for other potentiostats.

Repeats: The number of cycles to be performed. Each cycle consists of a scan from the lower vertex to upper vertex and back to the lower vertex, or vice versa.

Current range: More than one current range is available to measure a wide scale of current magnitudes accurately**.** Although users can let the SUI automatically choose the current ranges used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when these two options should be used.

Use the lowest range possible: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

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Use a higher current range, corresponding to: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when the Squidstat switches between current ranges.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.4   DC Potential Linear Sweep

This method tile runs linear 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 in the tile shows a profile of potential (E) vs. time (t) during a DC potential linear sweep method step.
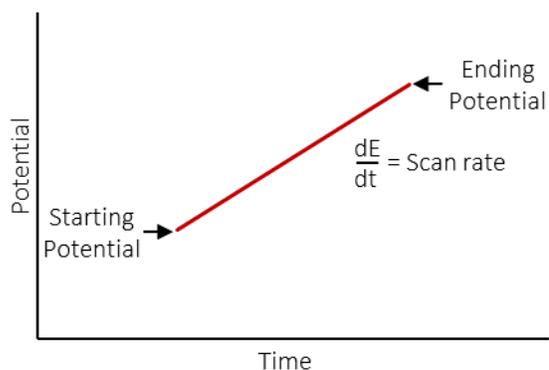
Starting potential: The scan starts from this potential.

With respect to: The reference potentials against which the working electrode potentials are set. Users can select either "open circuit" or "reference". Open circuit is the open circuit potential, which is fixed throughout the experiment. The reference is the potential of the electrode where the reference electrode clip is attached. This reference potential can change during the experiment depending on the electrode.

Ending potential: The scan ends at this potential.

Scan rate: The rate of change of potential with respect to time. The maximum scan rate of the Squidstat Plus is 180 V/s. The maximum scan rate of the Squidstat Prime and Solo is 30 V/s.

Sample at intervals of: The interval between two consecutive data points. It can be chosen based on interval of time (seconds) or voltage (mV). Care should be taken at high scan rates because at very high scan rates, the Squidstat will not be able to record data at very small intervals. This is also true for other potentiostats.

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Current range: More than one current range is available to measure a wide scale of current magnitudes accurately**.** Although users can let the SUI automatically choose the current ranges used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when these two options should be used.

Use the lowest range possible: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

Use a higher current range, corresponding to: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when the Squidstat switches between current ranges.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.5   DC Current Linear Sweep

This method tile runs a galvanostatic linear sweep. In this step, current is scanned from a starting current to the ending current at a fixed scan rate. The graphic in the tile shows a profile of current (i) vs. time (t) during a DC Current Linear Sweep method step.
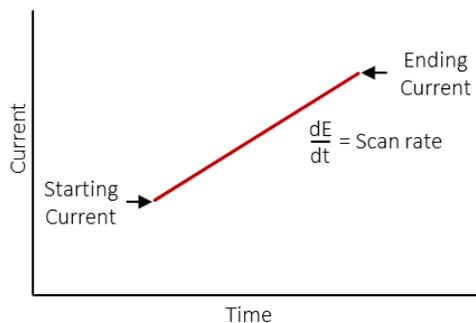
Starting current: The scan starts at this current value.

Ending current: The scan ends at this current value.

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

Sample at intervals of: The interval between two consecutive data points. It can be chosen based on interval of time (seconds) or current (mA / µA / nA).

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous method tile, select "No". In cases where this method tile

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is set to loop to run multiple times before moving on to the next tile, selecting "Yes" will create a new .CSV for each individual loop.

11.6 Open Circuit

This step records open circuit potential (OCP) for a user-specified amount of time or other user-specified ending conditions. During this time, current does not flow through the current carrying cables, but voltage data is graphed and recorded. The graphic in the tile shows a profile of potential vs current, where current is zero as in open circuit.
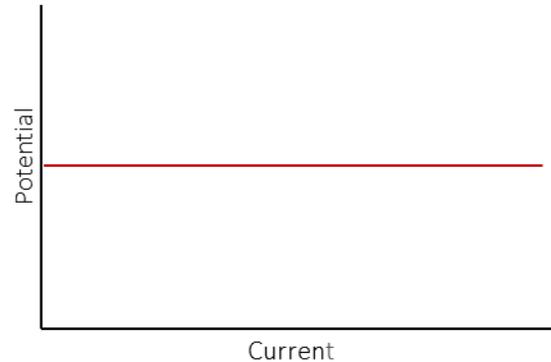
Sampling interval: The time difference between two consecutive data points.

Ending Conditions: This method will end whenever the first of any of the following conditions are met. Users can choose to fill in each condition or leave one or more conditions blank. But, at least one must be filled in for the experiment to work properly.

Maximum duration: The Squidstat will measure OCP for this amount of time if no other ending conditions are met first.

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

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

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

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

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11.7 Constant Power Charge/Discharge

This method tile charges or discharges an electrochemical device (eg. Battery, Fuel Cells, Capacitors) under constant power until one of the ending conditions is met. The graphic shows a profile of power (P) during a constant power charge or discharge step.
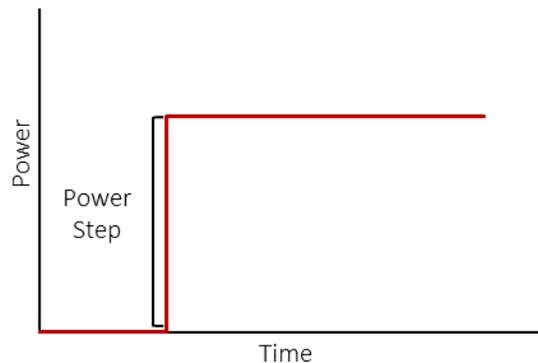
Load power: The Squidstat charges or discharges an electrochemical device at this power.

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

Ending Conditions: This method will end whenever the first of any of the following conditions are met. Users can choose to fill in each condition or leave one or more conditions blank. But, at least one must be filled in for the experiment to work properly.

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

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

> *Note*: *The potential at the working electrode clip is equal to cell voltage only if the reference electrode clip is attached to the counter electrode.*

Maximum duration: The experiment will run for this amount of time if no other ending conditions are met first.

Maximum capacity: The step will end if the capacity (current × time) is higher than this value. To remove this ending condition, enter zero.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

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11.8 Constant Resistance Discharge

This method tile discharges an electrochemical device (eg. Battery, Fuel Cells, Capacitors) under constant load (resistance) until one of the ending conditions are met. This method cannot charge an electrochemical device. The graphic shows a profile of The profile of load (resistance) during a Constant Resistance Discharge step.

Load resistance: The Squidstat discharges an electrochemical device at this resistance. The set value for resistance should be positive.

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

Ending Conditions: This method will end whenever the first of any of the following conditions are met. Users can choose to fill in each condition or leave one or more conditions blank. But, at least one must be filled in for the experiment to work properly.

> **Minimum cell voltage**: The step will end if the cell voltage becomes lower than this value.

Note: The potential at the working electrode clip is equal to cell voltage only if the reference electrode clip is attached to the counter electrode.

> **Maximum duration**: The experiment will run for this amount of time if no other ending conditions are met first.

> **Maximum capacity**: The step will end if the capacity (current × time) becomes higher than this value. To remove this ending condition, enter zero.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

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11.9 Maximum Power Charge/Discharge

This method tile charges/discharges an electrochemical device at maximum power (eg. Batteries, Fuel Cells, Super capacitors) until one of the ending conditions are met. The user does not have to enter a value for the power. The algorithm used to reach maximum power is based on the Maximum Power Point Tracking (MPPT) Hill Climbing Algorithm. The graphic in the tile shows a profile of a typical response of the MPPT algorithm to a current perturbation.
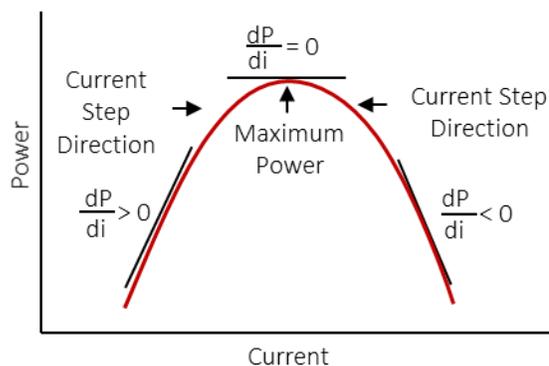
For example, during maximum power discharge, a positive current step is applied. If the change in power is positive, the magnitude of the current increases. If the change in power is negative, the magnitude of the current decreases. If there is no change in power, maximum power is achieved.

