

Model 2450-EC and 2460-EC Electrochemistry Lab Systems

User's Manual

077110401 / December 2015

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KEITHLEY

A Tektronix Company

Model 2450-EC and 2460-EC

Electrochemistry Lab Systems

User's Manual

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The following safety precautions should be observed before using this product and any associated instrumentation. Although some instruments and accessories would normally be used with nonhazardous voltages, there are situations where hazardous conditions may be present.

This product is intended for use by qualified personnel who recognize shock hazards and are familiar with the safety precautions required to avoid possible injury. Read and follow all installation, operation, and maintenance information carefully before using the product. Refer to the user documentation for complete product specifications.

If the product is used in a manner not specified, the protection provided by the product warranty may be impaired.

The types of product users are:

Responsible body is the individual or group responsible for the use and maintenance of equipment, for ensuring that the equipment is operated within its specifications and operating limits, and for ensuring that operators are adequately trained.

Operators use the product for its intended function. They must be trained in electrical safety procedures and proper use of the instrument. They must be protected from electric shock and contact with hazardous live circuits.

Maintenance personnel perform routine procedures on the product to keep it operating properly, for example, setting the line voltage or replacing consumable materials. Maintenance procedures are described in the user documentation. The procedures explicitly state if the operator may perform them. Otherwise, they should be performed only by service personnel.

Service personnel are trained to work on live circuits, perform safe installations, and repair products. Only properly trained service personnel may perform installation and service procedures.

Keithley Instruments products are designed for use with electrical signals that are measurement, control, and data I/O connections, with low transient overvoltages, and must not be directly connected to mains voltage or to voltage sources with high transient overvoltages. Measurement Category II (as referenced in IEC 60664) connections require protection for high transient overvoltages often associated with local AC mains connections. Certain Keithley measuring instruments may be connected to mains. These instruments will be marked as category II or higher.

Unless explicitly allowed in the specifications, operating manual, and instrument labels, do not connect any instrument to mains.

Exercise extreme caution when a shock hazard is present. Lethal voltage may be present on cable connector jacks or test fixtures. The American National Standards Institute (ANSI) states that a shock hazard exists when voltage levels greater than 30 V RMS, 42.4 V peak, or 60 VDC are present. A good safety practice is to expect that hazardous voltage is present in any unknown circuit before measuring.

Operators of this product must be protected from electric shock at all times. The responsible body must ensure that operators are prevented access and/or insulated from every connection point. In some cases, connections must be exposed to potential human contact. Product operators in these circumstances must be trained to protect themselves from the risk of electric shock. If the circuit is capable of operating at or above 1000 V, no conductive part of the circuit may be exposed.

Do not connect switching cards directly to unlimited power circuits. They are intended to be used with impedance-limited sources. NEVER connect switching cards directly to AC mains. When connecting sources to switching cards, install protective devices to limit fault current and voltage to the card.

Before operating an instrument, ensure that the line cord is connected to a properly-grounded power receptacle. Inspect the connecting cables, test leads, and jumpers for possible wear, cracks, or breaks before each use.

When installing equipment where access to the main power cord is restricted, such as rack mounting, a separate main input power disconnect device must be provided in close proximity to the equipment and within easy reach of the operator.

For maximum safety, do not touch the product, test cables, or any other instruments while power is applied to the circuit under test. ALWAYS remove power from the entire test system and discharge any capacitors before: connecting or disconnecting cables or jumpers, installing or removing switching cards, or making internal changes, such as installing or removing jumpers.

Do not touch any object that could provide a current path to the common side of the circuit under test or power line (earth) ground. Always make measurements with dry hands while standing on a dry, insulated surface capable of withstanding the voltage being measured.

For safety, instruments and accessories must be used in accordance with the operating instructions. If the instruments or


accessories are used in a manner not specified in the operating instructions, the protection provided by the equipment may be impaired.


Do not exceed the maximum signal levels of the instruments and accessories, as defined in the specifications and operating information, and as shown on the instrument or test fixture panels, or switching card.


When fuses are used in a product, replace with the same type and rating for continued protection against fire hazard.

Chassis connections must only be used as shield connections for measuring circuits, NOT as protective earth (safety ground) connections.

If you are using a test fixture, keep the lid closed while power is applied to the device under test. Safe operation requires the use of a lid interlock.


If a  screw is present, connect it to protective earth (safety ground) using the wire recommended in the user documentation.

The  symbol on an instrument means caution, risk of danger. The user must refer to the operating instructions located in the user documentation in all cases where the symbol is marked on the instrument.

The  symbol on an instrument means caution, risk of electric shock. Use standard safety precautions to avoid personal contact with these voltages.

The  symbol on an instrument shows that the surface may be hot. Avoid personal contact to prevent burns.

The  symbol indicates a connection terminal to the equipment frame.

If this  symbol is on a product, it indicates that mercury is present in the display lamp. Please note that the lamp must be properly disposed of according to federal, state, and local laws.

The **WARNING** heading in the user documentation explains dangers that might result in personal injury or death. Always read the associated information very carefully before performing the indicated procedure.

The **CAUTION** heading in the user documentation explains hazards that could damage the instrument. Such damage may invalidate the warranty.

Instrumentation and accessories shall not be connected to humans.

Before performing any maintenance, disconnect the line cord and all test cables.

To maintain protection from electric shock and fire, replacement components in mains circuits — including the power transformer, test leads, and input jacks — must be purchased from Keithley Instruments. Standard fuses with applicable national safety approvals may be used if the rating and type are the same. Other components that are not safety-related may be purchased from other suppliers as long as they are equivalent to the original component (note that selected parts should be purchased only through Keithley Instruments to maintain accuracy and functionality of the product). If you are unsure about the applicability of a replacement component, call a Keithley Instruments office for information.

To clean an instrument, use a damp cloth or mild, water-based cleaner. Clean the exterior of the instrument only. Do not apply cleaner directly to the instrument or allow liquids to enter or spill on the instrument. Products that consist of a circuit board with no case or chassis (e.g., a data acquisition board for installation into a computer) should never require cleaning if handled according to instructions. If the board becomes contaminated and operation is affected, the board should be returned to the factory for proper cleaning/servicing.

Safety precaution revision as of January 2013.

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Model 2450-EC and 2460-EC User's Manual

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Introduction

The Model 2450 or Model 2460 Electrochemistry Lab Systems are alternatives to traditional potentiostats. These systems include a SourceMeter® SMU Instrument test scripts for performing common electrochemistry tests, an electrochemistry translation cable that connects the instrument to a 2, 3, or 4-terminal electrochemical cell, and a USB flash drive that contains the test scripts and documentation.

The Models 2450-EC and 2460-EC test scripts enable the user to perform the following applications:

- **Cyclic Voltammetry:** Potential is swept at a user-programmable scan rate between two to four defined potential vertices while current is measured.
- **Open Circuit Potential:** Measures the cell potential difference between two electrodes with high input impedance as a function of time.
- **Potential Pulse and Square Wave with Current Measure:** Sources potential at programmable peak and base levels while current is recorded at a user-defined position at the pulse peak level.
- **Current Pulse and Square Wave with Potential Measure:** Sources current at programmable peak and base levels while potential is recorded at the pulse peak level.
- **Chronoamperometry:** Potential is stepped to a programmed value while the resulting current is measured as a function of time.
- **Chronopotentiometry:** Current is stepped to a programmed value while the resulting potential is measured as a function of time.

The contents of the Model 2450-EC and Model 2460-EC Electrochemistry Lab Systems consist of the following:

- One Model 2450 or Model 2460 SourceMeter® SMU Instrument with six electrochemistry test scripts in internal memory.
- One Electrochemistry Translation Cable Accessory Kit which includes one cable and four insulated miniature alligator clips.
- A USB flash drive containing the files and folders in the following table:

| File or folder | Description |
|---------------------------------|---|
| CyclicVoltammetry.tsp | Test script to perform cyclic voltammetry |
| OpenCircuitPotential.tsp | Test script to measure the open circuit potential |
| Chronoamperometry.tsp | Test script to measure the current as a function of time at a constant potential |
| Chronopotentiometry.tsp | Test script to measure the potential as a function of time at a constant current |
| CurrentPulseAndSquareWave.tsp | Test script to output a current pulse or square wave while measuring the potential |
| PotentialPulseAndSquareWave.tsp | Test script to output a current pulse or square wave while measuring the current |
| 07711040x.pdf | User instructions for the Model 2450-EC and Model 2460-EC Electrochemistry Lab System (this document) |
| 07134730x.pdf | Model 2450/60-EC Electrochemistry Lab System Quick Start Guide |

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WARNING

For protection against electrical shock while using the Keithley Instruments Model 2450, Model 2460 or other source measure units (SMU), be sure to read all the safety information and obey all warning and caution statements.

The safety interlock circuit should not be activated if working with less than 42 V, but the circuit must be activated to enable the high voltage output. The interlock helps facilitate safe operation of the equipment in a test system. Bypassing the interlock could expose the operator to hazardous voltages that could result in personal injury or death.

NOTE

The SourceMeter® instruments must have the version of firmware listed below:

- Model 2450 must have firmware version 1.3.0 or later.
- Model 2460 must have firmware version 1.3.0 or later.

Cyclic voltammetry

Cyclic voltammetry (CV), a type of potential sweep method, is the most commonly-used measurement technique. CV involves linearly sweeping an electrode's potential as a function of time and measuring the resulting current that flows through the circuit (typically a three-electrode electrochemical cell). The resulting I-V data provides important electrochemical properties about the analyte under investigation.

You can make cyclic voltammetry measurements using the test script included with the Keithley Instruments Model 2450-EC or Model 2460-EC Electrochemistry Lab System. The Models 2450-EC and 2460-EC contain a test script (Cyclic Voltammetry) for performing CV measurements automatically on the front panel of the instruments.

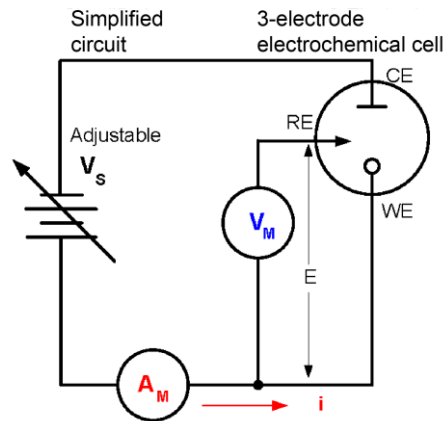
The CV test script has adjustable parameter settings and enables real-time graphing of a voltammogram on the display of the 2450 or 2460 instrument without using a computer. Test parameters are input through a series of dialog boxes that appear on the front panel of the instrument. After the test executes, the data is stored on a USB drive on the front panel of the instrument.

The following topics in this section describe how to set up and run the `CyclicVoltammetry.tsp` test script.

Simplified cyclic voltammetry basics

A typical electrochemical measurement circuit consisting of an electrochemical cell, an adjustable voltage source (V_s), an ammeter (A_M), and a voltmeter (V_M), is shown in the following figure.

Figure 1: Simplified measurement circuit for performing cyclic voltammetry



The three electrodes of the electrochemical cell are the working electrode (WE), reference electrode (RE), and the counter, or auxiliary, electrode (CE). The voltage source (V_s) for the potential scan is applied between the working electrode and the counter electrode. The potential (E) between the reference electrode and the working electrode is measured with the voltmeter (V_M). The overall voltage (V_s) is adjusted to maintain the targeted potential at the working electrode with respect to the reference electrode. The resulting current (i) flowing to the working electrode is measured with the ammeter (A_M). This process is usually repeated for a range of E .

An example procedure to make a measurement for each point in the scan (E_i):

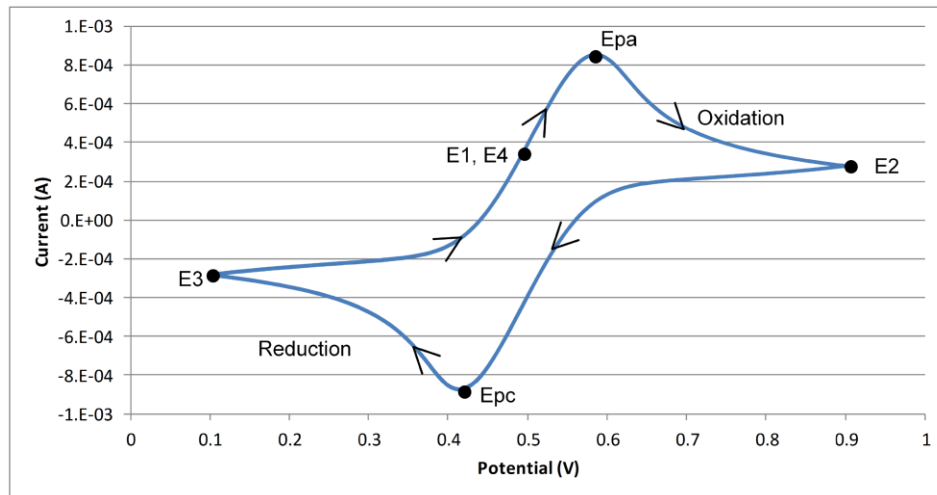
1. Select a potential (E) for RE with respect to WE.
2. Adjust the voltage across the entire cell (CE to WE) to get desired E (closed loop control).
3. Measure i .
4. Select (step) a new E and repeat the procedure until the scan is finished. The procedure can be a single sweep between two potentials (linear sweep voltammetry) or one in which the sweep is inverted when a certain potential is reached (cyclic voltammetry). This cycle may be repeated multiple times during an experiment.
5. Plot the results and derive parameters of interest from the data.

Once the experiment is finished, the measured current is plotted as a function of the potential in a graph known as a voltammogram. The example voltammogram in the following figure shows four voltage vertices:

- E1 (initial potential)
- E2 (second, switching potential)
- E3 (third, switching potential)
- E4 (final potential)

The voltage peaks in the waveform are the anodic (E_{pa}) and the cathodic (E_{pc}) peak potentials.

Figure 2: Example voltammogram generated by a 2450-EC



In this example, the scan begins at E1 and the potential becomes more positive, causing the anodic current (I_{pa}) to rise rapidly and peak at the anodic peak potential (E_{pa}).

At E2, the scan direction switches to negative for the reverse scan. As the current becomes more negative, cathodic current flows as the electrode process is reduced. A cathodic peak potential occurs at E_{pc} .

At E3, the direction reverses again and the voltage is swept until it reaches E4. From the potential sweep, important information about the experiment can be derived and analyzed.

Device connections

NOTE

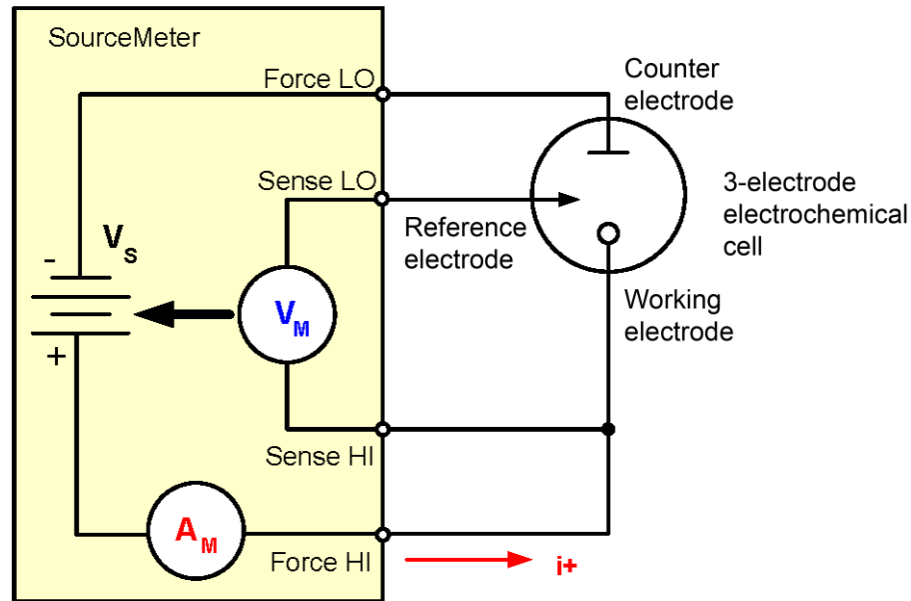
Before making physical connections from the source-measure unit (SMU) to the cell, the output of the SMU must be set to the high-impedance output-off state. To set the output-off state to high impedance:

1. Press the **MENU** key.
2. Under Source, select **Settings**.
3. Set the Output Off State to High Impedance.
4. Press the **HOME** key to return to the home screen.

Before beginning the experiment, the 2450 or 2460 must be properly connected to the three-electrode electrochemical cell. To perform cyclic voltammetry using a SMU, the instrument is set to force voltage and measure current in a four-wire (remote sense) configuration. Using the electrochemistry translation cable, connect the four terminals of the SMU to the three-terminal electrochemical cell as shown in the following figure. A description of the connections follows the figure.

Refer to the Model 2450-EC / 2460-EC Electrochemistry Lab System Quick Start Guide for information on making connections from the SourceMeter®.