Minimum cell voltage: The step will end if voltage is lower than this voltage.

> *Note: The potential at the working electrode clip is equal to cell voltage only if the reference electrode clip is attached to the counter electrode.*

Maximum duration: The experiment will run for this amount of time if no other ending conditions are met first.

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

Maximum capacity: The step will end if the capacity (Current × time) is higher than this value. To remove this ending condition, enter zero.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

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11.10 Normal Pulse Potential Voltammetry

This method tile will run a normal pulse voltammetry experiment. In this experiment, pulses of potential are superimposed over a constant set potential. Therefore, the baseline of the pulses does not change. The height of the pulses changes steadily at a fixed step. The pulse width and the pulse period are the same for each pulse. Each datapoint is taken once at the end of the pulse duration. The graphic shows a profile of potential (E) vs. time (t) of a Normal Pulse Potential Voltammetry method step.
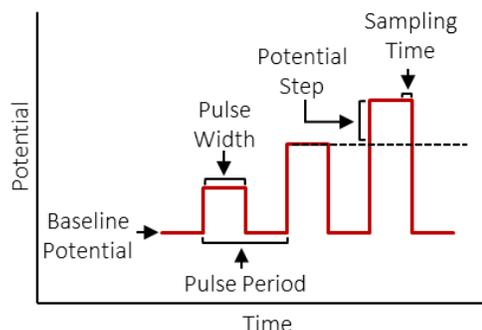
Baseline potential: The Squidstat will apply this value as a base potential.

With respect to: The reference potentials against which the working electrode potentials are set. Users can select either "open circuit" or "reference". Open circuit is the open circuit potential, which is fixed throughout the experiment. The reference is the potential of the electrode where the reference electrode clip is attached. This reference potential can change during the experiment depending on the electrode.

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

Final potential. The experiment will end if the magnitude of the potential pulse is equal or more than this potential.

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

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

Current range: More than one current range is available to measure a wide scale of current magnitudes accurately**.** Although users can let the SUI automatically choose the current ranges used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when these two options should be used.

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Use the lowest range possible: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

Use a higher current range, corresponding to: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when the Squidstat switches between current ranges.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.11 Normal Pulse Current Voltammetry

This method tile will run normal pulse voltammetry (galvanostatic) experiment. In this experiment, pulses of currents are superimposed over a constant set current. Therefore, the baseline of the pulses does not change. The height of the pulses changes steadily at a fixed step. The pulse width and the pulse period are the same for each pulse. Each datapoint is taken at the end of the pulse width. The graphic in the tile shows a profile of current (i) vs. time (t) of a Normal Pulse Current Voltammetry method step.
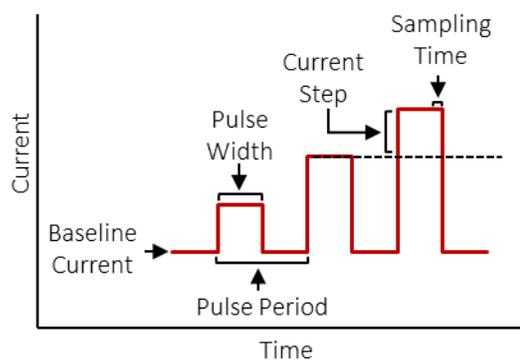
Baseline current: The Squidstat will apply this value as a base current.

Current step: This is the magnitude difference between consecutive current pulses.

Final current. The step will end if the magnitude of the current pulse is equal or more than this potential.

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

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

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Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.12 Differential Pulse Potential Voltammetry

This method tile will run a differential pulse voltammetry (potentiostatic) experiment. In this experiment, pulses of potentials are superimposed on a potential staircase. The baseline value of each pulse of potential, before and after the pulse width, differs by a constant value. However, the height of each potential pulse is the same.
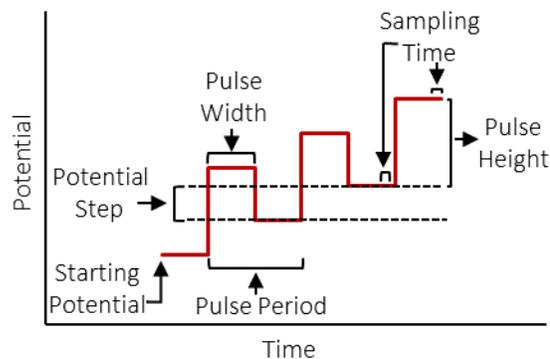
The time between the consecutive pulses is the difference between the pulse period and the pulse width. The current response is sampled at two points, one at the end of the pulse width [i(τ)] and another immediately before the pulse [i(τ')]. The difference in current response sampled at these two points is plotted as a difference [$\delta i = i(\tau) - i(\tau')$] against the baseline potential. The graphic shows a profile of potential applied vs. time of a Differential Pulse Potential Voltammetry (DPV) method step.

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

With respect to: The reference potentials against which the working electrode potentials are set. Users can select either "open circuit" or "reference". Open circuit is the open circuit potential, which is fixed throughout the experiment. The reference is the potential of the electrode where the reference electrode clip is attached. This reference potential can change during the experiment depending on the electrode.

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

Final potential. The step will end if the value of the baseline is equal or more than this value.

Pulse height: The magnitude of each pulse.

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

Pulse period: This amount of time will be applied between the start of consecutive pulses and the pulse width.

Current range: More than one current range is available to measure a wide scale of current magnitudes accurately. Although users can let the SUI automatically choose the current ranges used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when these two options should be used.

Use the lowest range possible: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

Use a higher current range, corresponding to: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when the Squidstat switches between current ranges.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.13 Differential Pulse Current Voltammetry

This method tile will run a differential pulse voltammetry (galvanostatic) experiment. In this experiment, pulses of current are superimposed on a current staircase. The baseline value of each pulse of current, before and after the pulse width, differs by a constant value. However, the height of each pulse of current is the same.

The time between the consecutive pulses is the difference between the pulse period and the pulse width. The potential response is sampled at two points, one at the end of the pulse width [E(τ)] and another immediately before the pulse [E(τ')]. The difference in potential response sampled at these two points is plotted as a difference [$\delta E = E(\tau) - E(\tau')$] against the baseline current. The graphic shows a profile of current applied vs. time of a Differential Pulse Current Voltammetry method step.

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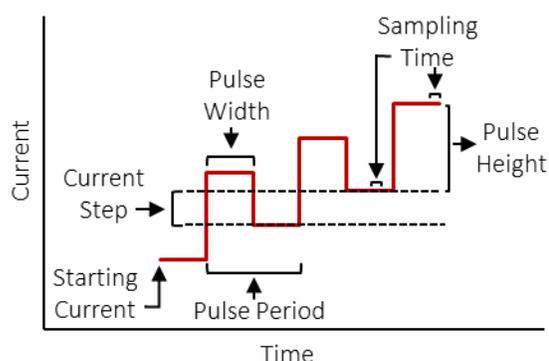
Starting current: The Squidstat will apply this value at the beginning of the scan.

Current step: This is the difference between consecutive return currents after the pulses.

Final current. The step will end if the value of the baseline is equal or more than this value.

Pulse height: The magnitude of each pulse.

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

Pulse period: This amount of time will be applied between start of consecutive pulses and the pulse width.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

11.14 Square Wave Potential Voltammetry

This method will run a square wave voltammetry (potentiostatic) experiment. In this experiment, square pulses of potential are superimposed on a potentiostatic staircase. Square wave consists of a forward pulse and a reverse pulse of equal magnitudes and duration. The inverse of the total duration of a pulse is the frequency. Current responses are sampled at two points, one at the end of the forward pulse (i_f) and another at the end of the reverse pulse (i_r).
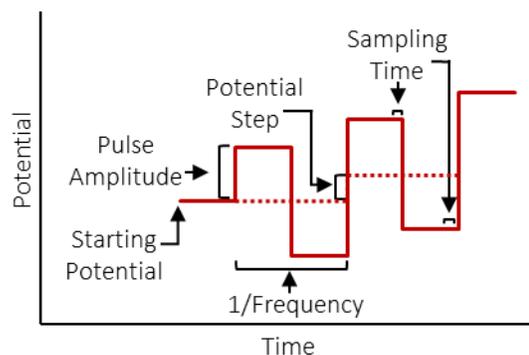
The difference in current sampled at these two points ($\Delta i = i_f - i_r$) is plotted against the potential of the corresponding staircase. The graphic shows a profile of potential applied vs. time of a Square Wave Potential Voltammetry method step.

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

With respect to: The reference potentials against which the working electrode potentials are set. Users can select either "open circuit" or "reference". Open circuit is the open circuit potential, which is fixed throughout the experiment. The reference is the potential of the electrode where

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the reference electrode clip is attached. This reference potential can change during the experiment depending on the electrode.

Potential step: This is the difference between base potentials of consecutive square pulses. Enter a positive value for a positive scan and a negative value for the negative scan.

Final potential. The step will end if the baseline potential of a square pulse is equal or more than this value.

Pulse amplitude: The magnitude of each pulse.

Frequency. The number of pulses per second.

Starting potential:	-1.4	V
with respect to:	reference	
Quiet time:	5	seconds
Voltage step:	0.002	V
Final potential:	0.2	V
with respect to:	reference	
Pulse amplitude:	0.025	V
Square wave frequency:	5	Hz

Current ranging

| Approx. max current: | 1 | mA |

Current range: More than one current range is available to measure a wide scale of current magnitudes accurately**.** Although users can let the SUI automatically choose the current ranges used during the experiment (Autorange), this can result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when these two options should be used.

Use the lowest range possible: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

Use a higher current range, corresponding to: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when the Squidstat switches between current ranges.

Start a new .CSV file: By selecting "Yes", a new .CSV file for this method will be created each time it runs. Use this option to create a separate data file of this method from the rest of the other methods in the sequence of the overall experiment. If a user wants to append the data from this method to the .CSV file from the previous 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 "Yes" will create a new .CSV for each individual loop.

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12 Electrochemical Impedance Spectroscopy (EIS)

12.1 EIS Potentiostatic

This method tile runs a potentiostatic electrochemical impedance spectroscopy experiment. Sine waves of potential varying in frequencies, but all with the same amplitude, are applied to an electrochemical device in an increasing or decreasing order of frequencies. The current response is then measured. The graphic shows a profile of voltage (E) vs. time (t) during a potentiostatic electrochemical impedance spectroscopy (EIS) step.
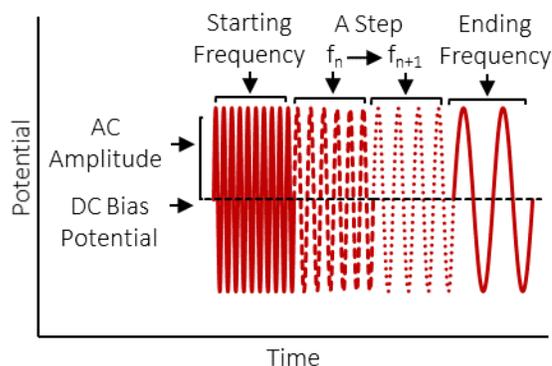
Quiet time: The DC bias potential will be applied for this amount of time before starting frequency scan.

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

DC bias potential: This potential will be applied as the baseline potential.

With respect to: The reference potentials against which the working electrode potentials are set. Users can select either "open circuit" or "reference". Open circuit is the open circuit potential, which is fixed throughout the experiment. The reference is the potential of the electrode where the reference electrode clip is attached. This reference potential can change during the experiment depending on the electrode.

AC amplitude: This is the amplitude of the potentiostatic sine waves. The maximum voltage amplitude that can be set is 1 V.

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12.2   EIS Galvanostatic

This method tile runs a galvanostatic electrochemical impedance spectroscopy experiment. Sine waves of current varying in frequencies, but all with the same amplitude, are applied to an electrochemical device in an increasing or decreasing order of frequencies. The voltage response is then measured. The graphic shows a profile of current (i) vs. time (t) during a galvanostatic electrochemical impedance spectroscopy (EIS) step.
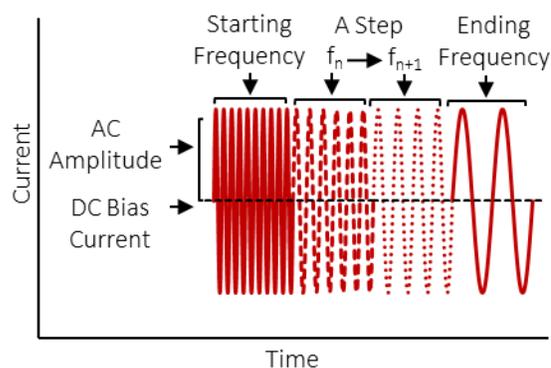
Quiet time: The DC bias current will be applied for this amount of time before starting sine waves.

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 frequency of the sine waves will change logarithmically.

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

AC amplitude: This is the amplitude of the galvanostatic sine waves. The maximum current amplitude that can be set is 100 mA.

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13   Pre-built Experiments

The pre-built experiments that appear in the Categories panel under the Run an Experiment tab are described in this chapter, unless already previous described.

13.1   Charge/Discharge 1 (Galvanostatic Cycling with Potential Limitation)

This experiment repeatedly charges or discharges an electrochemical device under test. For each charge and discharge phase, the electrochemical device undergoes a constant current (CC) segment and then a constant voltage (CV) segment. The accompanying graphic for the experiment shows a profile of current applied vs. time. Please note that during the rest period, the potentiostat is in open circuit potential mode.

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

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

Upper voltage limit: The Squidstat will apply the specified charging current until this voltage is reached.

Lower voltage limit: The Squidstat will apply the specified discharging current until this voltage is reached.

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

Charge Current
   Max (CC phase): This is the current applied during charging.

   Min (CV phase): The Squidstat will stop charging if the current is lower than this value when maintaining the upper voltage limit for the rest phase duration.

Discharge Current
   Max (CC phase): This is the current applied during discharge.

   Min (CV phase): The Squidstat will stop discharging if the current is lower than this value when maintaining the lower voltage limit for the rest phase duration.

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Sampling Interval

During CC phases: The time difference between two consecutive data points during constant current phases.

During CV phases: The time difference between two consecutive data points during constant voltage phases.

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

13.2 Charge/Discharge 2 (Const R discharge, const P charge)

This experiment discharges an electrochemical device with a constant resistance load and charges the device with a constant power source. The accompanying graphic for the experiment shows a profile of power and resistance applied during charging and discharging cycles, respectively vs. time. Please note that during the rest period, the potentiostat is in open circuit potential mode.

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

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

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

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

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

Maximum capacity: The charging phase will end if the capacity (Current × time) is higher than this value. To make this option ineffective, enter zero.

Constant resistance discharge
Load resistance: The Squidstat will discharge the electrochemical device at this load.

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

Maximum capacity: The discharging phase will end if the capacity (Current × time) is higher than this value. To make this option ineffective, enter zero.

Rest period
Duration: The Squidstat will measure open circuit potential of the electrochemical device for this duration after a charge/discharge cycle.

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

13.3 Charge/Discharge 3 (Max P discharge, const P charge)

This experiment discharges the electrochemical device under test using maximum power point tracking and charges it with a constant power source. The accompanying graphic for the experiment shows a profile of power applied during charging and discharging cycles vs. time. Please note that during discharge, the cell is discharged at maximum power determined using Maximum Power Point Tracking (MPPT) Hill Climbing Algorithm. Please note that during the rest period, the potentiostat is in open circuit potential mode.