Figure 3: Connecting a SourceMeter to an electrochemical cell for cyclic voltammetry



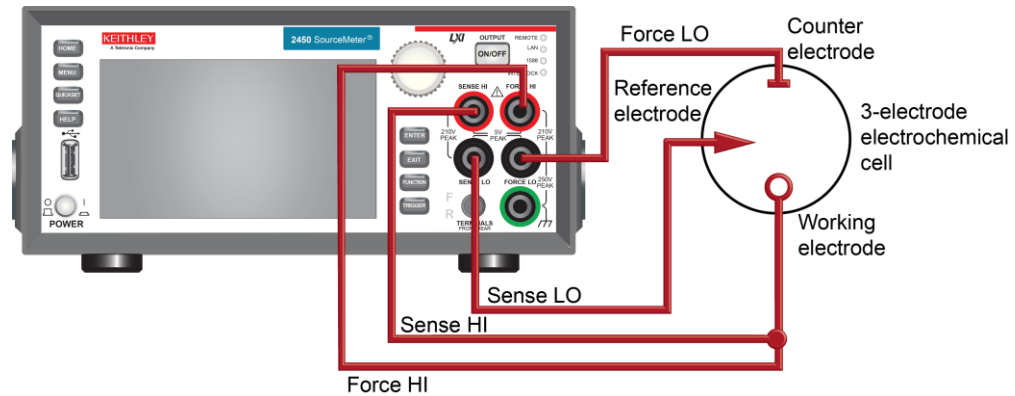
- Connect the Force HI and Sense HI terminals to the working electrode. The voltage at this terminal is forced and the current is measured from the working electrode to the counter electrode.
- Connect the Sense LO terminal to the reference electrode.
- Connect the Force LO terminal to the counter electrode.

The SMU measures the voltage difference between the working and reference electrodes (between Sense HI and Sense LO terminals) and ensures the voltage is kept constant. When the instrument is programmed to source voltage in a remote sense configuration, internal sensing provides a feedback voltage that is measured and compared to the programmed voltage level. If the feedback voltage is less than the programmed voltage level, the voltage source is increased until the feedback voltage equals the programmed voltage level. This compensates for the voltage drop in the test leads and ensures that the programmed voltage level is delivered to the cell.

Once the hardware is properly configured, the cyclic voltammetry test can be executed.

The figure below shows the front-panel connections on the 2450; the connections are the same for the 2460.

Figure 4: Model 2450 or 2460 front-panel connections



Test script parameters

The cyclic voltammetry test sets certain parameters for the cyclic voltammetry test for you, and then prompts you to enter additional parameters. The parameters set by the script follow.

- Potential range: ± 5 V
- Voltage step size during ramping:
 - 100 μ V (1 mV per second \leq scan rate < 35 mV per second)
 - 1 mV (35 mV per second \leq scan rate < 350 mV per second)
 - 10 mV (350 mV per second \leq scan rate \leq 3500 mV per second)
- Scan rate: 1 mV per second to 3500 mV per second
- Current measurement range (full scale):
 - 2450: 10 μ A, 100 μ A, 1 mA, 10 mA, 100 mA, 1 A
 - 2460: 1 mA, 10 mA, 100 mA, 1 A, 4 A, 5 A, 7 A
- Number of cycles: 1 to 100
- Sampling interval units: Points/Test, Points/Cycle, Seconds/Point, Points/Second

Test procedure overview

The following list outlines the basic steps to perform a cyclic voltammetry sweep and save the data to a USB flash drive using the 2450 or 2460. Details of each step are described more thoroughly in the following sections.

1. Select the TSP command set on the 2450 or 2460.
2. Before making physical connections from the SourceMeter to the cell, set the output of the SourceMeter to the high-impedance output-off state.
3. Prepare the analyte and assemble the electrochemical cell.
4. Connect the SourceMeter instrument output terminals to the three electrodes from the electrochemical cell using the electrochemistry translation cable.
5. Execute the test script.
6. Measure the open circuit potential (Eoc).
7. Define the potential sweep parameters (number of vertices, voltage magnitude, and reference voltage).
8. Select the scan rate.
9. Choose the number of scan cycles.
10. Select the current measurement range.
11. Specify the sampling interval units.
12. Enter the file name if you choose to save the data to the USB flash drive.
13. Start the experiment.
14. While the scan is generating, the results are displayed on the graph tab.
15. When the scan completes, you have the option to finish or repeat the test.
16. View the results saved to the USB flash drive.

Step 1: Select the TSP command set

By default, the command set is SCPI. Configure the Model 2450 or 2460 to use the TSP command set.

To configure the 2450 or 2460 to use the TSP command set:

1. Press the **MENU** key.
2. Under System, select **Settings**.
3. Select the button next to Command Set and select **TSP**.
4. When prompted to reboot, select **Yes**.

Step 2: Place the output in the high-impedance output-off state

Before making physical connections from the SourceMeter to the cell, set the output of the instrument to the high-impedance output-off state. When the high impedance output-off state is selected, the output relay opens, disconnecting the instrument from the load.

To set the output of the 2450 or 2460 to the high-impedance output-off state:

1. Press the **MENU** key.
2. Under Source, select **Settings**.
3. Select the button next to Output Off State and select **High Impedance**.
4. Press the **HOME** key to return to the home screen.

Step 3: Prepare the analyte and assemble the electrochemical cell

Step 4: Connect the four output terminals of the SourceMeter instrument to the three electrodes of the electrochemical cell using the supplied cable assembly

Specific information on making connections from the SourceMeter to the device is described in [Device connections](#) (on page 1-5) and also in the Model 2450-EC / 2460-EC Electrochemistry Lab System Quick Start Guide.

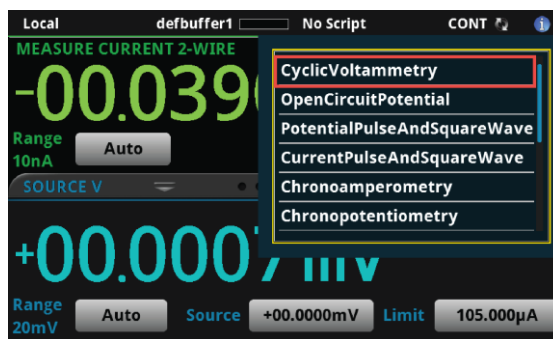
Step 5: Execute the cyclic voltammetry test script

You can execute the script directly from the USB flash drive, internal instrument memory, or using Keithley Test Script Builder (TSB), which is a full-featured editor available for download from the [Keithley Instruments website](http://www.tek.com/keithley) (<http://www.tek.com/keithley>).

To execute the script:

1. At the top of the home screen, touch the active script indicator. A list of available scripts is displayed.

Figure 5: List of available internal scripts

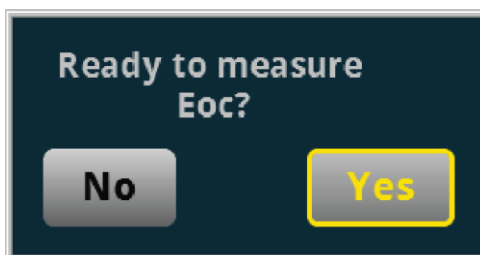


2. Select `CyclicVoltammetry`. The script begins to execute immediately.

Step 6: Acquire the open circuit potential (Eoc)

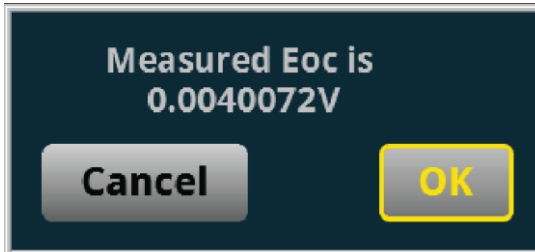
When the script has executed, it prompts you to indicate if you are ready to measure the open circuit potential (Eoc). If the experiment is set up and measurements are ready to be taken, select **Yes** to begin the test. Select **No** to quit the program.

Figure 6: Ready to measure Eoc?



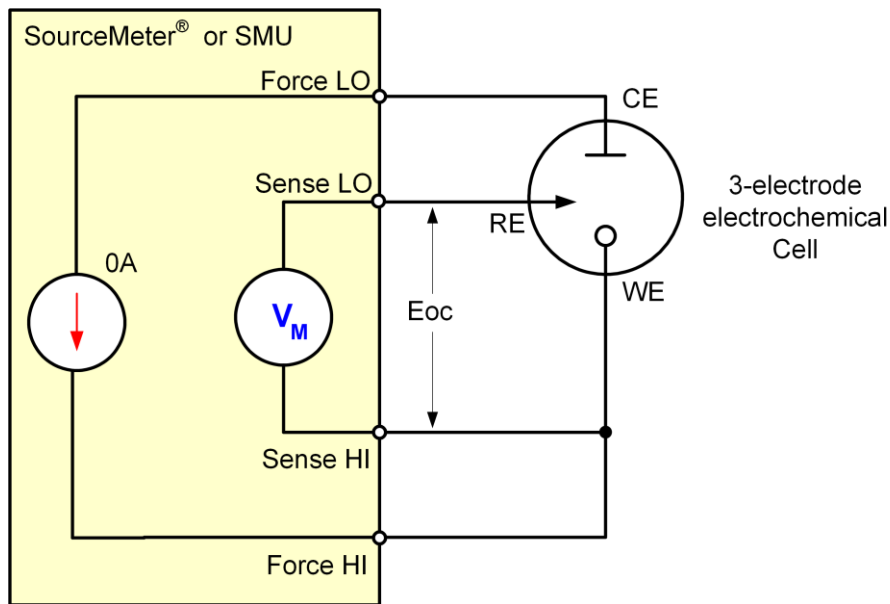
The instrument measures the open circuit potential (E_{oc}) of the electrochemical cell in voltmeter mode, which has high input impedance. After the E_{oc} is measured, the value is displayed in a user prompt, as shown in the following figure. Select **OK** if this value is acceptable; select **Cancel** to quit the program. The measured E_{oc} value is saved in the internal buffer of the instrument, `eocBuffer`.

Figure 7: Measured open circuit potential



When the open circuit potential is measured, no current or voltage is applied to the cell. This voltage measurement is made using the Model 2450 or 2460 to source current and measure voltage (see the following figure). The instrument sources 0 A on the 1e-6 range using a 4-wire configuration.

Figure 8: Measuring open circuit potential of an electrochemistry cell



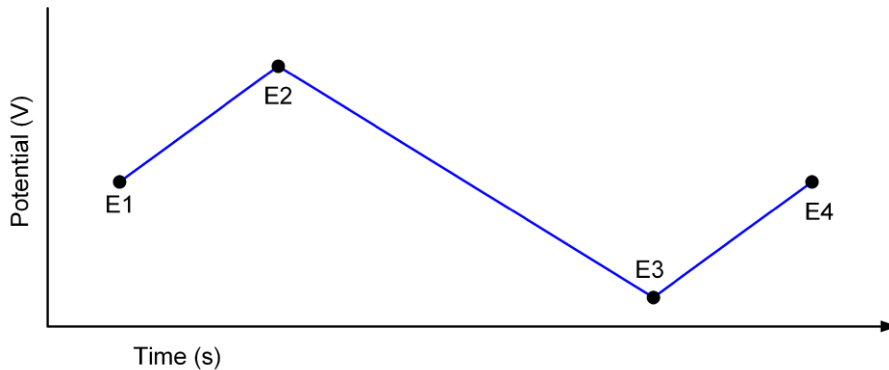
You can use this potential measurement as the zero measurement when defining the voltage vertices. When you do this, the E_{oc} measurement gets added to the voltage.

Step 7: Define the potential sweep parameters

After the open circuit voltage is measured, the parameters of the potential sweep must be defined: Number of vertices, voltage magnitude, and reference voltage.

You can select up to four voltage vertices, which are defined as E1 (or E initial), E2, E3, and E4 in the voltage versus time plot in the following figure. The slope of the lines provides the scan rate that is used in the sweep.

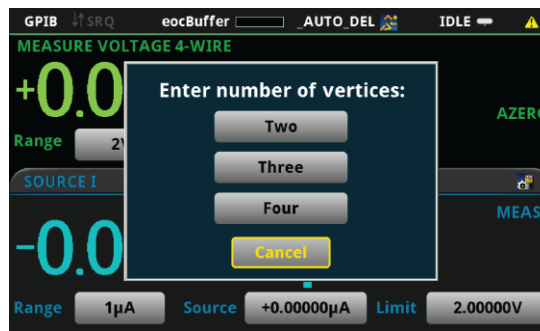
Figure 9: Potential sweep versus time of cyclic voltammetry example



Voltage vertices

You are prompted to enter the number of voltage vertices to be defined: **Two**, **Three**, or **Four** (see the following figure). For two vertices, only E1 and E2 are defined. For three vertices, E1, E2, and E3 must be defined. For four vertices, E1, E2, E3, and E4 must be defined.

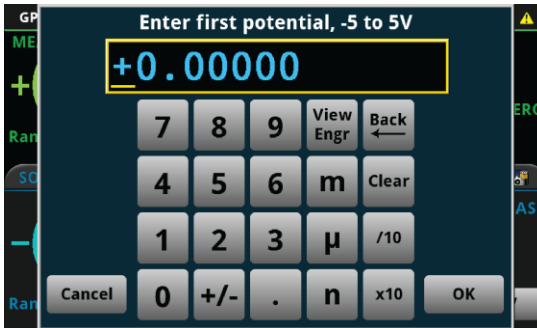
Figure 10: Enter number of vertices



Voltage magnitude

After the voltage vertices are defined, you must specify the magnitude of each vertex in the range of $\pm 5V$.

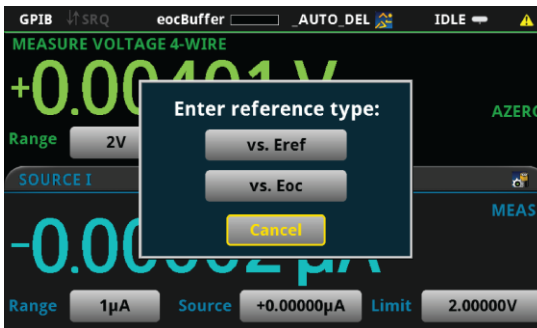
Figure 11: Enter potential of vertex



Reference voltage

After each magnitude is defined, you must select the reference type: Applied potential versus the reference potential (vs. Eref) or applied potential versus open circuit potential (vs. Eoc). If you select **vs. Eoc**, the acquired open circuit voltage is added to the voltage output. If you select **vs. Eref**, the specified voltage output is used as the applied potential.

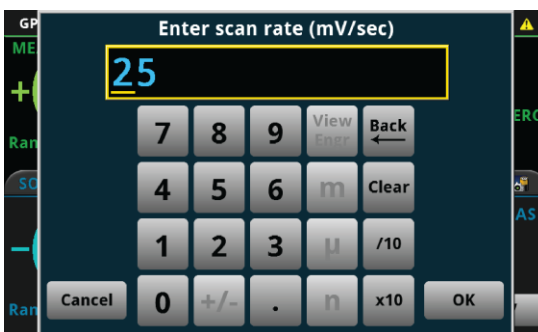
Figure 12: Enter reference type for each potential



Step 8: Select the scan rate

After sweep parameters have been defined, you are prompted to enter the scan rate in units of millivolts per second (see the following figure). The scan rate, which is defined as the change of the potential as a function of time ($\Delta V/\Delta t$), specifies the rate at which the potential is linearly swept during the experiment. You can select a scan rate of 1 mV per second to 3500 mV per second.

Figure 13: Enter scan rate (mV per second)



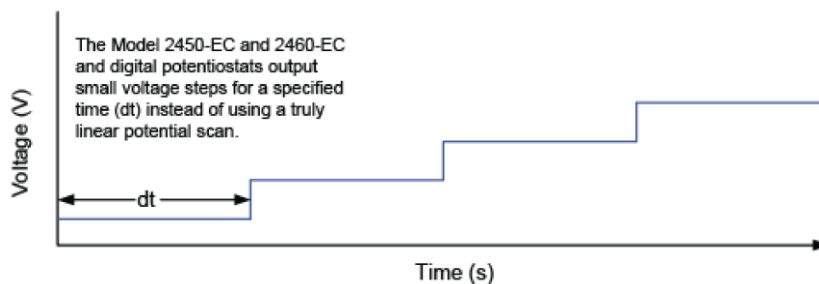
The Models 2450 and 2460 do not output a truly linear voltage; they output very small, digitized steps from 0.1 mV to 10 mV, depending upon the scan rate. While this will not cause significant errors in normal geometry cells, such as 10 mL analytic and most galvanic cells, there might be errors when using micro and ultramicro electrodes. The voltage step sizes used are calculated during scanning based on the user set scan rate:

- 100 μV (1 mV per second \leq scan rate < 35 mV per second)
- 1 mV (35 mV per second \leq scan rate < 350 mV per second)
- 10 mV (350 mV per second \leq scan rate \leq 3500 mV per second)

Analog integration current method

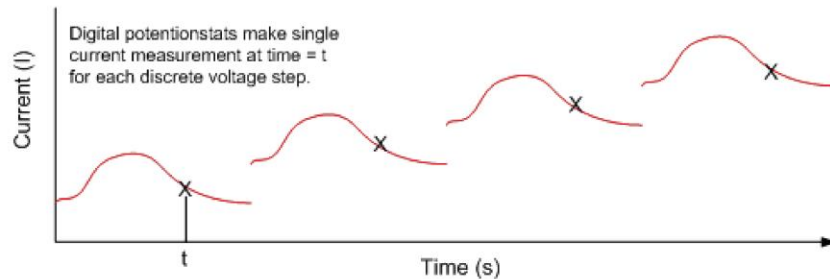
The 2450-EC and 2460-EC SourceMeters as well as other digital potentiostats generate a potential scan using very small discrete voltage steps. See the below figure.