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

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

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

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

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

Maximum capacity: The charging phase will end if the capacity (Current × time) is higher than this value. To make this option ineffective, enter zero.

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Maximum power discharge

    Lower voltage limit: The Squidstat will discharge the electrochemical device at maximum power until this voltage is reached.

    Maximum capacity: The discharging phase will end if the capacity (Current × time) is higher than this value. To make this option ineffective, enter zero.

Rest period

    Duration: The Squidstat will measure open circuit potential of the electrochemical device for this duration before starting a charge/discharge cycle.

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

13.4 Charge/Discharge 4 (Galvanostatic Cycling with Potential Limitation) with EIS

This experiment repeatedly charges and discharges an electrochemical device under test. For each charge and discharge phase, the electrochemical device undergoes a constant current (CC) segment and then a constant voltage (CV) segment. In between each charge/discharge cycle, the potentiostat performs a potentiostatic electrochemical impedance spectroscopy (EIS) scan.
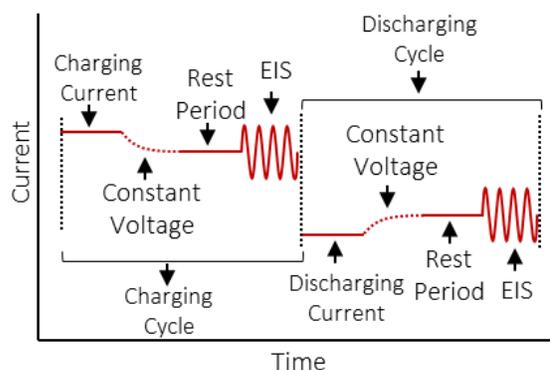
This experiment can only be run with the Squidstat Plus. It cannot be run with the Squidstat models that do not include EIS capabilities. The accompanying graphic for the experiment shows a profile of current applied during charging and discharging cycles vs. time. Please note that during the rest period, the potentiostat is in open circuit potential mode.

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

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

Upper voltage limit: The Squidstat will apply the specified charging current until this voltage is reached.

Lower voltage limit: The Squidstat will apply the specified discharging current until this voltage is reached.

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Rest phase duration: This is the amount of time the Squidstat will spend between constant voltage and EIS segments of the experiment.

Charge Current
   Max (CC phase): This is the current applied during charging.

   Min (CV phase): The Squidstat will perform EIS if the current is lower than this value when maintaining the upper voltage limit for the rest phase duration.

Discharge Current
   Max (CC phase): This is the current applied during discharge.

   Min (CV phase): The Squidstat will perform EIS if the current is lower than this value when maintaining the lower voltage limit for the rest phase duration.

Sampling Interval
   During CC phases: The time difference between two consecutive data points during constant current phases.

   During CV phases: The time difference between two consecutive data points during constant voltage phases.

   During rest phases. The time interval between two consecutive data points during rest phases.

EIS parameters
   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.

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

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

13.5 Galvanostatic Intermittent Titration Technique (GITT)

In this experiment, a charging current of certain magnitude is applied for a certain amount of time (current pulse duration). Then, the current is interrupted or the potentiostat goes to OCP mode for a certain amount of time (relaxation time). This cycle is continued until the potential hits the upper voltage limit. Afterwards, discharging current of same magnitude is applied similarly until the potential reaches the lower voltage limit. The

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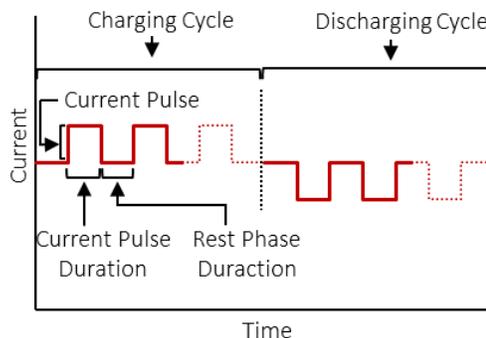
accompanying graphic for the experiment shows a profile of current pulses applied during charging and discharging cycles vs. time.

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

Starting phase:	Charge first ∨	
Upper voltage limit:	3.6	V
Lower voltage limit:	2.8	V
Current pulse:	100	mA ∨
Current pulse duration:	15	minutes
Current pulse sampling interval:	10	s
Rest phase duration:	15	minutes
Rest phase sampling interval:	10	s

Upper voltage limit: The Squidstat will apply the specified charging current until this voltage is reached.

Lower voltage limit: The Squidstat will apply the specified discharging current until this voltage is reached.

Current pulse: 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 before interrupting current.

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

Rest phase duration: The amount of time after current interruption before the next current pulse is applied. 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.

13.6 Potentiostatic intermittent titration technique (PITT)

In this experiment, during charging step, potential is applied in steps until the upper potential limit is reached. Then the direction of the potential steps is reversed until the lower potential limit is reached during discharging step. Each potential step is applied for a certain duration of time 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 accompanying

graphic for the experiment shows a profile of potential applied during charging and discharging cycles vs. time.

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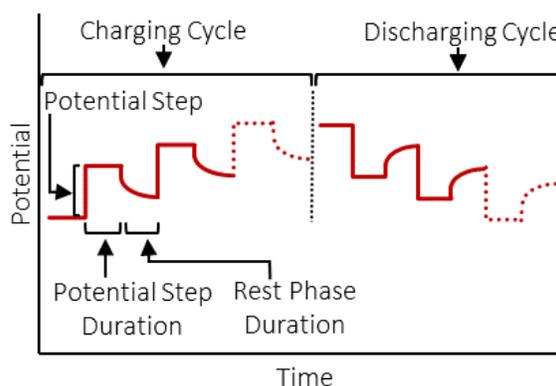
Starting phase: Select whether the cycle is to begin by charging or discharging.

Upper voltage limit: The Squidstat will apply the specified charging current until this voltage is reached.

Lower voltage limit: The Squidstat will apply the specified discharging current until this voltage is reached.

Starting phase:	Charge first ▾	
Upper voltage limit:	3.6	V
Lower voltage limit:	2.8	V
Potential step size:	0.02	V
Applied voltagephase duration:	15	minutes
Applied voltage phase sampling interval:	10	s
Rest phase duration:	15	minutes
Rest phase sampling interval:	10	s

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 potential step.

Rest phase duration: The amount of time after current interruption before the next current pulse is applied. 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.

13.7 Rotating Ring Disk Electrode (RRDE)

This experiment is built to run a rotating ring disk electrode (RRDE) experiment. In a typical RRDE experiment, the ring electrode is set at a fixed voltage bias with respect to the reference electrode or to the disk. The disk is usually subjected to a steady state potential scan. Currents measured at the disk and the ring are plotted against the potential of the disk electrode. Please refer to the application note, "Can Squidstat Potentiostats Be Configured as Bipotentiostats for RRDE?" for instructions on cell cable connections before running the experiment.

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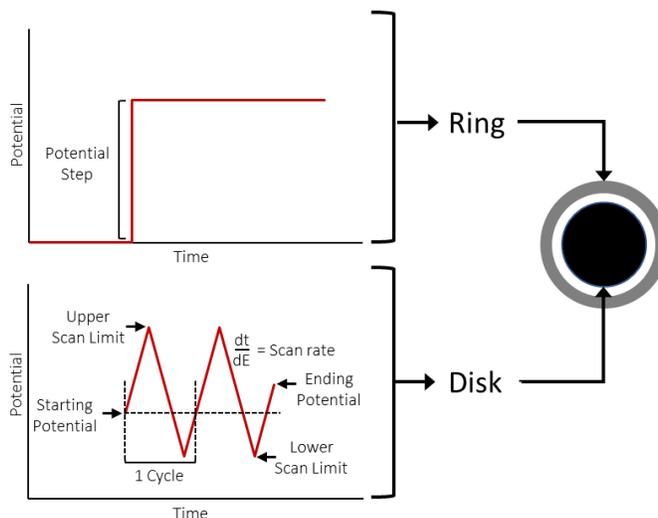
Disk instrument: Select which Squidstat is being used to control the disk. The selected Squidstat can be either a single-channel or multichannel Squidstat. If a multichannel Squidstat (like the 4-channel Squidstat Prime) is selected, the specific channel controlling the disk will need to be assigned.