Figure 14: Potential scan



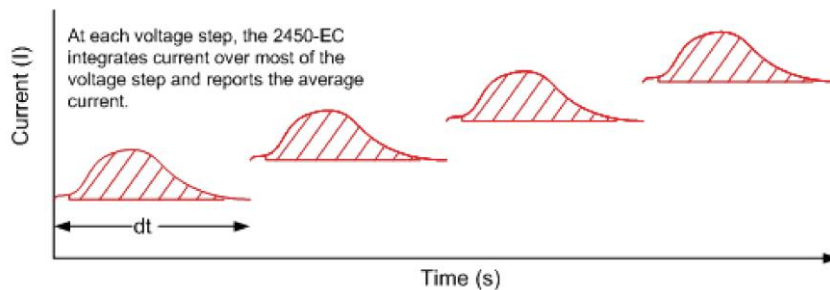
For the 2450-EC and 2460-EC, the voltage step size depends on the scan rate and can vary from 100 μV to 10 mV. To measure the resulting current at each step, many digital potentiostats make a measurement at a fixed point during the voltage step which may not be necessarily at the same time for each step. See the following figure.

Figure 15: Voltage step measurements



Conversely, the 2450-EC and 2460-EC instruments use an internal analog integrator to integrate current over most of the voltage step and report the average current over the aperture time (dt) of the voltage step. See the following figure. This average current is used in the generation of the voltammogram.

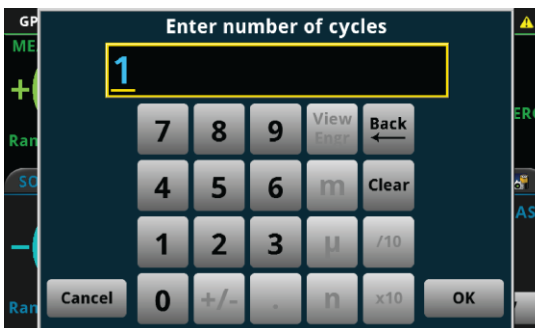
Figure 16: Current integration



Step 9: Select the number of scan cycles

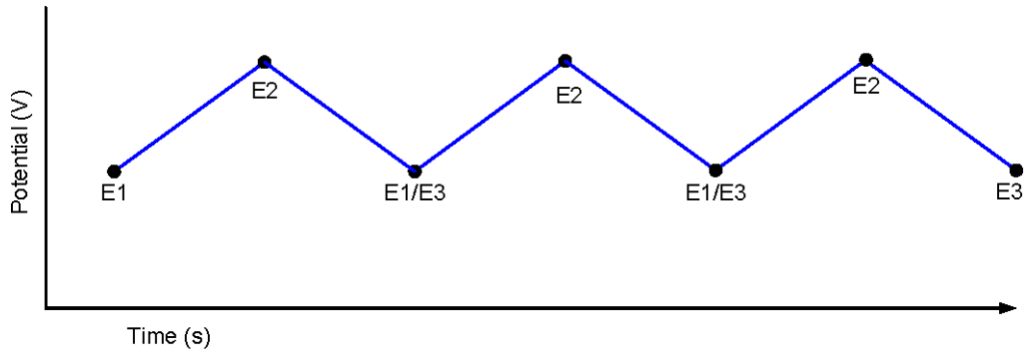
After entering the scan rate, you are prompted to enter the number of cycles (see the following figure). The number of cycles determines how many times each scan will be repeated. The number of cycles can be set from 1 to 100.

Figure 17: Enter number of cycles



The following figure shows an example of a potential versus time graph showing three cycles of a three-vertex voltage scan.

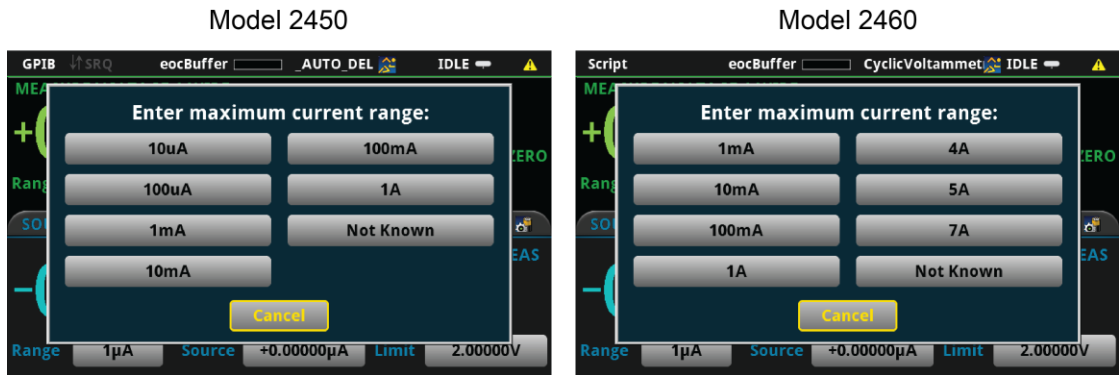
Figure 18: Example potential versus time graph



Step 10: Select the current measurement range

After selecting the number of scan cycles, you are prompted to select the maximum current range to measure the current from the scan. The available current ranges vary depending on if you are using a Model 2450 or a Model 2460. These ranges are shown in the following figure.

Figure 19: Enter maximum current range

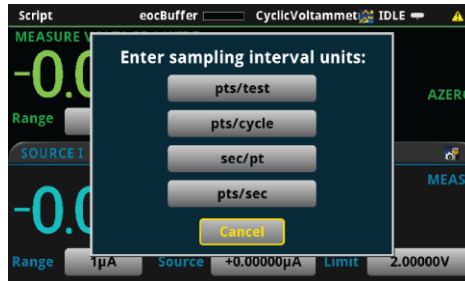


Step 11: Select the sampling interval units

During the scan, many small voltage steps are output, depending on the scan rate. To limit the number of points returned to the active buffer (current, voltage, and time readings), choose how often to make measurements by selecting one of the sampling interval units shown in the user prompt in the following figure. These sampling interval units are specified in terms of which units of measure should be used to store the sweep data in the active buffer of the instrument, `cvBuffer`.

The units and applicable range of values are defined in the table that follows the figure.

Figure 20: Enter sampling interval units

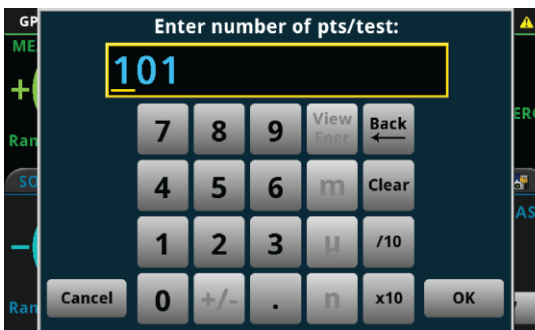


Range of values for sampling interval units

| Sampling interval unit | Description | Range of values |
|------------------------|--|-----------------|
| pts/test | The total number of points acquired during a test, regardless of how many cycles | 10 to 10,000 |
| pts/cycle | The total number of points acquired in each cycle | 10 to 10,000 |
| sec/pt | The number of seconds per each point | 0.01 to 100 |
| pts/sec | The total number of points taken per second | 0.01 to 100 |

Based on the sampling interval unit you select, you are then prompted to enter a magnitude for the interval selected. The following figure shows the user prompt to enter the number of pts/test.

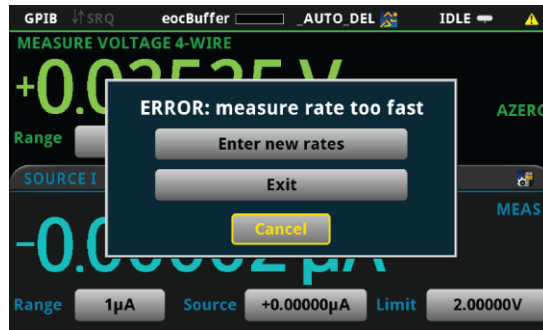
Figure 21: User prompt: Enter the number of points per test



NOTE

If you input test parameters that would cause the measurements to be taken at a faster rate than the instrument is sourcing, you will get the error message shown below, prompting you to enter a new scan rate and sampling interval unit.

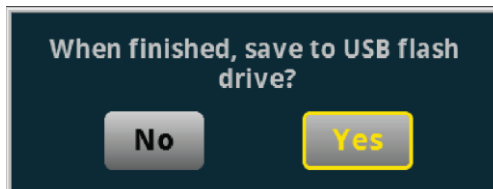
Figure 22: Measure rate too fast



Step 12: Save the data to the USB flash drive on the front panel

After the sample interval units have been selected, you are prompted to save the readings from the test to the front-panel USB flash drive. In addition to the current, voltage, and relative timestamp measurements saved during the scan, the test parameters are also saved to the USB flash drive.

Figure 23: Save the data to the USB flash drive



If you choose to save the data, you are prompted to enter a file name, as shown in the following figure. The data will be saved as a .CSV file. Once the file name is entered, select **OK**.

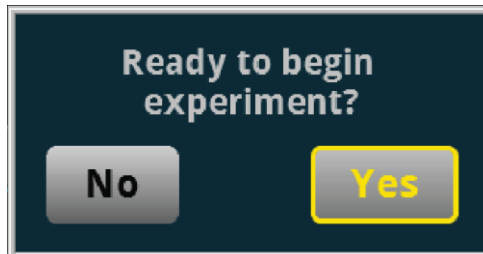
Figure 24: Enter the file name



Step 13: Start the experiment

After the prompts to save your measurements, you are prompted to begin the experiment. If you want to begin, select **Yes**; the scan will start immediately. If you want to end the program, select **No**.

Figure 25: Ready to begin experiment?



At any time during the scan, you can press the **OUTPUT ON/OFF** switch on the 2450 or 2460 front panel to stop the test.

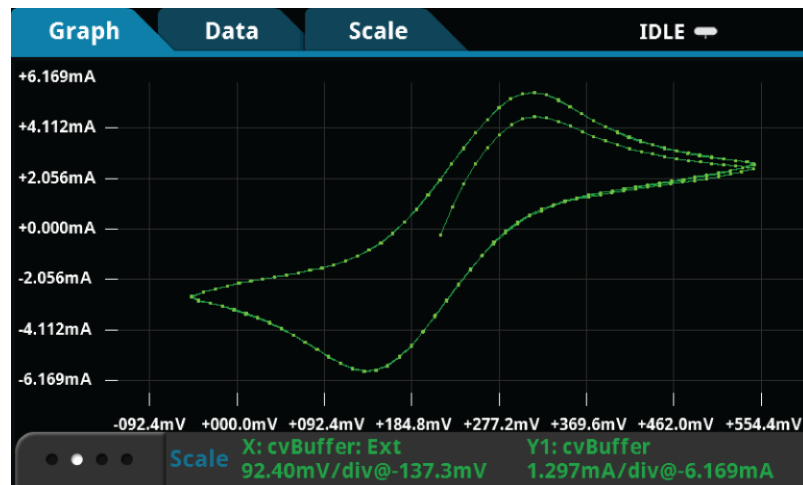
Step 14: View the graph

Upon execution of the scan, the display automatically shows the current measurements plotting in real time on the Graph screen.

To view the voltage instead of the time on the x-axis:

1. Select the **Data** tab on the Graph screen.
2. Select the button next to Graph Type and change the data to **Scatter IV**. The graph will automatically update to show the voltage on the x-axis, as shown in the following figure.

Figure 26: Voltammogram generated by a 2450 from an Fe(CN) experiment



3. To view the numeric data during the scan, press the **HOME** key to view both the measured current and the source voltage.

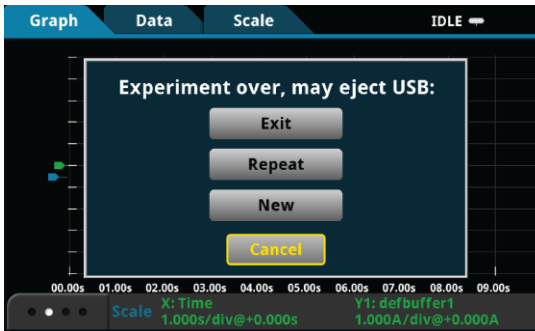
NOTE

When the scan is finished, you can save a screen capture of the graph to a USB drive inserted into the USB port of the SourceMeter by simultaneously pressing the HOME key and the ENTER key.

Step 15: Finish the experiment and select the next action

Once the experiment is completed, you will be prompted to select what the next action is: Exit, Repeat, or New. If you select **Exit**, the test ends. If you select **Repeat**, the test is repeated using the input parameters that you previously selected. If you saved data, you are prompted to enter a new file name for the repeated test. If you select **New**, the test script begins again and new test parameters can be input.

Figure 27: Experiment over, select the next action



Summary of user inputs

The following table summarizes the user prompts for the test script.

| Prompt message | User inputs and range of values | Notes |
|--|--|--|
| Ready to measure Eoc? | Yes or No | When you select Yes , the instrument sources 0 A and measures the voltage drop (Eoc) across the cell. |
| Measured Eoc is ____ (value) _____ | OK or Cancel | The dialog box displays the measured open circuit voltage. If the Eoc value is acceptable, press OK to continue the test. If it is not acceptable, select Cancel to exit the test. |
| Enter number of vertices: | Two, Three, or Four | The specified vertices are the two to four potentials used in the C-V scan. |
| Enter first potential (-5 to 5V) | Enter the initial potential from -5V to 5V and select OK | This is the first point of the C-V sweep, or E1. |
| Enter reference type: | vs. Eref or vs. Eoc | vs. Eoc sets the "zero value" which is referenced to the previously measured value for Eoc; vs. Eref uses the setup's reference electrode. |
| Enter second, third, fourth potential (-5 to 5V) | Enter the potentials from -5 V to 5 V and select OK | Repeated for each of up to four vertices, depending on what is specified in the previous menus. |
| Enter reference type: | vs. Eref or vs. Eoc | Repeated for each of up to four vertices, depending on what is specified in previous menus. |
| Enter scan rate (mV/sec) | Enter the scan rate from 1 mV per second to 1000 mV per second and select OK | The scan rate defines the rate at which the voltage sweeps between the vertices. |
| Enter number of cycles | Enter the number of cycles of the scan to be repeated (an integer from 1 to 100) | This is the number of times to repeat the scan. |
| Enter maximum current range: | For the Model 2450: 1 mA, 10 mA, 100 mA, 1 A, 4 A, 5 A, or 7 A ranges For the Model 2460: 10 µA, 100 µA, 1 mA, 10 mA, 100 mA, or 1 A ranges | The test script detects which instrument is being used and provides appropriate current ranges for each. |
| Enter sampling interval units (measurement rate) | pts/test (10 to 10,000) | Total number of points in the test. |
| | pts/cycle (10 to 10,000) | Number of points in each cycle. |
| | sec/pt (.01 to 100) | Number of seconds per each point. |
| | pts/sec (.01 to 100) | Number of points taken per second. |

| Prompt message | User inputs and range of values | Notes |
|-----------------------------------|--|--|
| Enter number of __ (units) __: | Enter the magnitude of the sampling interval that you will be using and select OK | Based on the sampling interval units selected, you are prompted for the magnitude of the interval. |
| When finished, save to USB drive? | Yes or No | You choose whether to save the data. |
| Enter filename: | Enter the string that will be used and select OK | The string entered here will be used to name the file in the format _____.CSV. |
| Ready to begin experiment? | Yes or No | Select Yes to run the test or No to exit the program. |

After the experiment is finished, you are prompted again:

| Prompt message | User inputs and range of values | Notes |
|---------------------------------|---------------------------------|---|
| Experiment over, may eject USB: | Exit, Repeat, or New | Select Exit to exit the program. To repeat the same experiment, select Repeat . Select New to return to the beginning of the script and enter new test parameters. |

NOTE

Select **Cancel** on any of the input displays to exit the program.

Step 16: View the results saved to the USB flash drive

If you choose to save the data to the USB flash drive, the readings will be saved in a .CSV file after the test is finished.

To view the readings, open the file in a spreadsheet program on a computer. The figure below is an example of how the data appears (only a few of the readings from the test are shown). Current, voltage, time, and general parameters for the test are included in the file.

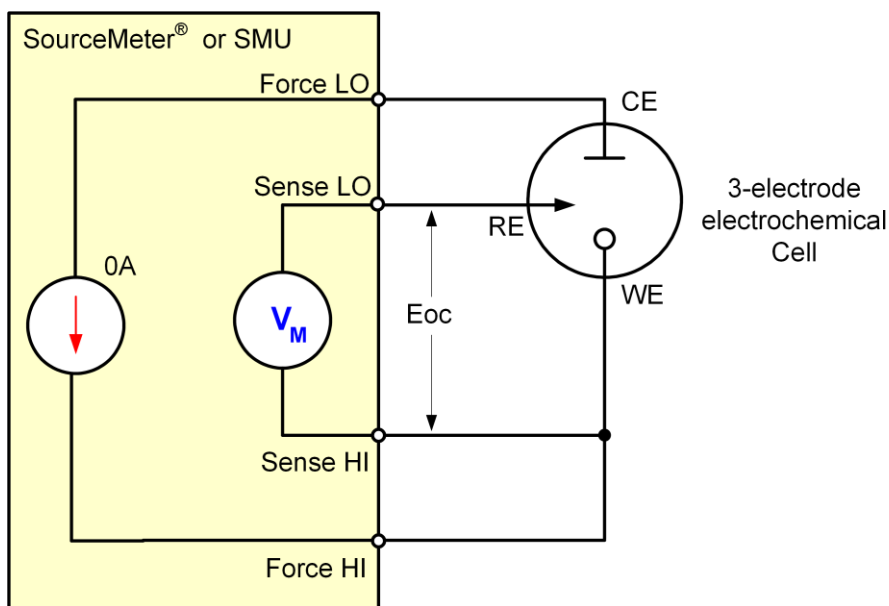
Figure 28: Example data as it appears in a .csv file

| | | |
|-----------------------|--------------|----------|
| General Parameters: | | |
| EOC potential = | 0.215446 | V |
| fileName = | fecn23test7 | |
| Source Parameters: | | |
| Source Range = | 2 | |
| # of Vertices = | 4 | V |
| Vertex 1 = | 0.215446 | V |
| Vertex 2 = | 0.55 | V |
| Vertex 3 = | -0.05 | V |
| Vertex 4 = | 0.215446 | V |
| Source Rate = | 25 | mV/sec |
| # of Cycles = | 3 | |
| Measure Parameters: | | |
| Current Range = | 0.1 | A |
| Sampling Interval = | 2 | pts/sec |
| nplc | 0.18 | |
| Calculated Parameters | | |
| stepSize = | 0.0001 | V |
| sourceDelay = | 0.004 | seconds |
| Voltage | Current | Seconds |
| 0.21545 | -0.000263035 | 0 |
| 0.22795 | 0.00089106 | 0.479199 |
| 0.24045 | 0.00181773 | 0.979192 |
| 0.25295 | 0.00260912 | 1.47921 |
| 0.26545 | 0.00328355 | 1.97919 |
| 0.27795 | 0.00383069 | 2.47919 |
| 0.29045 | 0.00423351 | 2.9792 |
| 0.30295 | 0.00447714 | 3.47921 |
| 0.31545 | 0.00456108 | 3.97919 |
| 0.32795 | 0.00450899 | 4.47918 |

Open circuit potential

The open circuit potential (OPC) of an electrochemical cell is a voltage measurement made between the reference and working electrodes. When the open circuit potential is measured, a voltmeter with high impedance is used to measure the voltage with no current or voltage applied to the cell. Because of their high input impedance, the 2450 and 2460 can be used to make OPC measurements when configured in a 4-wire configuration as shown in the figure below. In this setup, the instrument is configured to measure voltage and source 0 A. If the OPC is measured prior to performing cyclic voltammetry, there is no need to manually change any test leads between measurements because the instrument can automatically change functions internally.

Figure 29: Measuring the open circuit potential



Device connections

Connections are made from the 2450 or 2460 to the electrochemical cell using the Electrochemistry Translation Cable Accessory Kit cable as described in the Electrochemistry Translation Cable Accessory Kit Quick Start Guide.

Test procedure overview

The following list outlines the basic steps to perform potential measurements and save the data to a USB flash drive using the Model 2450 or 2460. Details of each step are described more thoroughly in the following sections.

1. Select the TSP command set on the Model 2450 or 2460.
2. Before making physical connections to the cell, set the output of the 2450 or 2460 to the high-impedance output-off state.
3. Prepare the analyte and assemble the electrochemical cell.
4. Connect the 2450 or 2460 output terminals to the electrochemical cell.
5. Execute the test script.
6. Select and adjust each settings group in the Main Menu:
 - a. Set the potential range.
 - b. Set the number of samples.
 - c. Set the measurement interval.
7. After you are finished changing the settings, choose whether to save results.
8. Start the experiment and view the measurement results.
9. View the data on the graph.
10. After the experiment completes, choose whether to view experiment statistics, repeat the experiment, change settings, or exit.
11. View the results saved to the USB flash drive.

Test script parameters

The open circuit potential test script (OpenCircuitPotential.tsp) prompts the user to adjust certain test parameters as listed below:

Potential Range:

- **2450:** 0.02 V, 0.2 V, 2 V, 20 V
- **2460:** 0.2 V, 2 V, 7 V, 10 V, 20 V

Number of Samples: $1 \leq n \leq 100,000$

Measure Interval: $0.75s \leq \text{measurement interval} \leq 100 s$

Step 1: Select the TSP command set

To configure the Model 2450 or 2460 to use the TSP command set:

1. Press the **MENU** key.
2. Under System, select **Settings**.
3. Select the button next to Command Set and select **TSP**.
4. When prompted to reboot, select **Yes**.

Step 2: Place the output in the high-impedance output-off state

Before making physical connections from the instrument to the cell, set the output of the instrument to the high-impedance output-off state. When the high impedance output-off state is selected, the output relay opens, disconnecting the instrument from the load.

To set the output of the Model 2450 or 2460 to the high-impedance output-off state:

1. Press the **MENU** key.
2. Under Source, select **Settings**.
3. Select the button next to Output Off State and select **High Impedance**.
4. Press the **HOME** key to return to the home screen.

Step 3: Prepare the analyte and assemble the electrochemical cell

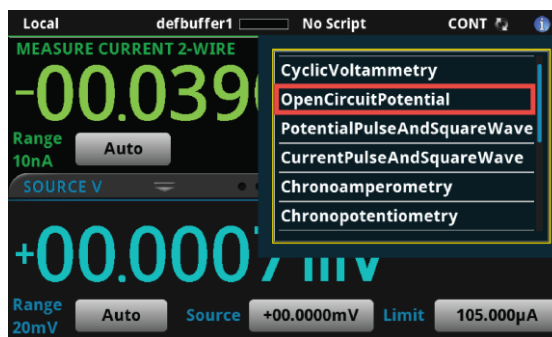
Step 4: Connect the four output terminals of the instrument to the electrochemical cell

Specific information on making connections from the instrument to the device is described in the Model 2450-EC / 2460-EC Electrochemistry Lab System Quick Start Guide.

Step 5: Execute the open circuit potential test script

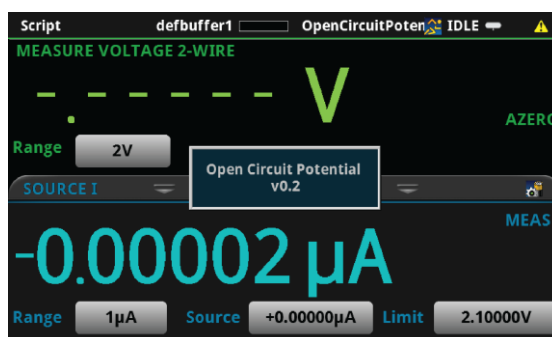
1. At the top of the home screen, touch the active script indicator. A list of available scripts is displayed.

Figure 30: Selecting the OpenCircuitPotential test script



2. Select OpenCircuitPotential. The script begins to execute immediately and the following screen will be displayed for one second.

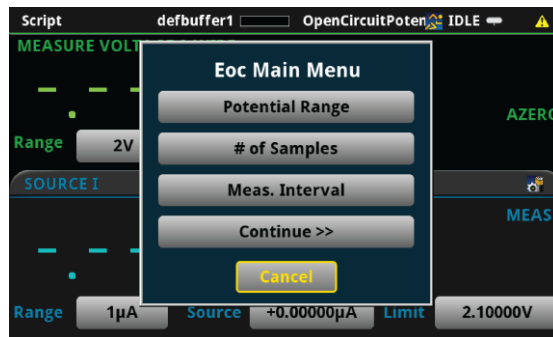
Figure 31: Start of Open Circuit Potential test script



Step 6: Select a settings group to adjust from the Main Menu

After the test begins, the parameters of the open circuit must be defined from the Main Menu. These parameters are the potential range, the number of samples, and the measure interval time. Once these are defined, select Continue>> to proceed with the test.

Figure 32: Open Circuit Potential Main Menu

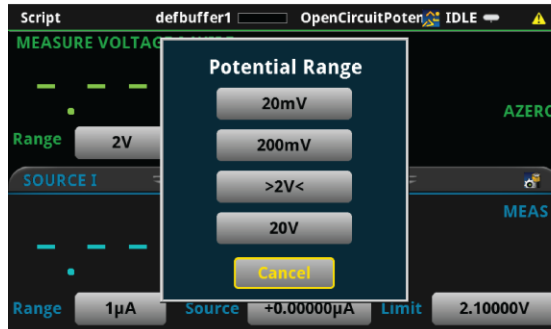


Potential range

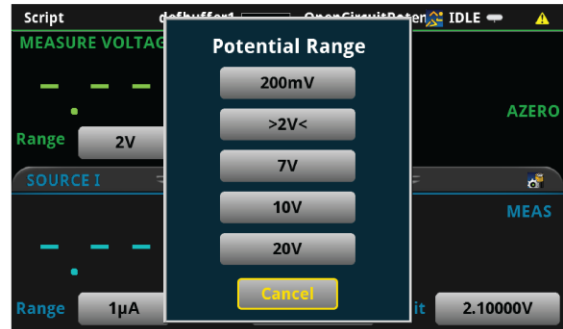
When you select Potential Range, you are prompted to enter the voltage measurement range. The available voltage ranges vary depending on whether you are using a Model 2450 or a Model 2460. These ranges are shown in the following figure.

Figure 33: Maximum potential measurement range

Model 2450



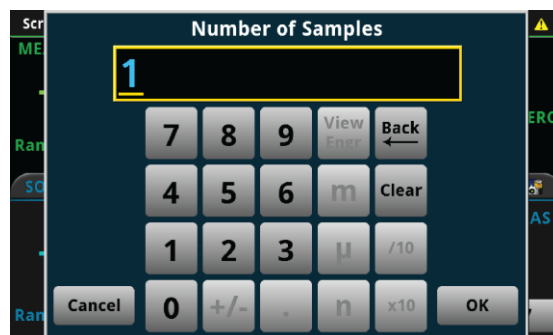
Model 2460



Number of samples

After you select the potential range, you must specify the number of open circuit potential measurements to make. The number of measurements can range from 1 to 100,000.

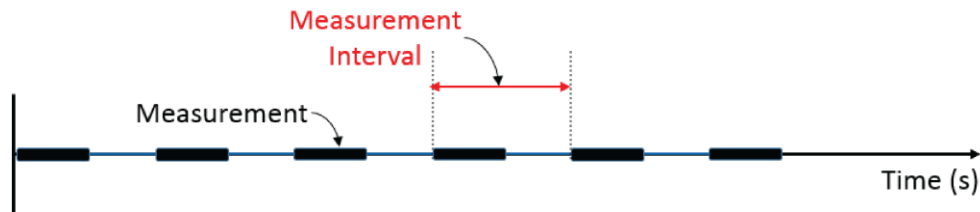
Figure 34: Number of samples



Measurement interval

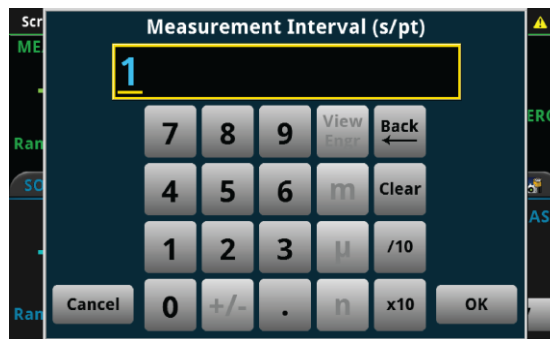
After selecting the number of measurements to acquire, choose the measurement interval time in seconds/point. The measurement interval time is described graphically in the following figure.

Figure 35: Measurement interval time



You must specify a measurement interval time between 0.75 and 100 s/pt.

Figure 36: Measurement interval

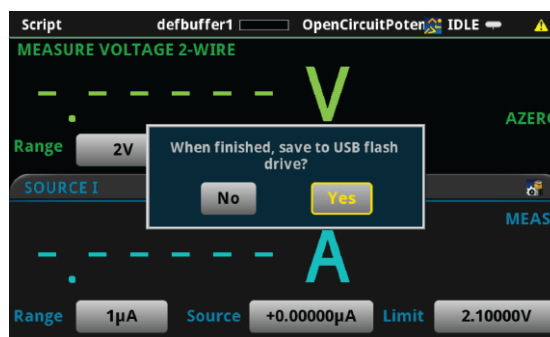


After the test parameters are defined, you can select Continue>> to proceed with the test script.

Step 7: Save the data to the USB flash drive on the front panel

After defining the test parameters, you are prompted to save the readings from the test to the front-panel USB drive. In addition to the current, voltage, and relative timestamps acquired during the test, the user input parameters and statistics (min, max, standard deviation, etc.) about the data are also saved to the USB drive.

Figure 37: Save the data to the USB drive

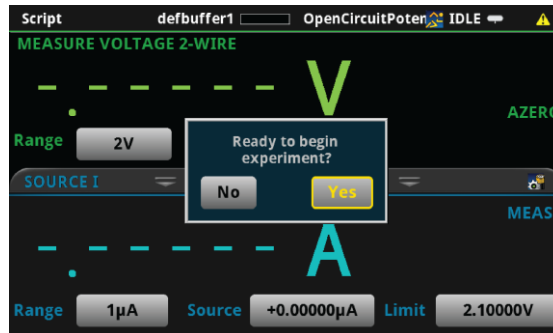


If you choose to save the data, a `.CSV` file will be created with a name containing the date and time of creation. If there are more than 10,000 measurements stored, the process of saving the data may take several minutes.

Step 8: Start the experiment

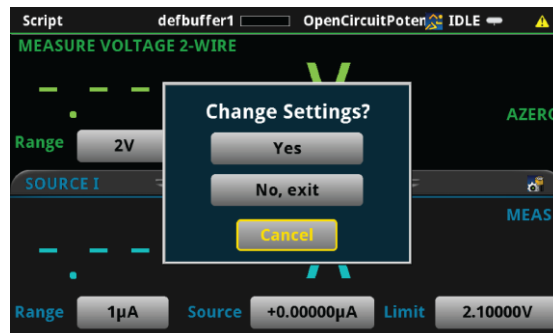
After the prompt to save your measurements, you are prompted to begin the experiment. If you want to begin, select **Yes**; the test will start immediately.

Figure 38: Start the experiment dialog



If you select **No**, you will have the option to exit or change the settings. If you select **Yes**, the program will take you back to the Main Menu. If you select **No, exit**, the program will terminate and your settings will be lost.

Figure 39: Change settings dialog



Once the test begins, the User swipe screen will appear where you will be able to see the progress of the experiment as show in the following figure.

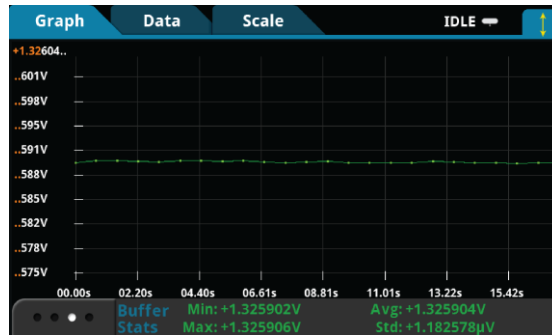
Figure 40: Measuring Eoc



Step 9: View the graph

To view the data graphically in real time, press the MENU key and then Graph. An x-y plot of the measured potential as a function of time will be displayed as shown below.

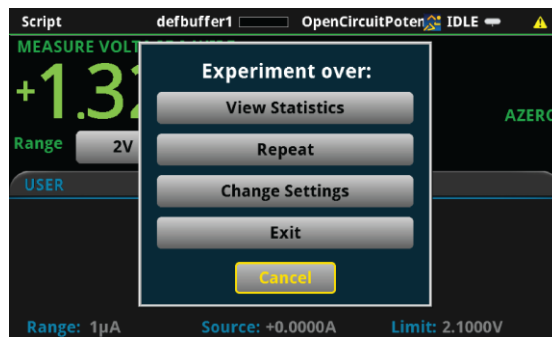
Figure 41: Voltage versus time graph



Step 10: View the data

After the experiment is finished, you will be prompted whether to **View Statistics** for the test data, **Repeat** the experiment using the same settings, **Change Settings** in the Main Menu, or **Exit** the program.

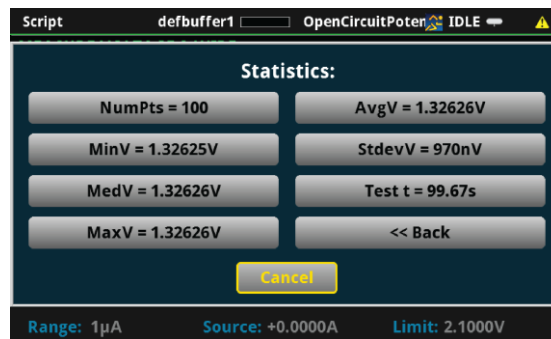
Figure 42: Experiment over dialog



View Statistics

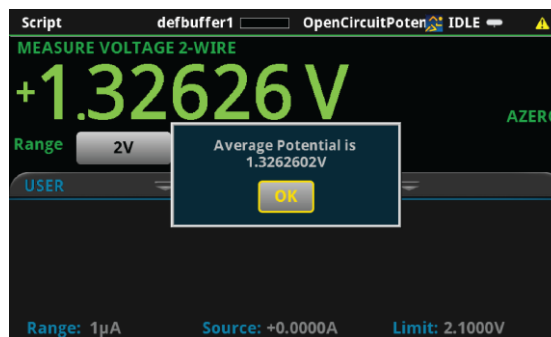
If you select **View Statistics**, you will see information about data as shown in the figure below. This includes the total number of points in the test (**NumPts**), the minimum measured potential (**MinV**), the median measured potential (**MedV**), the maximum measured potential (**MaxV**), the average measured potential (**AvgV**), and the standard deviation of the measured potential (**StdevV**), and the duration of the test (**Test t**).

Figure 43: Viewing statistics



You can select any one of the statistics to see a higher resolution view of the number displayed, or you can select **<<Back** or **Cancel** to go back to the Experiment Over menu.

Figure 44: Experiment over dialog



Repeat

If you select Repeat, the test will begin again using the same settings as originally set. You will be prompted to save the data.

Change Settings

If you select Change Settings, you will return to the Main Menu to start the test again.

Exit

You will be asked if you want to exit the program.

Step 11: View the results saved to the USB drive

If you choose to save the data to the USB flash drive, the readings will be saved in a `.csv` file after the test is finished.

To view the readings, open the file in a spreadsheet program on a computer. The figure below is an example of how the data appears (only a few readings from the test are shown). Current, potential, relative timestamps and general parameters and statistics for the test are included in the file.