Disk channel: Used to select which channel on the selected Squidstat will control the disk. In cases where a single-channel Squidstat is assigned to control, this will default to Channel 1.

Quiet time: The starting potential will be applied for the length of time input here before the scanning portion of the RRDE experiment begins.

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. Reference potential is the potential of the electrode that the reference electrode clip (RC) is attached to.

Ending potential: The potential that is applied at the end of the scan. This can be set with respect to the open circuit potential or the reference potential. Reference potential is the potential of the electrode that the reference electrode clip (RC) is attached to.

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

Sample at intervals of: The interval between two consecutive current data points. It can be set based on an interval of time (s) or potential (mV).

Disk current ranging: More than one current ranges exist in Squidstats to measure a wide scale of current magnitudes accurately. Although user can let SUI choose the current range corresponding to a magnitude of current (Autorange), in some cases this will result in noise and loss of data. Therefore, options are provided for autorange and fixed range modes. Please refer to the application note titled *Why Do Potentiostats Click* for more guidance about when either of these two options should be used.

Autorange: This is the default setting. The best current range for the highest achievable accuracy is automatically selected, and in cases where multiple current ranges must be used, the Squidstat will automatically switch to different current ranges.

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Approx. max current: This is an advanced setting. Here, the user selects the desired current range by entering the value corresponding to the desired range. This is useful to avoid noise created when changing current ranges.

Ring instrument: Used to select which Squidstat is being used to control the ring. The selected Squidstat can be either a single-channel or multichannel Squidstat. If a multichannel Squidstat (like the 4-channel Squidstat Prime) is selected, the specific channel controlling the ring will need to be assigned.

Ring channel: Used to select which channel on the selected Squidstat will control the ring. In cases where a single-channel Squidstat is assigned to control, this will default to Channel 1.

Potential: This is the constant potential that is applied at the ring electrode. Set this potential with respect to the reference. For bipotentiostat mode, make sure the reference electrode clip (RC) of the channel connected to the ring electrode is also attached to the same reference electrode that the RC of the disk electrode is connected to. For scanning bipotentiostat mode, attach the RC of the channel connected to the ring electrode to the disk electrode. The potential of the ring should be set against the reference for this mode to work.

Ring current ranging: Same concept as disk current ranging, explained above.

14   Preset Graphs

Several pre-built settings to represent data are available in the SUI whenever viewing data (but excluding Manual Mode). These pre-built settings are often referred to by their names such as Bode Plot, Nyquist Plot, etc. In this chapter, brief descriptions of the plots are provided.

14.1   Voltammogram (I vs. E)

Voltammograms are often used to represent data taken during voltammetry experiments such as cyclic voltammetry and linear sweep voltammetry. In these plots, current (i) is plotted on the Y-axis and potential (E) is plotted on the X-axis.

Voltammograms are often used as a qualitative way to extract information about the electrochemical processes involved in the system. Examples include 1) the number of redox reactions, 2) the reversibility of the reactions, 3) the effect of capacitance, 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 find redox reactions and the effect of the solid-electrolyte interphase (SEI). By using the data to model these electrochemical reactions, reaction mechanisms can also be inferred.

14.2   Polarization Plots (E vs. i)

Polarization plots are used to represent data taken during steady state measurements of electrochemical power systems, such as fuel cells and batteries. A steady state measurement is usually a potential or current scan with a very slow rate. At each potential or current increment

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made by the potentiostat, the system under test settles down and reaches a steady state. The time taken to reach a steady state depends on the system.

In these plots, potential (E) is plotted on the Y-axis and current (i) is plotted on the X-axis. This plot represents the performance of the electrochemical system. From these plots, users are generally interested in finding the voltage at a given current. These plots generally have three regions: activation overpotential, iR (or ohmic) overpotential, and mass transfer overpotential. By studying these regions, a user can analyze sources of performance losses or devise ways to increase the performance.

14.3 Cottrell Plot (i vs $t^{-1/2}$)

Cottrell Plots are a popular way to represent data from chronoamperometry experiments. In this plot, the time-dependent current (i) response is plotted on the Y-axis and the negative square root of time ($t^{-1/2}$) is plotted on the X-axis. If the plot shows a linear regression between the current and the negative square root of time, it is an indication of a diffusion-limited process. From the slope of this linear regression, the diffusion coefficient (D) can be calculated using the Cottrell equation:

$$i(t) = \frac{nFAD^{1/2}C}{\pi^{1/2}t^{1/2}}$$

Where n is the stoichiometric number of electrons, F is the Faraday constant, A is the surface area of the electrode, and C is the bulk concentration.

14.4 Sands Plot (i vs $\tau^{-1/2}$)

Sands Plots can be used to represent data from chronopotentiometry experiments. In this plot, current (i) is plotted on the Y-axis and the negative square root of a transition time ($\tau^{-1/2}$) is plotted on the X-axis. The transition time (τ) is the time from the application of a constant current until the potential response shows a steep shift to a higher magnitude. Therefore, $\tau^{-1/2}$ is obtained at varying magnitudes of current. If the plot shows a linear regression, it is an indication of a diffusion-limited process. From the slope of this linear regression, the diffusion coefficient (D) can be calculated using the Sands equation:

$$i = \frac{nFAD^{1/2}\pi^{1/2}C}{2\tau^{1/2}}$$

Where n is the stoichiometric number of electrons, F is the Faraday constant, A is the surface area of the electrode, and C is the bulk concentration.

This plot is less popular than the Cottrell plot because of the difficulty in accounting for the double-layer capacitive effect that makes measuring τ challenging.

14.5 Anson Plot (Q vs $t^{1/2}$)

Anson Plots are a popular way to represent data from a chronocoulometry experiments. In this plot, time-dependent charge (Q) is plotted on the Y-axis and the positive square root of time ($t^{1/2}$) is

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plotted on the X-axis. The linear relation between Q and $t^{1/2}$ indicates a diffusion-limited process. The mathematical form for this plot is obtained by integrating the Cottrell equation:

$$Q(t) = \frac{nFAD^{1/2}Ct^{1/2}}{\pi^{1/2}}$$

Where n is the stoichiometric number of electrons, F is the Faraday constant, A is the surface area of the electrode, D is the diffusion coefficient, and C is the bulk concentration.

14.6   Tafel Plot (log i vs. E)

In this plot, log of current (log i) is plotted on the Y-axis and overpotential (η) or potential (E) is plotted on the X-axis. Overpotential is defined as:

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

Where, E_{eq} is the equilibrium potential at the working electrode given by the Nernst Equation and E is the actual electrode potential. In practice, E_{eq} can be considered steady-state potential at open-circuit as current is zero and the system is at steady-state or in other words equilibrium. The overall 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 electrochemistry. The relation between the log i and η is given by the famous Tafel equation:

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

Where, a and b are constants. In short, the linear region of the log i vs. E is fitted with a linear regression. The current intercept (i.e. where η = 0) of this linear regression gives the exchange current density (i_o) and the slope (η/log i) gives the Tafel slope. If it is a reduction reaction, the Tafel slope is cathodic (b_c) and if it is an oxidation reaction, the Tafel slope is anodic (b_a). In the field of corrosion, the i_o is also called the corrosion current (i_{corr}) from which 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.

In the field of electrocatalysis, i_o is the fundamental parameter of an electrochemical reaction, equivalent to equilibrium rate constant. It is a measure of how facile the reaction kinetics is: the higher the i_o, the faster is the reaction rate. From the Tafel slope, the reaction mechanism can also be formulated.

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14.7 Polarization Resistance (i$_{ocp}$ vs. E)

In this plot, current near open circuit potential (i$_{ocp}$) is plotted on the Y-axis and the potential (E) near the open circuit potential is plotted on the X-axis. To obtain this plot, a very slow linear voltammetry is taken within a few millivolts of an open circuit potential (OCP). The plot of current (i$_{ocp}$) and voltage (E) is expected to be linear. From the inverse of the slope (ΔE/Δi$_{ocp}$), which gives the polarization resistance (R$_p$), the corrosion current (i$_{corr}$) can be calculated:

$$R_p = \frac{\Delta E}{\Delta i_{ocp}}$$

$$i_{corr} = 2.303 \frac{b_a + b_c}{b_a b_c} \left(\frac{1}{R_p}\right)$$

Where b$_a$ is the anodic Tafel Slope and b$_c$ is the cathodic Tafel Slope. It's required that b$_a$ and b$_c$ be known beforehand to calculate i$_{corr}$. Because of this, this method can be considered a complimentary method to other methods to find the i$_{corr}$ from which the one-dimensional planar corrosion rate can be calculated using Faraday's law of electrolysis.

14.8 Ragone plot (Specific energy vs. Specific Power)

In this plot, specific energy (Wh/Kg) is plotted vs specific power (W/Kg). This plot is used to determine the energy that can be extracted from an electrochemical device at a given power. Usually, both the Y and X axes are plotted in log scale.

To obtain a Ragone plot, an electrochemical device needs to be discharged at different magnitudes of power. As the magnitude of power increases, the time taken to discharge the device will decrease. The energy of the device at the given power can be calculated by multiplying the applied power by the time taken to discharge the electrochemical device. Both the power and energy are normalized to the mass of the device to report them as specific power and specific energy. Alternatively, they can be normalized to the volume of the device too.

Generally, supercapacitors have high specific power but low specific energy, and batteries have low specific power but high specific energy. Fuel cells have even higher specific energy than batteries.

14.9 Bode Plot (log |Z| and ϕ vs. log f)

When viewing EIS data, a Bode plot is used to plot the log of the modulus of impedance (log |Z|) on the primary Y-axis and the log of frequency (log f) on the X-axis. On the secondary Y-axis, phase angle (ϕ) is plotted. Therefore, the frequency is explicit in this plot and any frequency-dependent phenomenon is easily discernible. It is also easy to distinguish between capacitive vs. resistive vs. inductive phenomena by looking at the phase angle.

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14.10 Nyquist Plot (Z_{im} vs. Z_{re})

When viewing EIS data, a Nyquist plot is used to plot the imaginary impedance (Z_{im} or Z") on the Y-axis and the real impedance (Z_{re} or Z') on the X-axis:

$$Z = Z_{re} - jZ_{im}$$

Where j is the imaginary number $\sqrt{-1}$. In this plot, frequency and phase angle are not explicit. In addition, large impedance values overshadow small impedance values so that these small impedance values are not clearly visible. However, a Nyquist plot can give more information about the charge-transfer processes and diffusion-limited processes involved in electrochemical reactions. Also, solution resistance and charge-transfer resistances can be easily calculated from the X-intercepts. Nyquist plots are also considered to be more visually appealing and more popular than Bode plots.

14.11 Lissajous plot (i_{ac} vs. E_{ac})

In a Lissajous plot, AC current (i_{ac}) is plotted on the Y-axis and AC potential (E_{ac}) is plotted on the X-axis. This plot is useful to assess the linearity of the electrochemical system under test at a given frequency. The linearity is established if one can find a central axis passing through the origin, about which the plot shows symmetry. This is the case when the plot is a circle or an elliptic. If the plot is distorted, the system is non-linear. Generally, at high frequency, this plot is almost a straight line with a positive slope. A middle frequency range, this plot is circular. At low frequency range, this plot is again almost linear with a negative slope.

This plot is out of fashion. Few potentiostats offers option to plot Lissajous plot. The linearity of the system can be quantitively assessed using Kramers-Kronig test. A user must remember that a linear, stable and causal system satisfies Kramers-Kronig test, but vice-versa is necessarily not true.

14.12 Voltage (V) vs. Capacity (Ah/g or Ah/cm^2)

In this plot, voltage (V) is plotted against capacity (Ah/g or Ah/cm^2). In this instance, Voltage (V) refers to the difference between the cathode potential (E_c) and the anode potential (E_a) of an electrochemical device.

$$V = E_c - E_a$$

Potential (E) refers to the potential of the working electrode in a half-cell configuration such as in a three-electrode experiment.

To study the performance of batteries and supercapacitors, they are charged and discharged at a specified current density. In a battery experiment, the magnitudes of charge-discharge currents are alternatively determined based on the capacity of the battery.

For example, a 42 Wh laptop battery operating at a nominal voltage of 11.4 V has a capacity of 3.7 Ah (42 divided by 11.4 = 3.7). That means the battery will be fully discharged in 1 h if the discharge

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current is 3.7 A and the average value of the voltage during the discharge will be 11.4 V. In this case, the battery is said to be discharged at a rate of 1 C. In battery research, charge-discharge cycles are performed at various C rates. The corresponding voltage during charge-discharge is plotted on the Y-axis. Capacity (current × time) is plotted on the X-axis. At a rate of 1 C, full capacity should be obtained in 1 hour.

Sometimes, instead of capacity, time is plotted on the X-axis. This is generally the case for supercapacitors. In such a case, a user might call this a galvanostatic charge-discharge plot. From this plot, a user is interested in studying what the voltage will be at a given C rate.

14.13 Cyclic Efficiency vs. Cycle Number

Cyclic efficiency (also known as coulombic or Faraday efficiency) is the ratio of total charge obtained from a battery during discharge ($Q_{discharge}$) to total charge supplied to a battery during charge (Q_{charge}).

$$Efficiency = \frac{Q_{discharge}}{Q_{charge}}$$

Sometimes, the ratio is multiplied by 100 to express it as a percentage.

Oftentimes in battery research, a single battery is charged and discharged many times to study the long-term stability of a battery. The cyclic efficiency depends on the C rate. At low C rates, the cyclic efficiency tends to be stable over many cycles. When using high C rates, the cyclic efficiency tends to decrease relatively faster as compared to using low C rates. Of course, cycling stability is different from one battery to another.

Generally, supercapacitors have a greater efficiency over several cycles than the batteries. This is due to their intrinsic chemistry. One simple way to think about it is that at high rates (large currents) more heat is dissipated, therefore, stored energy of the device is lost in terms of heat. Another way to think about it is, higher C rates means greater stress. Any system tends to degrade faster at high stress than at lower stress.

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15   Appendix

15.1   How to reset Squidstat calibration files

The performance of a potentiostat can be checked using a dummy cell or using resistors and capacitors with accurate values. If the values of the resistors and capacitors are off by a significant deviation compared to the advertised performance specifications, a solution can be to reset the factory calibration of the Squidstat. This can be done by users independently and is a more convenient solution than having to ship the Squidstat to the Admiral Instruments office. Below is a step-by-step guide to recalibrate a Squidstat.

Step 1: Please close the Squidstat User Interface (SUI). Calibration cannot be done while the SUI is running.

Step 2: Please make sure the Squidstat is powered on and is connected to the computer via a USB cable. Find the port that connects your computer to the Squidstat.

In Windows 10, go to Settings and then Devices. Find the "Teensy USB serial (COM X)," where X is a number, among the list of the "Other devices". This can also be found using "Control Panel > Hardware and Sound > Devices and Printers" or going to Device Manager from Control Panel and then clicking on "Ports (COM and LPT)" in older versions of Windows.

This is to make sure that the hardware is properly connected to the computer. Please also note the COM port number that will be used in Step 8.

You may need to turn the Squidstat on and off or unplug and plug the USB cable while the Squidstat is on to see the "Teensy USB serial (COM X)."

Step 3: A folder will be provided named "Debugging tools". Open this folder and locate the folder titled "Potentiostat". Copy and paste this folder directly to the C:/ drive. You might need to unzip this folder before pasting into the C:/ drive.