Figure 45: Example data as it appears in a `.csv` file

| Keithley Instruments: Open Circuit Potential | | |
|--|------------------------------------|--------------|
| System Parameters | | |
| Date/Time | Tue Nov 10 10:20:54 2015 | |
| Model | 2460 | |
| Serial Number | 4054493 | |
| Firmware | 1.3.0s | |
| Program Version | v0.2 | |
| | | |
| Sampling | 1 s/pt | |
| Potential | 2 V | |
| | | |
| Quick Stats | | |
| 100 points recorded | 1.325838 V average | |
| 1.325832 V minimum | 2.43E-06 V standard deviation | |
| 1.325838 V median | 99.67774 seconds to run experiment | |
| 1.325842 V maximum | | |
| | | |
| Raw Data | | |
| Time(s) | Current(A) | Potential(V) |
| 0 | -3.03E-09 | 1.325838 |
| 0.982387 | -4.79E-11 | 1.325841 |
| 1.982435 | -4.74E-11 | 1.325842 |
| 2.982485 | -4.96E-11 | 1.325842 |
| 3.982534 | -4.89E-11 | 1.325842 |
| 4.98258 | -4.77E-11 | 1.325841 |
| 5.982636 | -4.88E-11 | 1.325842 |
| 6.98269 | -4.83E-11 | 1.32584 |
| 7.982749 | -4.98E-11 | 1.32584 |
| 8.982796 | -4.86E-11 | 1.32584 |

Potential Pulse and Square Wave

In the Potential Pulse and Square Wave application, the 2450 or 2460 supplies a series of up to 100,000 potential pulses. At the end of each pulse, the instrument will measure the resulting current. The user can select both the peak and base levels of the pulses as well as the period, pulse width, and sample time.

Device connections

Connections are made from the instrument to the electrochemical cell using the Electrochemistry Translation Cable Accessory Kit cable as described in the Electrochemistry Translation Cable Accessory Kit Quick Start Guide.

Test procedure overview

The following list outlines the basic steps to perform Potential Pulse and Square Wave measurements and save the data to a USB flash drive using the Model 2450 or 2460. Details of each step are described more thoroughly in the following sections.

1. Select the TSP command set on the Model 2450 or 2460.
2. Before making physical connections to the cell, set the output of the 2450 or 2460 to the high-impedance output-off state.
3. Prepare the analyte and assemble the electrochemical cell.
4. Connect the instrument output terminals to the electrochemical cell.
5. Execute the test script.
6. Select and adjust each settings group in the Main Menu:
 - a. Set the pulse level.
 - b. Set the current range.
 - c. Set the timing.
 - d. Set the end conditions.
7. After you are finished changing the settings, choose whether to save results.
8. Start the experiment and view the measurement results.
9. View the data on the graph.
10. After the experiment, choose whether to view experiment statistics, repeat the experiment, change settings, or exit.
11. View the results saved to the USB flash drive.

Test script parameters

The Potential Pulse and Square Wave test script (PotentialPulseAndSquareWave.tsp) prompts the user to adjust certain test parameters as listed below:

Pulse Levels

Peak Potential:

- $V_{\text{peak}} \leq \pm 20 \text{ V}$

Base Potential:

- $V_{\text{base}} \leq \pm 20 \text{ V}$

Current Range:

- 2450: 10 nA, 100 nA, 1 μA , 10 μA , 100 μA , 1 mA, 10 mA, 100 mA, 1 A
- 2460: 1 μA , 10 μA , 100 μA , 1 mA, 10 mA, 100 mA, 1 A, 4 A, 5 A, 7 A

Timing

Pulse Period:

2450

- $I_{\text{range}} = 1 \mu\text{A}$: $200 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$
- $I_{\text{range}} \geq 10 \mu\text{A}$: $4 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$

2460

- $I_{\text{range}} \leq 100 \mu\text{A}$: $80 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$
- $I_{\text{range}} \geq 1 \text{ mA}$: $10 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$

Pulse Width:

2450

- $I_{\text{range}} = 1 \mu\text{A}$: $100 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$
- $I_{\text{range}} \geq 10 \mu\text{A}$: $2 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$

2460

- $I_{\text{range}} \leq 100 \mu\text{A}$: $40 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$
- $I_{\text{range}} \geq 1 \text{ mA}$: $5 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$

Sample Time:

- $0.01 \text{ PLC} \leq \text{sample time} \leq 10 \text{ PLC}$
- $\text{sample time} \leq (\text{pulse width} - 0.001)\text{s}$

End Conditions:

Number of Cycles: $1 \leq \text{number of cycles} \leq 100,000$

Program Time: $10 \text{ ms} \leq \text{program time} \leq 100,000 \times \text{pulse period}$

Step 1: Select the TSP command set

To configure the Model 2450 or 2460 to use the TSP command set:

1. Press the **MENU** key.
2. Under System, select **Settings**.
3. Select the button next to Command Set and select **TSP**.
4. When prompted to reboot, select **Yes**.

Step 2: Place the output in the high-impedance off-state

Before making physical connections from the instrument to the cell, set the output of the instrument to the high-impedance output-off state. When the high impedance output-off state is selected, the output relay opens, disconnecting the instrument from the load.

To set the output of the Model 2450 or 2460 to the high-impedance output-off state:

1. Press the **MENU** key.
2. Under Source, select **Settings**.
3. Select the button next to Output Off State and select **High Impedance**.
4. Press the **HOME** key to return to the home screen.

Step 3: Prepare the analyte and assemble the electrochemical cell

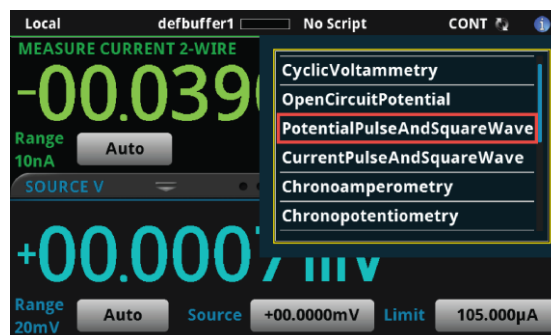
Step 4: Connect the four output terminals of the instrument to the electrochemical cell

Specific information on making connections from the instrument to the device is described in the Model 2450-EC / 2460-EC Electrochemistry Lab System Quick Start Guide.

Step 5: Execute the Potential Pulse and Square Wave test script

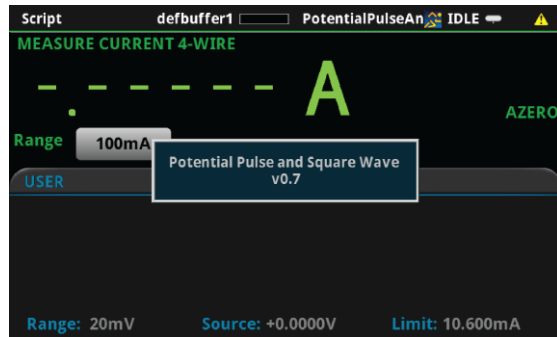
1. At the top of the home screen, touch the active script indicator. A list of available scripts is displayed.

Figure 46: Selecting the Potential Pulse and Square Wave test script



2. Select Potential Pulse and Square Wave. The script begins to execute immediately and the following screen will be displayed for one second.

Figure 47: Start of Potential Pulse and Square Wave test script

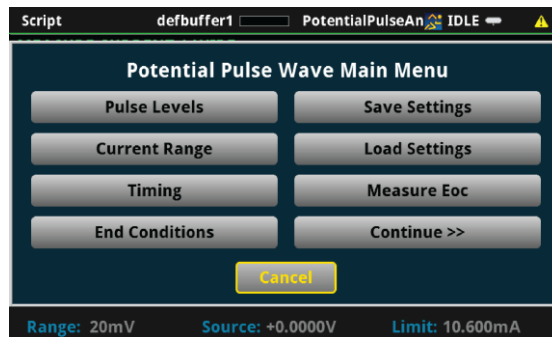


Step 6: Select and adjust each settings group in the Main Menu

After the program begins, the parameters of the Potential Pulse and Square Wave must be defined from the Main Menu. The four options on the left are families of settings that can be changed. **Pulse Levels** allows you to set the peak and base potentials of the pulse wave. **Current Range** allows you to change the range of the limiting current. **Timing** allows you to change the period and pulse width of the wave, as well as the sample time. Finally, **End Conditions** allows the user to select which parameters terminate testing.

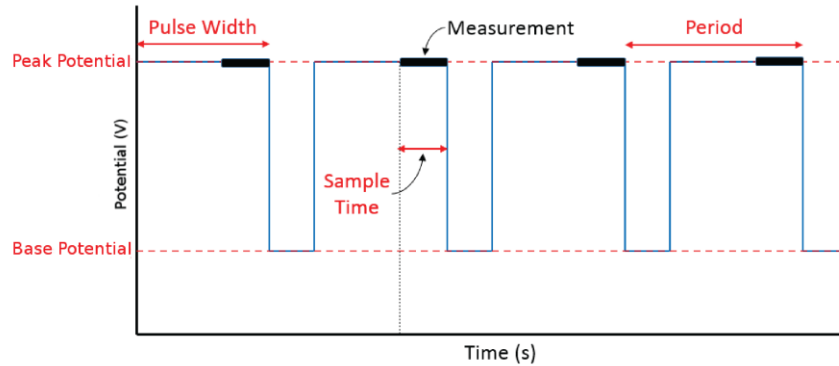
The option **Save Settings** allows you to save the present settings to a USB drive, while **Load Settings** allows you to load settings you have previously saved to a USB drive from this program. **Measure Eoc** enables you to measure the open-circuit potential. Press **Continue >>** when you have initialized all desired settings.

Figure 48: Main Menu



Various parameters of the pulse wave are displayed in the figure below. Though not pictured, there is also a short "grace period" between the end of the measurement and the end of the pulse to allow the instrument's analog-to-digital converter to finish.

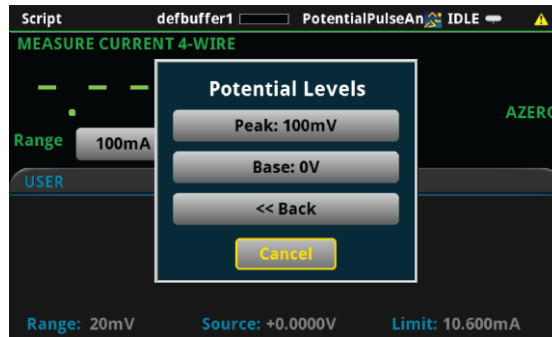
Figure 49: Potential versus Time



Pulse Levels

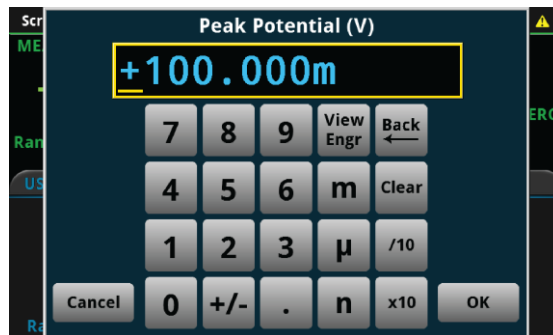
From the Main Menu, you can select **Pulse Levels** to bring up a menu that allows you to change the peak and base potentials in the pulse wave. You may also select **<< Back** to go back to the Main Menu or **Cancel** to exit the program.

Figure 50: Pulse Levels



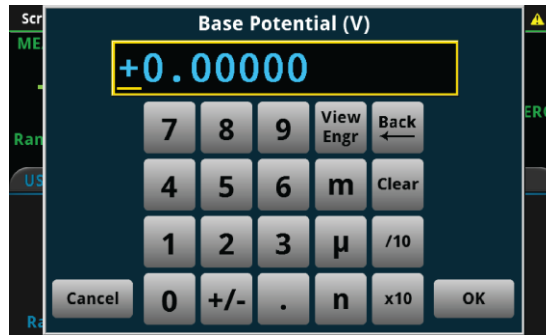
If you select the setting **Peak**, then you will have the option to change the peak potential. The peak potential is the maximum current the instrument will attempt to force, given that the produced current is within the selected current range.

Figure 51: Peak Potential



If you select the setting **Base**, then you will have the option to change the base potential.

Figure 52: Base Potential



Current Range

From the Main Menu, select **Current Range** to set the current range for the experiment. This range constrains the currents that the instrument is capable of sourcing through the device under test, so you should choose a range above what you expect the maximum current to be. The available current ranges for the 2450 and 2460 are shown in the following figure.

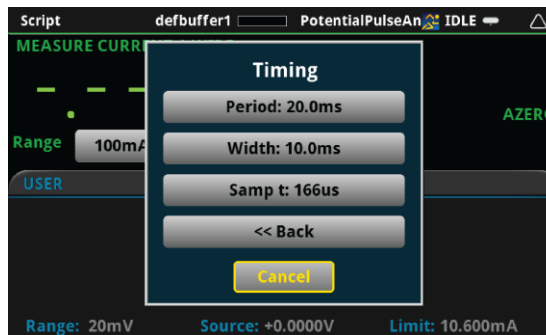
Figure 53: Maximum current measurement range



Timing Settings

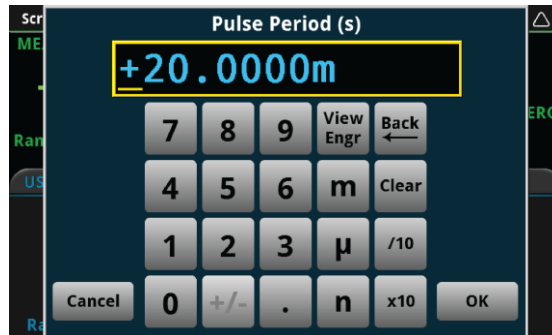
From the Main Menu, you can select **Timing** to bring up a menu that allows you to change the sample time, the pulse period, and the pulse width. You may also select **<< Back** to go back to the Main Menu or **Cancel** to exit the program.

Figure 54: Timing Menu



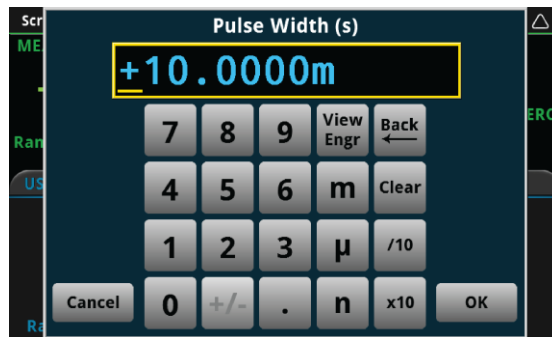
If you select the setting **Period**, then you will have the option to change the period of the pulse wave.

Figure 55: Pulse Period



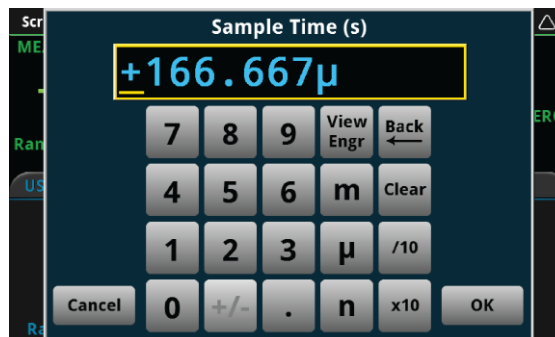
If you select the setting **Width**, then you will be able to change the pulse width of the wave.

Figure 56: Pulse Width



If you select the setting **Sample t** (short for sample time, this is the length of time over which the actual measurement will be taken), then you can change the sample time of the measurement. The measurement will be taken at the end of the pulse. There is a short "grace period" between the end of the measurement and the end of the pulse to allow the instrument's analog-to-digital converter to finish.

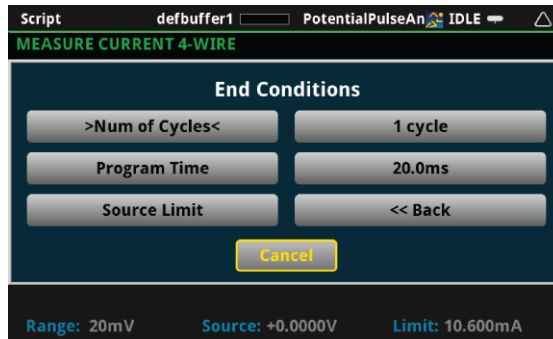
Figure 57: Sample Time



End Condition Settings

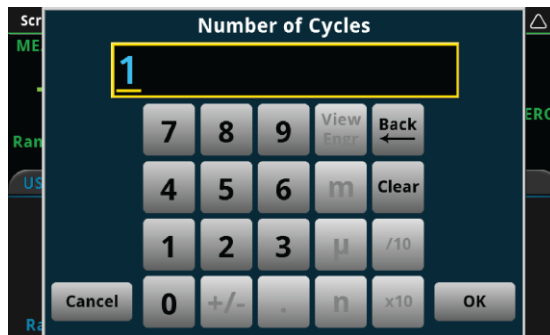
From the Main Menu, you can select **End Conditions** to bring up a menu that allows you to choose the conditions that will stop the pulse wave. **Num of Cycles** and **Program Time** are adjustable end conditions, so that you can choose how many cycles or how much time until the pulse wave terminates. Conversely, **Source Limit** is either on or off, and will terminate the program if the instrument attempts to exceed the source limit. Either **Num of Cycles** or **Program Time** must be selected, but not both. You may also select **<< Back** to go back to the Main Menu or **Cancel** to exit the program.

Figure 58: End Condition Menu



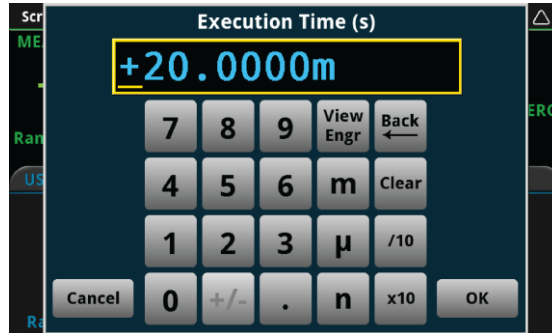
If you select the setting **Num of Cycles**, then you will be able to change the number of cycles of the pulse wave to be executed. The number of cycles can be between 1 and 100,000. Selecting this option will automatically enable the Number of Cycles end condition and disable the Program Time end condition.

Figure 59: Number of Cycles



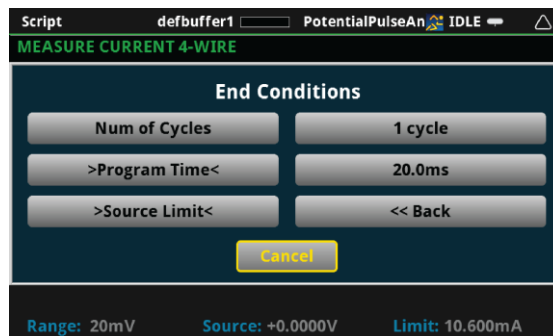
If you select the setting **Program Time**, then you can change the amount of time during which pulses will be generated. After the specified program time, the pulse wave will stop at the nearest half-cycle. Selecting this option will automatically enable the Program Time end condition and disable the Number of Cycles end condition.

Figure 60: Execution Time



If **Source Limit** is enabled and bracketed by "> <", that indicates that the condition is active and the pulse wave will be terminated if the measured current hits the source range limit.

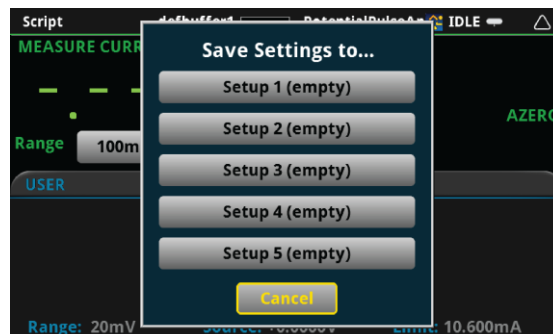
Figure 61: Source limit active



Save Settings

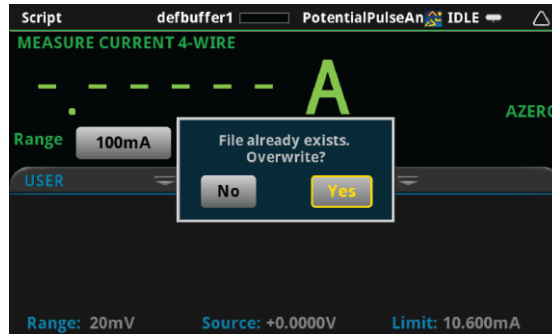
From the Main Menu, you can select **Save Settings** if you want to save the present settings to a USB drive for use or reference later. The settings will be saved in a .csv file. Many spreadsheet programs can open and read .csv files legibly. When a USB drive has been successfully recognized by the instrument, the **Save Settings** option will prompt you to choose from 1 of 5 setup files. All files labeled as **(empty)** have not yet been saved on your USB drive.

Figure 62: Save Settings



If you attempt to save the setup to a file that already exists on the USB drive—one that is not "empty"—the program will ask if you want to overwrite the existing file.

Figure 63: Overwrite



You will be notified if the save was successful.

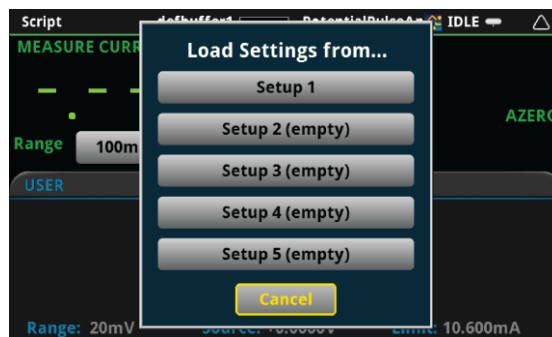
Figure 64: Save successful



Load Settings

From the Main Menu, you can select **Load Settings** if you want to load settings from a USB drive that have been previously saved via the Potential Pulse and Square Wave program. If the file for the settings has been altered, the program may not be able to load the settings. Numbers may be altered, but altering any text may cause an error while loading. When a USB drive has been successfully recognized by the instrument, the **Load Settings** option will prompt you to choose from one of five setups to load. Setups labeled as **(empty)** do not have setups associated with them, and will not be able to be loaded.

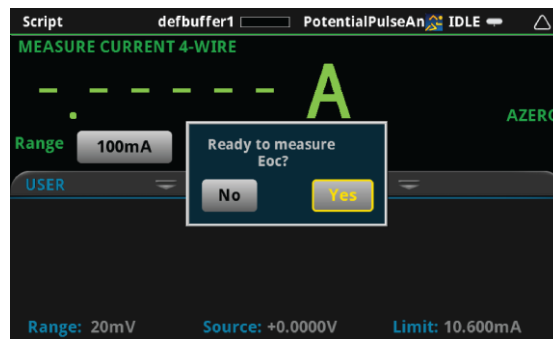
Figure 65: Load Settings



Measure open circuit potential

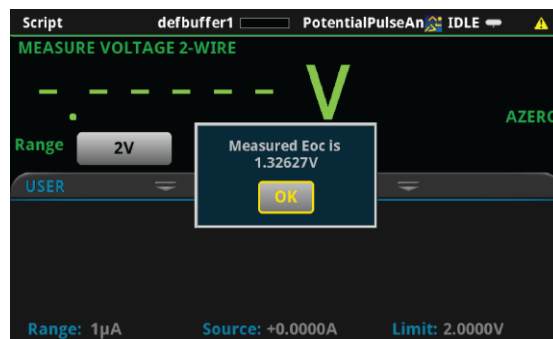
From the Main Menu, you can select **Measure Eoc** to measure the open-circuit potential of the device under test. The open-circuit potential is the potential across the terminals under a no-load condition. You will first be prompted whether you are ready to measure the open-circuit potential. Please ensure that the device under test is connected to the instrument in a proper 4-wire configuration before pressing **Yes**. You may also press **No** to return to the Main Menu.

Figure 66: Measure Eoc



A prompt will return to you the measured open circuit potential.

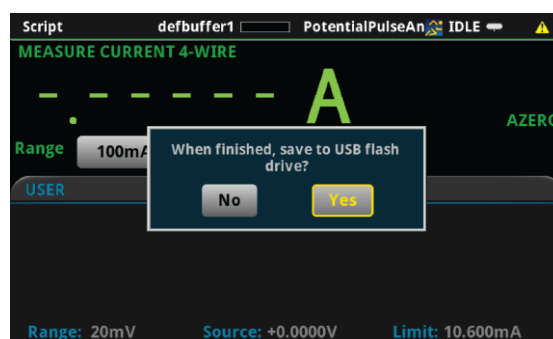
Figure 67: Measured Eoc



Step 7: Save the data to the USB drive on the front panel

After defining the test parameters, you may select **Continue >>** from the Main Menu to start the experiment. The program will first ask you if you want to save the results of the experiment.

Figure 68: Save Results

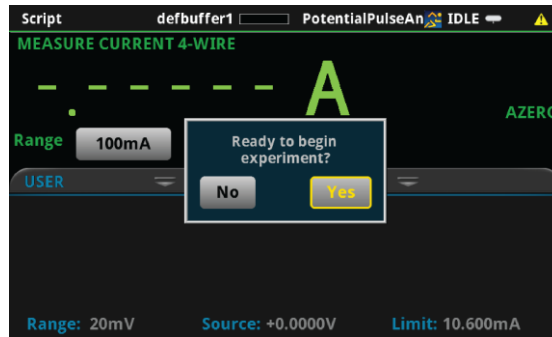


If you choose **Yes**, a `.CSV` file will be created with a name containing the date and time of its creation, and results will be saved to it at the end of the experiment. Note that if there are more than 10,000 measurements made, the process of saving may take several minutes.

Step 8: Start the experiment

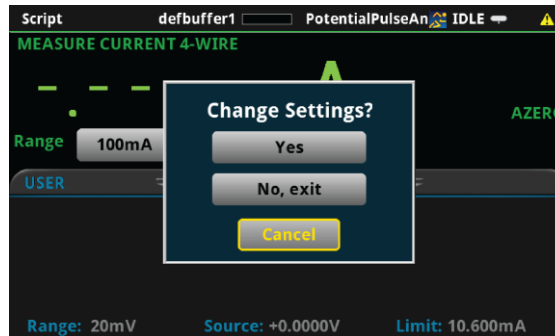
After the prompt to save your measurements, you are prompted to begin the experiment. If you want to begin, select **Yes**; the test will start immediately.

Figure 69: Start the Experiment



If you select **No** to the previous question, you will have the option to exit or change settings. If you select **Yes**, the program will take you back to the Main Menu. If you select **No, exit**, the program will terminate and any unsaved settings will be lost.

Figure 70: Change Settings Dialog



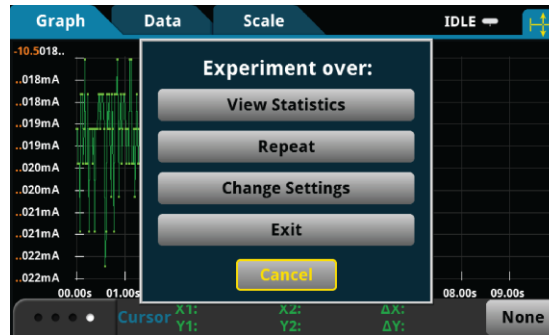
Step 9: View the graph

As the test begins an x-y plot of the measured current as a function of time will be displayed. After exiting the script, the graph may be viewed at any time by pressing the MENU key, then Graph.

Step 10: View the data

After the experiment is finished, you will be prompted whether to **View Statistics** for the test data, **Repeat** the experiment using the same settings, **Change Settings** in the Main Menu, or **Exit** the program.

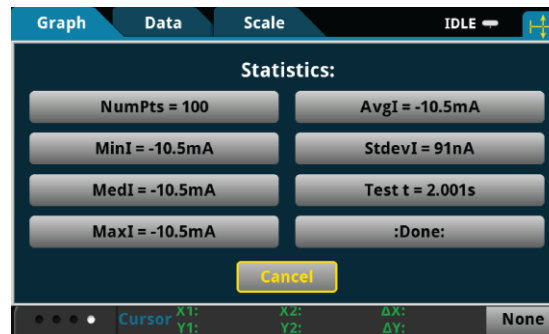
Figure 71: Experiment Over Dialog



View Statistics

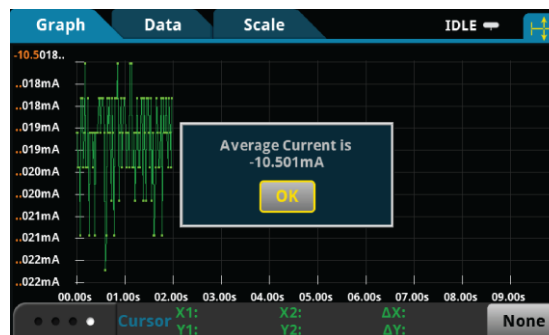
If you select **View Statistics**, you will see information about data as shown in the figure below. This includes the total number of points in the test (**NumPts**), the minimum measured current (**MinI**), the median measured current (**MedI**), the maximum measured current (**MaxI**), the average measured current (**AvgI**), and the standard deviation of the measured current (**StdevI**), and the duration of the test (**Test t**).

Figure 72: Viewing statistics



You may select any one of the statistics to view a higher resolution view of the number displayed, or you can select **<<Return** or **Cancel** to go back to the **Experiment over** menu. If you have set up more than one step you can select **Next Step>>** to view statistics for the next step.

Figure 73: Statistics



Repeat

If you select **Repeat**, the test will begin again using the same settings as originally set. You will be prompted to save the data.

Change Settings

If you select Change Settings, you will return to the Main Menu to start the test again.

Exit

You will be asked if you want to exit the program.

Step 11: View the results saved to the USB drive

If you choose to save the data to the USB flash drive, the readings will be saved in a `.csv` file after the test is finished.

To view the readings, open the file in a spreadsheet program on a computer. The figure below is an example of how the data appears (only a few readings from the test are shown). Measured current and general parameters and statistics for the test are included in the file.

Figure 74: Example data as it appears in a .csv file

| Keithley Instruments: Potential Pulse and Square Wave | | | |
|--|--------------------------|----------|--|
| System Parameters | | | |
| Date/Time | Tue Nov 10 14:54:54 2015 | | |
| Model | 2460 | | |
| Serial Number | 4054493 | | |
| Firmware | 1.3.0s | | |
| Program version | v1.0 | | |
| Waveform Parameters | | | |
| Peak Intensity | 5 V | | |
| Base Intensity | -1 V | | |
| Period | 0.04 s | | |
| Pulse Width | 0.015 s | | |
| Sampling Time | 0.000167 s | | |
| # of Cycles | 100 | | |
| Duration of Experiment | 4.00205 s | | |
| Cause of termination: Completed specified number of cycles | | | |
| Other Parameters | | | |
| Range | 0.1 A | | |
| Quick Stats | | | |
| | 100 points recorded | 0.104998 | |
| | 0.104995124 A minimum | 1.29E-06 | |
| | 0.104997933 A median | 4.002049 | |
| | 0.105000742 A maximum | | |
| Raw DataWave # | | | |
| | Potential | | |
| 1 | 0.104999 A | | |
| 2 | 0.104997 A | | |
| 3 | 0.104999 A | | |
| 4 | 0.104999 A | | |

Current Pulse and Square Wave

In the Current Pulse and Square Wave application the 2450 or 2460 supplies a series of up to 100,000 current pulses. At the end of each pulse the 2450 or 2460 will measure the resulting potential. The user can select both the peak and base levels of the pulses as well as the period, pulse width, and sample time.

Device connections

Connections are made from the instrument to the electrochemical cell using the Electrochemistry Translation Cable Accessory Kit cable as described in the Electrochemistry Translation Cable Accessory Kit Quick Start Guide.

Test procedure overview

The following list outlines the basic steps to perform Current Pulse and Square Wave measurements and save the data to a USB drive using the Model 2450 or 2460. Details of each step are described more thoroughly in the following sections.

1. Select the TSP command set on the Test Model 2450 or 2460.
2. Before making physical connections to the cell, set the output of the 2450 or 2460 to the high-impedance output-off state.
3. Prepare the analyte and assemble the electrochemical cell.
4. Connect the instrument output terminals to the electrochemical cell.
5. Execute the test script.
6. Select and adjust each settings group in the Main Menu:
 - a. Set the current levels.
 - b. Set the potential range.
 - c. Set the timing.
 - d. Set the end conditions.
7. After you are finished changing the settings, choose whether to save results.
8. Start the experiment and view the measurement results.
9. View the data on the graph.
10. After the experiment, choose whether to view experiment statistics, repeat the experiment, change settings, or exit.
11. View the results saved to the USB drive.

Test script parameters

The Current Pulse and Square Wave test script (CurrentPulseAndSquareWave.tsp) prompts the user to adjust certain test parameters as listed below:

Pulse Levels

Peak Current:

- 2450: $I_{\text{peak}} \leq \pm 1.05 \text{ A}$
- 2460: $I_{\text{peak}} \leq \pm 7.35 \text{ A}$

Base Current:

- 2450: $I_{\text{peak}} \leq \pm 1.05 \text{ A}$
- 2460: $I_{\text{peak}} \leq \pm 7.35 \text{ A}$

Potential Range:

- 2450: 20 mV, 200 mV, 2 V, 20 V
- 2460: 200 mV, 2 V, 7 V, 10 V, 20 V

Timing

Pulse Period:

2450

- $I_{\text{peak}} \leq 1.05 \mu\text{A}$: $200 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$
- $I_{\text{peak}} > 1.05 \mu\text{A}$: $4 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$

2460

- $I_{\text{peak}} \leq 105 \mu\text{A}$: $80 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$
- $I_{\text{peak}} > 105 \mu\text{A}$: $10 \text{ ms} \leq \text{pulse period} \leq 3600 \text{ s}$

Pulse Width:

2450

- $I_{\text{peak}} \leq 1.05 \mu\text{A}$: $100 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$
- $I_{\text{peak}} > 1.05 \mu\text{A}$: $2 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$

2460

- $I_{\text{peak}} \leq 105 \mu\text{A}$: $40 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$
- $I_{\text{peak}} > 105 \mu\text{A}$: $5 \text{ ms} \leq \text{pulse width} \leq 99 \% \text{ of the pulse period}$

Sample Time:

- $0.01 \text{ PLC} \leq \text{sample time} \leq 10 \text{ PLC}$
- $\text{sample time} \leq (\text{pulse width} - 0.001) \text{ s}$

End Conditions

Number of Cycles: $1 \leq \text{number of cycles} \leq 100,000$

Program Time: $10 \text{ ms} \leq \text{program time} \leq 100,000 \times \text{pulse period}$

Step 1: Select the TSP command set

To configure the Model 2450 or 2460 to use the TSP command set:

1. Press the **MENU** key.
2. Under System, select **Settings**.
3. Select the button next to Command Set and select **TSP**.
4. When prompted to reboot, select **Yes**.

Step 2: Place the output in the high-impedance output-off state

Before making physical connections from the instrument to the cell, set the output of the instrument to the high-impedance output-off state. When the high impedance output-off state is selected, the output relay opens, disconnecting the instrument from the load.