Step 4: In the folder "Debugging tools," locate the sub folder with the serial number of your Squidstat. If you have a Squidstat Plus or a Solo, the folder will have a file called "cal.csv" inside. If you are recalibrating a Squidstat Prime, in the folder with the serial number of your prime, four folders labeled Ch0, Ch1, Ch2 and Ch3 will be found. If you are facing the front panel of a Squidstat Prime, Ch0 is the first channel on your left that is labeled Ch 1. Likewise, Ch1 is Ch 2, Ch2 is Ch 3 and Ch3 is Ch 4 on the Squidstat Prime. So, inside the folder Ch0, you will find calibration file "cal.csv" for the channel 1 of a Squidstat Prime and so on.

Step 5: Copy the file "cal.csv". Go to C:/ drive and find the folder "Potentiostat" that was previously copied and pasted. Open the folder and keep opening the subsequent folders, "SquidstatCalibrator", "Bin" and "Debug". After you open the last folder titled "Debug", paste the

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"cal.csv" file into this folder. Already two files, an .exe file "SquidstatCalibrator.exe" and a .txt file "SquidstatCalibrator.txt" should be present is this folder.
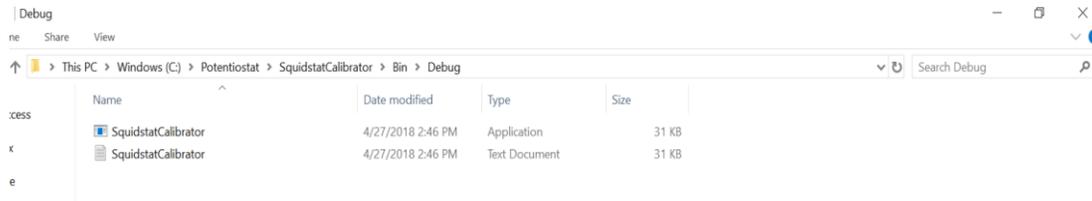

Step 6: Now open the Windows command prompt. To open the command prompt, first click the windows icon or start menu. In the search box, type command prompt or just cmd and right-click the result and select "run as an administrator".

Step 7: Open the folder or directory "Debug" where the "cal.csv" file was copied and pasted in the command prompt window by using "cd" command as shown below:

<div align="center">cd C:/potentiostat/squidstatcalibrator/bin/debug</div>

This command is not case-sensitive.
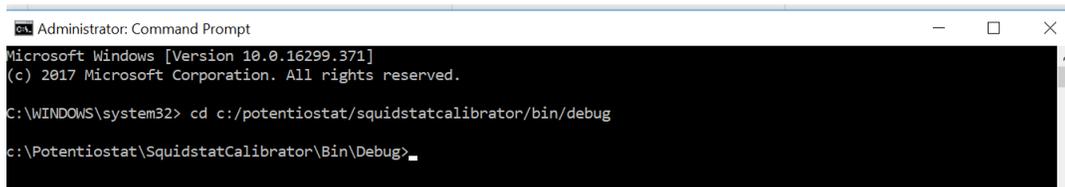
Step 8: Run squidstatcalibrator.exe file in the "Debug" folder by using the following command if you are calibrating a Squidstat Plus or Solo.

<div align="center">squidstatcalibrator.exe v2 0 COMX calibrate</div>

In this command, replace X with the COM port number you found in **Step 2**. For example, if you found "Teensy USB serial (COM 2)" in **Step 2**, you will type:

<div align="center">squidstatcalibrator.exe v2 0 COM2 calibrate</div>

If you are recalibrating a channel of a Squidstat Prime, use the following command:

<div align="center">squidstatcalibrator.exe v2 N COMX calibrate</div>

Replace N with the corresponding folder number of your channel in the folder with serial number of your Squidstat Prime and X with the COM port number. For example, if you wish to recalibrate

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channel 3 of your Squidstat Prime and you found "Teensy USB serial (COM 1)" in **Step 2**, you should type:

squidstatcalibrator.exe v2 2 COM1 calibrate

Step 9: Press enter after typing the suggested command.

Step 10: Wait for a few seconds for the program to finish running. A command "press any key to continue" should appear if the calibration program ran successfully. If you get an error, double-check your command for spelling, and make sure you copied and pasted the right "cal.csv" file into the right location.

A common mistake is forgetting to close the Squidstat User Interface (SUI) software.

Please repeat the same process for other channels of a Squidstat Prime or other Squidstats.

Step 11: Test again the performance of the Squidstat using a dummy cell or a circuit. If you see significant deviation again, please contact Admiral Instruments.

15.2   Troubleshooting Squidstats

If a Squidstat is not behaving as expected, the problem might be with the Squidstat or with the experimental set up. Sometimes, it is obvious which one is at fault, while at other times, it might not be obvious.

Although this list of solutions to common issues is presented for customers to address problems on their own, customers are encouraged to contact Admiral Instruments at any time for live support. Our phone number is +1 480 256 8706 and email is support@admiralinstruments.com

15.2.1   Issue: Not sure if the Squidstat is working properly

To check whether a Squidstat is working properly or not, disconnect the Squidstat from the electrochemical cell and connect it to a dummy cell. If the dummy cell is not provided, use a resistor with a known value. Connect the working electrode clip and working electrode sense clip to one side of the resistor. Connect the reference electrode clip, counter electrode clip, and counter electrode sense clip to the other side of the resistor. Run a cyclic voltammetry experiment by setting the voltages limits as +0.5 and − 0.5 with respect to the reference. Use autorange or fixed range by calculating expected current from Ohm's law (V = iR). For example, if you use a 10 kΩ resistor, the maximum current should be ± 50 µA, irrespective of the scan rate.

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The current vs voltage graph should be a linear line. The forward and the backward sweep should overlap each other perfectly. The maximum current should be as calculated within the accuracy of the current range. The resistance can be calculated from the slope of the curve and should be within the accuracy of the resistance value of the resistor.

If the response is not as expected, repeat the experiment with a different resistor. If the problem still exists, repeat the experiment with a spare cell cable (if available). For a Squidstat Plus, this can be done using the short high-frequency cable. If the problem goes away when using a spare cable, the cell cable needs replacing. If the problem persists when using a spare cable, the Squidstat needs to be serviced. The first service step to try is resetting the calibration constants.

The same resistor can also be used to run chronopotentiometry or chronoamperometry. For chronoamperometry, set the voltage as + 0.5 V and see if you get the + 50 µA current for a 10 kΩ resistor. Alternatively, set the current at + 50 µA and see if you get +0.5 V for the 10 kΩ resistor during chronopotentiometry. If the values are within the specified accuracy of the instrument, the Squidstats are working well. If the values are off, the Squidstat might need to have its calibration constants reset.

15.2.2   Issue: Not sure the reference electrode is working properly

Most issues regarding an electrochemical cell arise due to a bad reference electrode. Always check if there are air or gas bubbles trapped inside the reference electrode body and the frit. By gentle tapping, you might be able to get rid of the bubbles. Also, check if the reference electrode is filled with the appropriate filling solution to the level recommended by the manufacturer.

Some of the issues that arise due to a faulty reference electrode are:
a. The voltages are off. In this case, the reference electrode might need recalibration. After recalibration, correct for the voltage offset during data analysis.
b. If you see a ringing phenomenon, the impedance of the reference electrode might be too high. Replace the frit following the instructions from the reference electrode manufacturer.
c. The reference electrode needs frequent recalibration. If so, the filling solution might be contaminated. Change the filling solution. If the problem is not solved, consider replacing the frit. Replace the frit following the instructions from the reference electrode manufacturer.
d. The level of the filling solution keeps decreasing. The frit might be leaky. Replace the frit following the instructions from the reference electrode manufacturer.
e. You can ultimately test if the reference electrode is working or not by replacing it with a platinum/palladium/gold/carbon wire and running the experiment. Alternatively, you can connect the reference electrode clip to the counter electrode and run the experiment. If the experiment runs with a shift in voltage, the reference electrode is the problem. You can try the suggestions listed above like replacing the filling solution and the frit before being absolutely convinced that the reference electrode needs to be replaced with a new one.