To set the output of the Model 2450 or 2460 to the high-impedance output-off state:

1. Press the **MENU** key.
2. Under Source, select **Settings**.
3. Select the button next to Output Off State and select **High Impedance**.
4. Press the **HOME** key to return to the home screen.

Step 3: Prepare the analyte and assemble the electrochemical cell

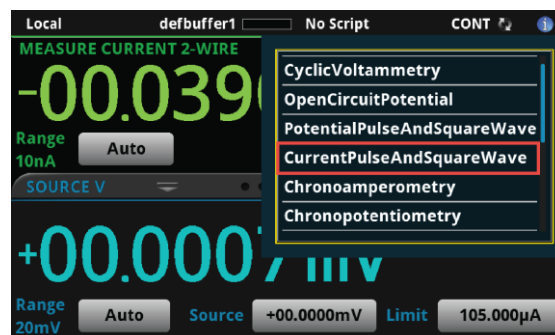
Step 4: Connect the four output terminals of the instrument to the electrochemical cell

Specific information on making connections from the instrument to the device is described in the Model 2450,60-EC Electrochemistry Lab System Quick Start Guide.

Step 5: Execute the Current Pulse and Square Wave test script

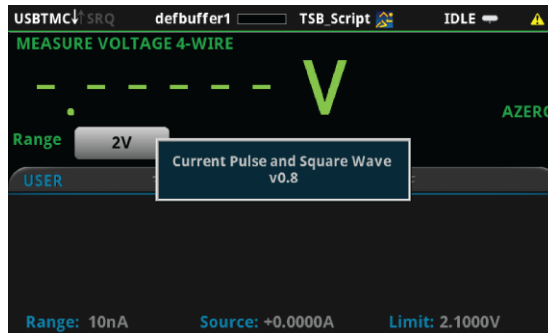
1. At the top of the home screen, touch the active script indicator. A list of available scripts is displayed.

Figure 75: Selecting the Current Pulse and Square Wave test script



2. Select Current Pulse and Square Wave. The script begins to execute immediately and the following screen will be displayed for one second.

Figure 76: Start of Current Pulse and Square Wave test script

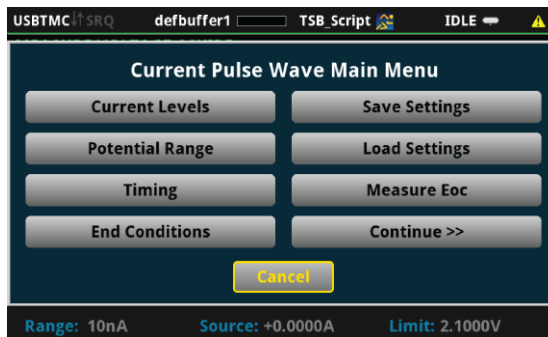


Step 6: Select and adjust each settings group in the Main Menu

After the program begins, the parameters of the Current Pulse and Square Wave must be defined from the Main Menu. The four options on the left are families of settings that can be changed. **Pulse Levels** allows you to set the peak and base currents of the pulse wave. **Potential Range** allows you to change the range of the limiting potential. **Timing** allows you to change the period and pulse width of the wave, as well as the sample time. Finally, **End Conditions** allows the user to select which parameters terminate testing.

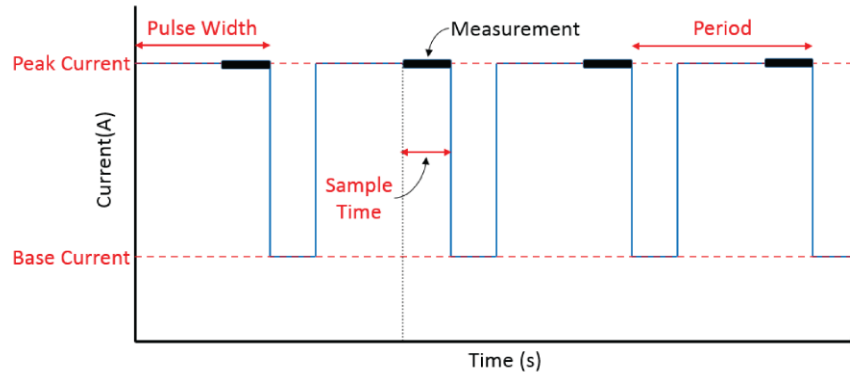
The option **Save Settings** allows you to save the present settings to a USB drive, while **Load Settings** allows you to load settings you have previously saved to a USB drive from this program. **Measure Eoc** enables you to measure the open-circuit potential. Press **Continue >>** when you have initialized all desired settings.

Figure 77: Main Menu



Various parameters of the pulse wave are displayed in the figure below. Though not pictured, there is also a short "grace period" between the end of the measurement and the end of the pulse to allow the instrument's analog-to-digital converter to finish.

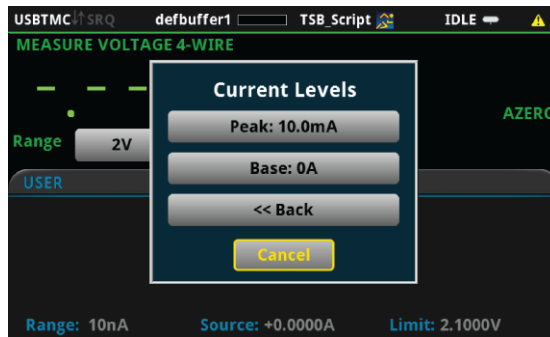
Figure 78: Current versus Time



Pulse Levels

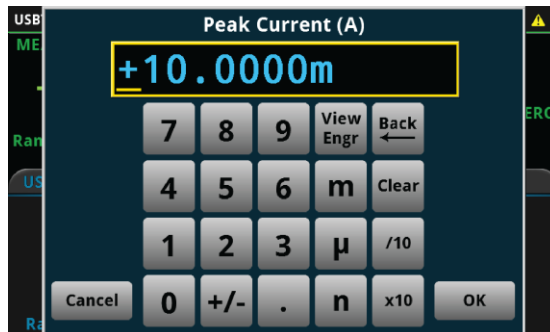
From the Main Menu, you can select **Pulse Levels** to bring up a menu that allows you to change the peak and base currents in the pulse wave. You may also select **<< Back** to go back to the Main Menu or **Cancel** to exit the program.

Figure 79: Pulse Levels



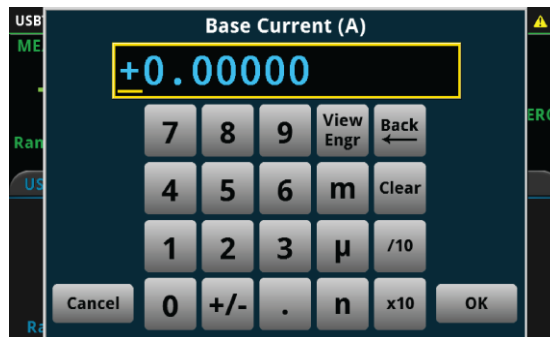
If you select the setting **Peak**, then you will have the option to change the peak current. The peak current is the maximum current the instrument will attempt to force, given that the produced potential is within the selected potential range.

Figure 80: Peak Current



If you select the setting Base, then you will have the option to change the base current.

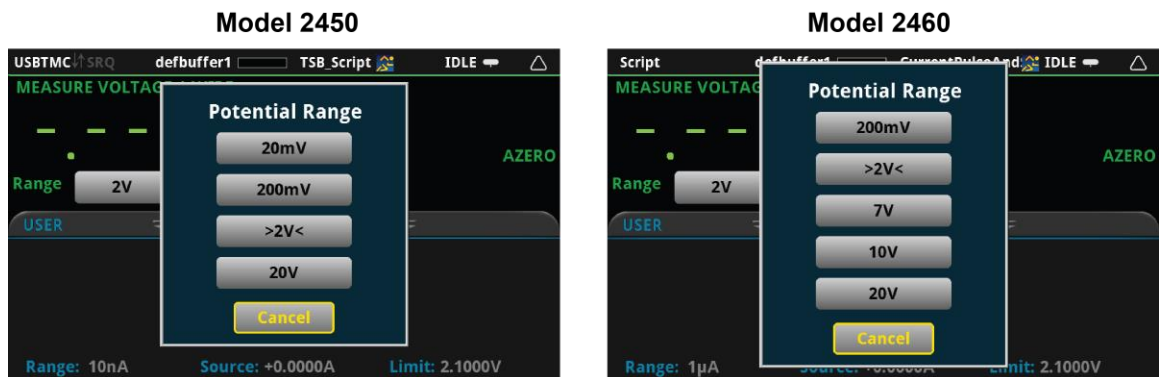
Figure 81: Base Current



Potential Range

From the Main Menu, select **Potential Range** to set the potential range for the experiment. This range constrains the potential that the instrument is capable of sourcing through the device under test, so you should choose a range above what you expect the maximum potential to be. The available potential ranges for the 2450 and 2460 are shown in the following figure.

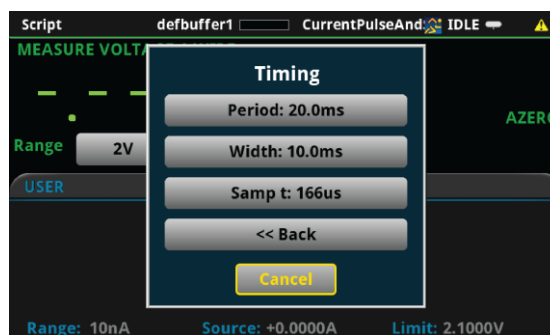
Figure 82: Potential Range



Timing Settings

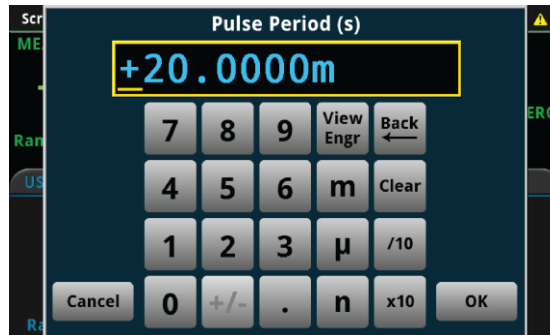
From the Main Menu, you can select **Timing** to bring up a menu that allows you to change the sample time, the pulse period, and the pulse width. You may also select **<< Back** to go back to the Main Menu or **Cancel** to exit the program.

Figure 83: Timing Menu



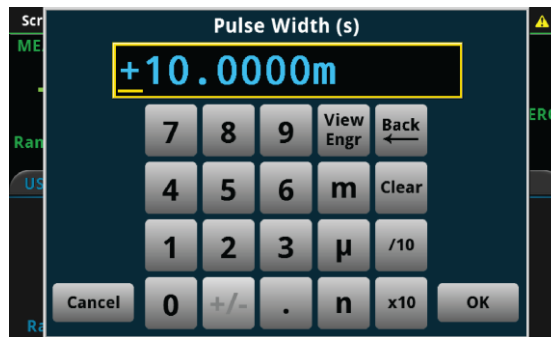
If you select the setting **Period**, then you will have the option to change the period of the pulse wave.

Figure 84: Pulse Period



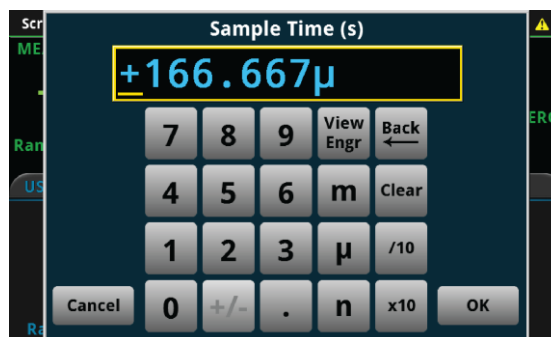
If you select the setting **Width**, then you will be able to change the pulse width of the wave.

Figure 85: Pulse Width



If you select the setting **Sample t** (short for sample time, this is the length of time over which the actual measurement will be taken), then you can change the sample time of the measurement (see the figure below). The measurement will be taken at the end of the pulse. There is a short "grace period" between the end of the measurement and the end of the pulse to allow the instrument's analog-to-digital converter to finish.

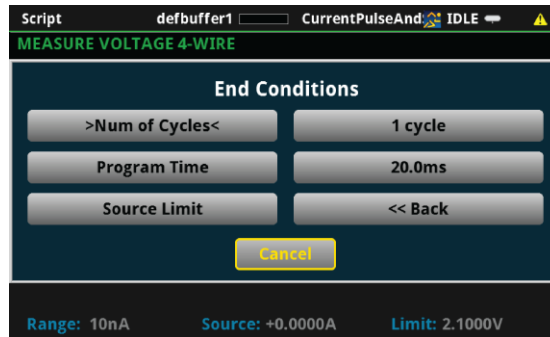
Figure 86: Sample Time



End Condition Settings

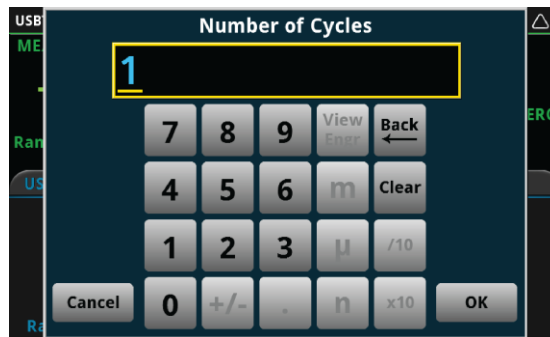
From the Main Menu, you can select **End Conditions** to bring up a menu that allows you to choose the conditions that will stop the pulse wave. **Num of Cycles** and **Program Time** are adjustable end conditions, so that you can choose how many cycles or how much time until the pulse wave terminates. Conversely, **Source Limit** is either on or off, and will terminate the program if the instrument attempts to exceed the source limit. Either **Num of Cycles** or **Program Time** must be selected, but not both. You may also select **<< Back** to go back to the Main Menu or **Cancel** to exit the program.

Figure 87: End Conditions Menu



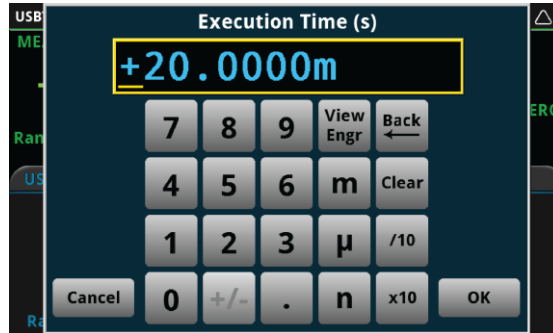
If you select the setting **Num of Cycles**, then you will be able to change the number of cycles of the pulse wave to be executed. The number of cycles can be between 1 and 100,000. Selecting this option will automatically enable the Number of Cycles end condition and disable the Program Time end condition.

Figure 88: Number of Cycles



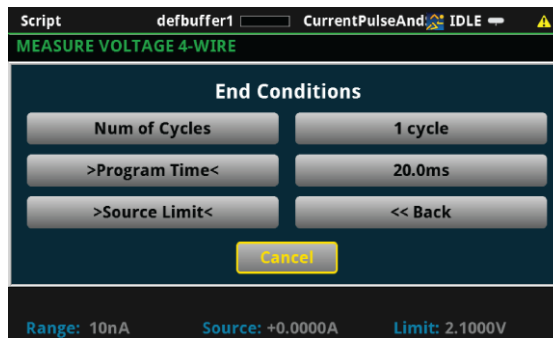
If you select the setting **Program Time**, then you can change the amount of time during which pulses will be generated. After the specified program time, the pulse wave will stop at the nearest half-cycle. Selecting this option will automatically enable the Program Time end condition and disable the Number of Cycles end condition.

Figure 89: Execution Time



If **Source Limit** is enabled and bracketed by "> <", that indicates that the condition is active and the pulse wave will be terminated if the measured current hits the source range limit.

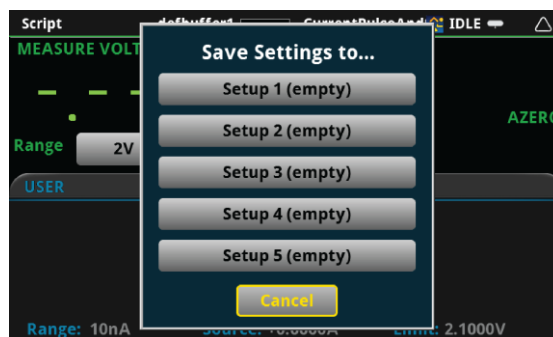
Figure 90: Source limit active



Save Settings

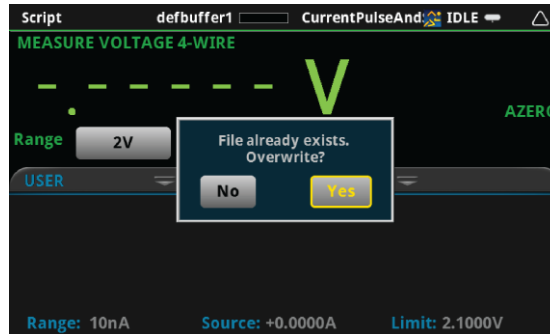
From the Main Menu, you can select **Save Settings** if you want to save the present settings to a USB drive for use or reference later. The settings will be saved in a .csv file. Many spreadsheet programs can open and read .csv files legibly. When a USB drive has been successfully recognized by the instrument, the **Save Settings** option will prompt you to choose from 1 of 5 setup files. All files labeled as **(empty)** have not yet been saved on your USB.