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15.2.3 Issue: Not sure the working electrode is working properly

If you suspect the working electrode is not working properly, repeat your experiment with an appropriate wire instead of your working electrode. Clean the wire before the experiment using the proper procedure to remove any organic and inorganic impurities. The wire should be kept steady to avoid noise during the experiment. Also keep in mind how much the wire is immersed into the solution. If the response is satisfactory, the working electrode might be the problem.

The working electrode surface might be contaminated with organic and inorganic impurities. Please clean the working electrode surface using proper procedures. Many solid electrodes can be cleaned and activated by polishing them. Please consult the instructions for proper polishing technique provided by the manufacturer of the electrodes or the polish.

15.2.4 Issue: Squidstat Prime or Solo is plugged in but won't power on

If the Squidstat Prime or Solo does not power on, check for a blown fuse by opening the AC inlet block on the back or side of the hardware enclosure. If it is blown, use the replacement in the same compartment. If no replacements remain, more can be purchased from typical hardware or electronics retail stores. Be sure to use slow-blow fuses that match the amperage value listed on the CE certification label next to the AC inlet block.

15.2.5 Issue: Squidstat hardware and software won't connect or sync

To re-establish communication between the Squidstat and your PC, follow these steps in order:
a. Ensure that the USB cable connecting the Squidstat to your PC is shorter than 13ft (4m). Eliminate the use of USB extension cables where possible.
b. In Windows 10, go to Settings and then Devices. Find the "Teensy USB serial (COM X)," where X is a number, among the list of the "Other devices". This can also be seen using "Control Panel > Hardware and Sound > Devices and Printers" or going to Device Manager from Control Panel and then clicking on "Ports (COM and LPT)" in older versions of Windows.
c. Appearance of the Teensy USB serial (COM X) ensures that the driver for the Squidstat is installed and that the potentiostat is being recognized.
d. If "Teensy USB serial (COM X)" cannot be found, re-install the MCU_USB_driver_install.exe file from the Squidstat Installation folder.
e. Disconnect the USB cable, cycle power to the Squidstat (making sure to disconnect the channel cables to any experiments), reconnect the USB, and restart the software.

15.2.6 Issue: There is unexpected noise observed on one or more channels

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 turning off the device suspected of causing interference to see if it disappears. If the device causing the interference cannot be moved elsewhere, we recommend plugging the device into a different power outlet.

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15.2.7   Issue: The real-time values are nonsensical in the manual control tab

It is likely that the Squidstat needs to be recalibrated. Please see the chapter on how to recalibrate a Squidstat. The first step is to contact Admiral Instruments and ask for a calibration file based on the serial number of the Squidstats

15.2.8   Issue: The voltage values are off

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 +/-10 V compliance range of the Squidstat Prime and Solo, the Squidstat probably needs recalibration. This might also be the case when the voltage values deviate significantly from an expected value.

15.2.9   Issue: The SUI stops after starting an experiment

This might be due to several reasons.
This might be due to improper connection of the cell cables. Please make sure that the electrode clips are connected properly to their respective electrodes.

   a. The firmware might not be up to date. Please always make sure that the latest version of the SUI is being used. Based on the version, the firmware might need updating. Please see how to update firmware.
   b. The Squidstat needs to have its calibration constants reset. Please see the chapter on how to recalibrate a Squidstat.
   c. The Squidstat will immediately stop if the current and voltage are out of the specified range. This can happen due to several reasons:

      • Due to entrapment of air or gas bubbles before and during the experiment. Find the bubbles and remove them.
      • The electrodes are not properly immersed into the electrolyte solution.
      • The electrode clips are not touching the electrodes. Make sure the clips are rust free and hold the electrodes properly.
      • The response of the electrochemical device under test is out of range. To know this, use the manual control tab to see what is the current near the set voltage or what is the voltage near the set current. If they are out of range, please adjust your experimental settings accordingly or use a potentiostat with a high current and voltage range.

15.2.10 Issue: Noise is observed during some intervals when running voltammetry

This is most likely due to auto-ranging. Please select the **Approx. max. current** option and enter the maximum current expected during the experiment in the input box. For more information, please review the application note titled, "Ever Wonder Why Your Potentiostat Starts Clicking During Experiments?".

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15.2.11 Issue: No data is being displayed when running a pulse experiment

This is most likely due to auto-ranging. Please select the **Approx. max. current** option and give the maximum current expected during the experiment in the input box. See the application note on auto ranging vs fixed ranging titled, "Ever Wonder Why Your Potentiostat Starts Clicking During Experiments?" Additionally, also try increasing the pulse duration if possible.

16 Calculating current/voltage accuracy and resolution

Here is an easy-to-follow guide to calculate accuracy and resolution of the Squidstats.

The specifications of the Squidstat Plus is used as an example. This same method can be followed to calculate current/voltage accuracy and resolution of other members of the Squidstat family of instruments.

Potential Accuracy

The specification of the Squidstat Plus for both applied and measured potential accuracy is 0.1% of reading, down to 1 mV.

For example, if you read or set potential at 5.2 V, the accuracy is: 0.1% of 5.2 V or;

$$\frac{0.1}{100} \times 5.2\,\text{V} = 0.0052\,\text{V}$$

In other words, the potential would be 5.2 ± 0.0052 V.

If the potential is 1 V or less, 0.1 % of 1 V is 0.001 V or 1 mV which is the maximum accuracy. So, if the potential is 0.5 V, the accuracy will still be ± 0.001 V.

Potential Resolution

The resolution of a digital potentiostat is associated with conversion of a digital signal to an analog signal in cases where potential is applied. When potential is measured, it is a conversion of an analog signal to a digital signal. A digital to analog converter (DAC) is used for the first process and an analog to digital converter (ADC) is used for the second process.

The maximum potential that can be applied using a Squidstat Plus is from -10 to +10 V; therefore, the potential range is [+10-(-10)] V= 20 V.

The DAC or ADC used in Squidstat Plus is a 16 bit processor, which means that it can digitize voltages from -10 to +10 V in 2^{16} (= 65536) digital steps. So, the resolution is:

$$\frac{20\,\text{V}}{65536} = 0.000305\,\text{V or } 305\,\mu\text{V}$$

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This means that the Squidstat Plus cannot distinguish between two voltage data points if the difference between them is less than 305 µV, or 0.305 mV. So, during a voltage ramp, the Squidstat Plus cannot sample or apply potential at intervals of less than 0.305 mV.

Current Accuracy

The Squidstat Plus has 8 current ranges:

97 mA	to	1 A
9.7 mA	to	97 mA
970 µA	to	9.7 mA
97 µA	to	970 µA
9.7 µA	to	97 µA
970 nA	to	9.7 µA
96 nA	to	970 nA
0 nA	to	96 nA

The performance specification of the Squidstat Plus for measured or applied current is 0.1% of range within 1 nA. For example, if the measured or applied current is 5.0 mA, the corresponding range is 970 µA to 9.7 mA, therefore the accuracy is within:

$$\frac{0.1}{100} \times 9.7 \text{ mA} = 0.0097 \text{ mA or } 9.7 \text{ µA}$$

In other words, the measured or applied current would be 5.0 mA ± 9.7 µA.

The lowest current range is 0 to 96 nA; therefore, the highest obtainable accuracy is ± 1 nA.

Current Resolution

Like the voltage resolution, the current resolution is also associated with the conversion of an analog signal to a digital signal and vice versa.

The specification of the Squidstat Plus for current resolution is 0.003% of range. Therefore, if a measured or applied current is 0.5 mA, the range is 970 µA to 9.7 mA. Then the resolution is;

$$\frac{0.003}{100} \times (9.7 \text{ mA} - 970 \text{ µA}) = 0.262 \text{ µA}$$

That means that the Squidstat Plus cannot distinguish between two current data points if the difference between them is less than 0.262 µA in the 970 µA to 9.7 mA range.

The best resolution obtainable is 2.9 pA in the range of 0 to 96 nA. When during a current ramp, the Squidstat Plus cannot sample or apply current at intervals of less than 2.9 pA in any current range.

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