Figure 91: Save Settings



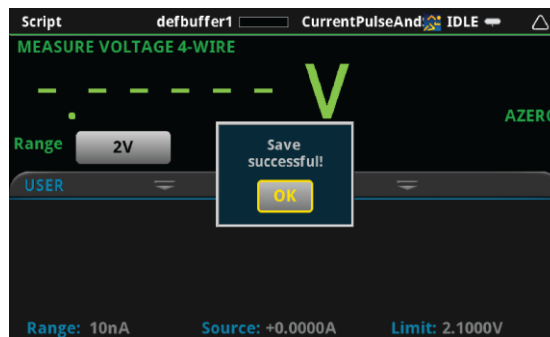
If you attempt to save the setup to a file that already exists on the USB drive—one that is not "empty"—the program will ask if you want to overwrite the existing file.

Figure 92: Overwrite?



You will be notified if the saving was successful.

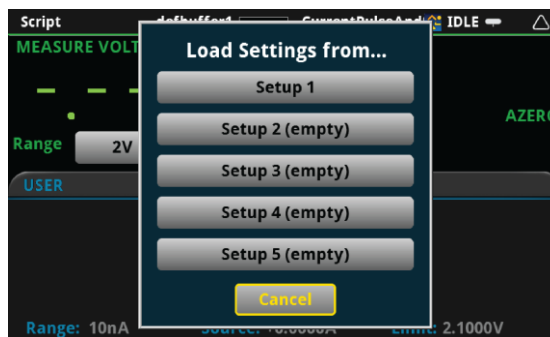
Figure 93: Save successful



Load Settings

From the Main Menu, you can select **Load Settings** if you want to load settings from a USB drive that have been previously saved via the Current Pulse and Square Wave program. If the file for the settings has been altered, the program may not be able to load the settings. Numbers may be altered, but altering any text may cause an error while loading. When a USB drive has been successfully recognized by the instrument, the **Load Settings** option will prompt you to choose from one of five setups to load. Setups labeled as **(empty)** do not have setups associated with them, and will not be able to be loaded.

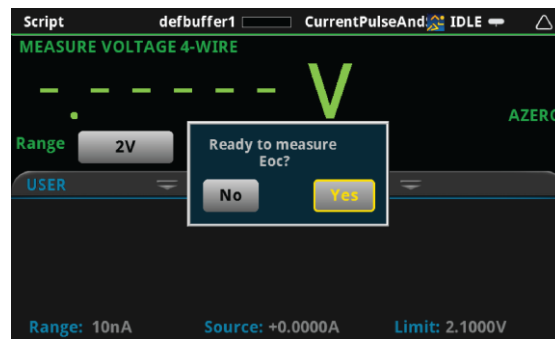
Figure 94: Load Settings



Measure open circuit potential

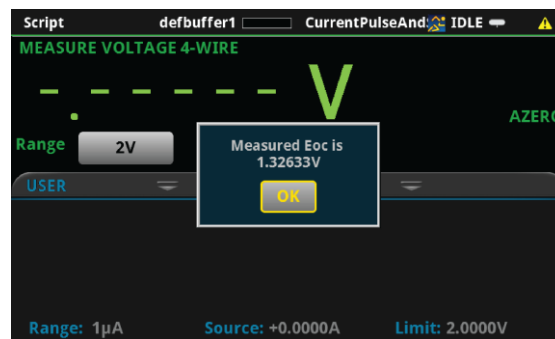
From the Main Menu, you can select **Measure Eoc** to measure the open-circuit potential of the device under test. The open-circuit potential is the potential across the terminals under a no-load condition. You will first be prompted whether you are ready to measure the open-circuit potential. Please ensure that the device under test is connected to the instrument in a proper 4-wire configuration before pressing **Yes**. You may also press **No** to return to the Main Menu.

Figure 95: Measure Eoc



A prompt will return to you the measured open circuit potential.

Figure 96: Measured Eoc



Step 7: Save the data to the USB drive on the front panel

After defining the test parameters, you may select Continue >> from the Main Menu to start the experiment. The program will first ask you if you want to save the results of the experiment.

Figure 97: Save Results

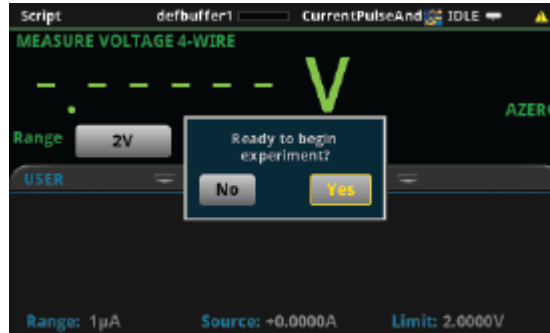


If you choose **Yes**, a `.csv` file will be created with a name containing the date and time of its creation, and results will be saved to it at the end of the experiment. Note that if there are more than 10,000 measurements made, the process of saving may take several minutes.

Step 8: Start the experiment

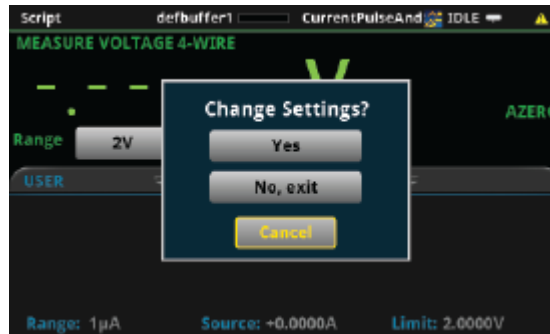
After the prompt to save your measurements, you are prompted to begin the experiment. If you want to begin, select **Yes**; the test will start immediately.

Figure 98: Start the Experiment



If you select **No** to the previous question, you will have the option to exit or change settings. If you select **Yes**, the program will take you back to the Main Menu. If you select **No, exit**, the program will terminate and any unsaved settings will be lost.

Figure 99: Change Settings Dialog



Step 9: View the graph

As the test begins an x-y plot of the measured current as a function of time will be displayed. After exiting the script, the graph may be viewed at any time by pressing the **MENU** key, then **Graph**.

Step 10: View the data

After the experiment is finished, you will be prompted whether to View Statistics for the test data, Repeat the experiment using the same settings, Change Settings in the Main Menu, or Exit the program.

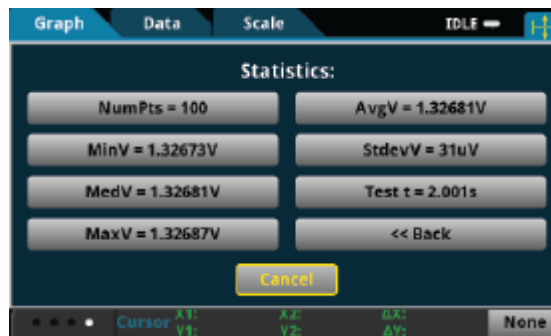
Figure 100: Experiment Over Dialog



View Statistics

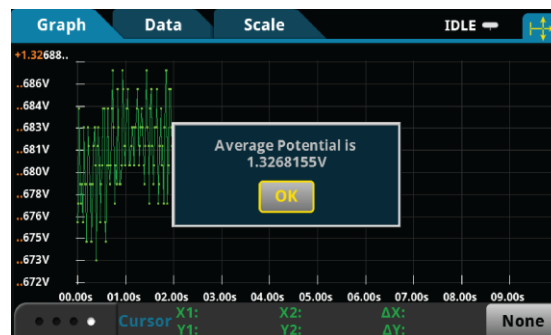
If you select **View Statistics**, you will see information about data as shown in the figure below. This includes the total number of points in the test (**NumPts**), the minimum measured potential (**MinV**), the median measured potential (**MedV**), the maximum measured potential (**MaxV**), the average measured potential (**AvgV**), and the standard deviation of the measured potential (**StdevV**), and the duration of the test (**Test t**).

Figure 101: Statistics



You may select any one of the statistics to view a higher resolution view of the number displayed, or you can select **<<Return** or **Cancel** to go back to the **Experiment over** menu. If you have set up more than one step you can select **Next Step>>** to view statistics for the next step.

Figure 102: Statistics Next Step



Repeat

If you select Repeat, the test will begin again using the same settings as originally set. You will be prompted to save the data.

Change Settings

If you select Change Settings, you will return to the Main Menu to start the test again.

Exit

You will be asked if you want to exit the program.

Step 11: View the results saved to the USB drive

If you choose to save the data to the USB drive, the readings will be saved in a .csv file after the test is finished.

To view the readings, open the file in a spreadsheet program on a computer. The figure below is an example of how the data appears (only a few readings from the test are shown). Measured potential and general parameters and statistics for the test are included in the file.

Figure 103: Example data as it appears in a .csv file

| Keithley Instruments: Current Pulse and Square Wave | | | |
|--|--------------------------|----------|----------------------|
| System Parameters | | | |
| Date/Time | Tue Nov 10 14:54:04 2015 | | |
| Model | 2460 | | |
| Serial Number | 4054493 | | |
| Firmware | 1.3.0s | | |
| Program version | v1.0 | | |
| Waveform Parameters | | | |
| Peak Intensity | 0.075 | A | |
| Base Intensity | 0 | A | |
| Period | 0.04 | s | |
| Pulse Width | 0.03 | s | |
| Sampling Time | 0.000167 | s | |
| # of Cycles | 100 | | |
| Duration of Experiment | 4.00208 | s | |
| Cause of termination: Completed specified number of cycles | | | |
| Other Parameters | | | |
| Range | 2 | V | |
| Quick Stats | | | |
| | 100 points recorded | 1.813023 | V average |
| | 1.812952519 V minimum | 3.46E-05 | V standard deviation |
| | 1.813035011 V median | 4.002079 | seconds to complete |
| | 1.813103914 V maximum | | |
| Raw Data | | | |
| Wave # | Potential | | |
| 1 | 1.81298 | V | |
| 2 | 1.81295 | V | |
| 3 | 1.81295 | V | |
| 4 | 1.81299 | V | |

Chronoamperometry

In Chronoamperometry the Model 2450 or 2460 steps the potential to a user-selected value where it is held constant for a specified period of time. While this potential is held, the instrument will measure the resulting current at a user-defined time intervals. The instrument can repeat this process for up to 8 defined steps.

Device connections

Connections are made from the Model 2450 or 2460 to the electrochemical cell using the Electrochemistry Translation Cable Accessory Kit cable as described in the Electrochemistry Translation Cable Accessory Kit Quick Start Guide.

Test procedure overview

The following list outlines the basic steps to perform chronoamperometry measurements and save the data to a USB flash drive using the Model 2450 or 2460. Details of each step are described more thoroughly in the following sections.

1. Select the TSP command set on the Model 2450 or 2460.
2. Before making physical connections to the cell, set the output of the 2450 or 2460 to the high-impedance output-off state.
3. Prepare the analyte and assemble the electrochemical cell.
4. Connect the instrument output terminals to the electrochemical cell.
5. Execute the test script.
6. Select and adjust each settings group in the Main Menu:
 - a. Set the step settings.
 - b. Set the current range.
 - c. Set the measurement setting.
7. After you are finished changing the settings, choose whether to save results.
8. Start the experiment and view the measurement results.
9. View the data on the graph.
10. After the experiment, choose whether to view experiment statistics, repeat the experiment, change settings, or exit.
11. View the results saved to the USB flash drive.

Test script parameters

The chronoamperometry test script (Chronoamperometry.tsp) prompts the user to adjust certain test parameters as listed below:

Step Potential: $V_{\text{step}} \leq \pm 20 \text{ V}$

Step Duration: $0.01 \text{ s} \leq \text{Time} \leq 99,999 \text{ s}$

Current Range:

- 2450: 10 nA, 100 nA, 1 μA , 10 μA , 100 μA , 1 mA, 10 mA, 100 mA, 1 A
- 2460: 1 μA , 10 μA , 100 μA , 1 mA, 10 mA, 100 mA, 1 A, 4 A, 5 A, 7 A

Number of Samples: $1 \leq n \leq 100,000$

Measure Interval: $0.01 \text{ s} \leq \text{measurement interval} \leq 100 \text{ s}$

Sample Time:

- $0.01 \text{ PLC} \leq \text{sample time} \leq 10 \text{ PLC}$
- $\text{sample time} \leq (\text{measurement interval} - 0.005)\text{s}$
- $\text{sample time} \leq (\text{step duration} - 0.005)\text{s}$

Step 1: Select the TSP command set

To configure the Model 2450 or 2460 to use the TSP command set:

1. Press the **MENU** key.
2. Under System, select **Settings**.
3. Select the button next to Command Set and select **TSP**.
4. When prompted to reboot, select **Yes**.

Step 2: Place the output in the high-impedance output-off state

Before making physical connections from the instrument to the cell, set the output of the instrument to the high-impedance output-off state. When the high impedance output-off state is selected, the output relay opens, disconnecting the instrument from the load.

To set the output of the Model 2450 or 2460 to the high-impedance output-off state:

1. Press the **MENU** key.
2. Under Source, select **Settings**.
3. Select the button next to Output Off State and select **High Impedance**.
4. Press the **HOME** key to return to the home screen.

Step 3: Prepare the analyte and assemble the electrochemical cell

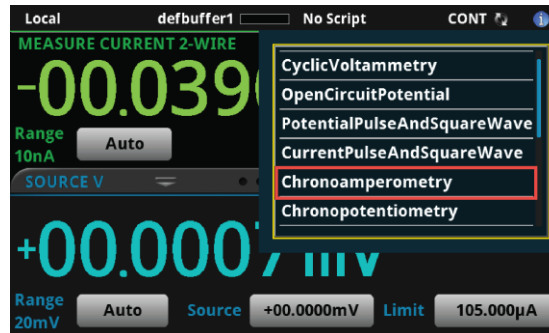
Step 4: Connect the four output terminals of the instrument to the electrochemical cell

Specific information on making connections from the instrument to the device is described in the Model 2450-EC / 2460-EC Electrochemistry Lab System Quick Start Guide.

Step 5: Execute the Chronoamperometry test script

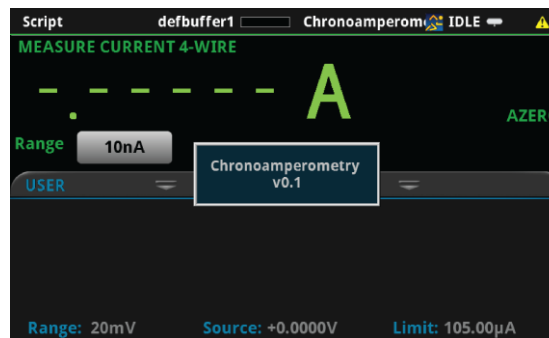
1. At the top of the home screen, touch the active script indicator. A list of available scripts is displayed.

Figure 104: Selecting the Chronoamperometry test script



2. Select Chronoamperometry. The script begins to execute immediately and the following screen will be displayed for one second.

Figure 105: Start of Open Circuit Potential test script

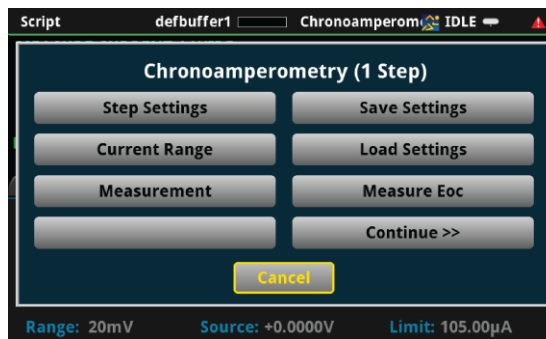


Step 6: Select and adjust each settings group in the Main Menu

After the program begins, the parameters must be defined from the Main Menu. The option **Step Settings** allows you to change the potential value and duration of the potential step, and to add up to 8 total steps in sequence. **Current Range** allows you to constrain the currents that the instrument is capable of sourcing through the device under test. **Measurement** allows you to set the measurement interval and the sample time.

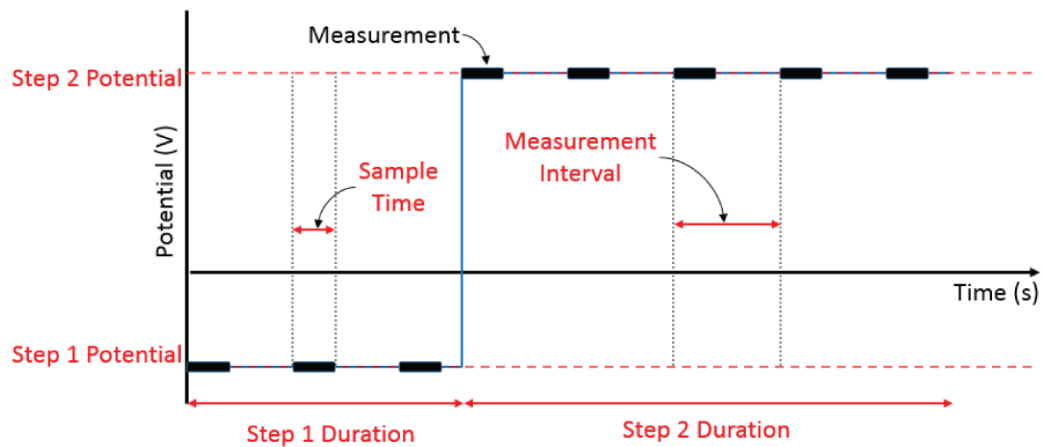
The option **Save Settings** allows you to save the present settings to a USB drive, while **Load Settings** allows you to load settings you have previously saved to a USB drive from this program. **Measure Eoc** enables you to measure the open-circuit voltage. Press **Continue >>** when you have initialized all desired settings.

Figure 106: Main menu



Several user-defined parameters are displayed in the figure below. Notice that there will always be a measurement at the very beginning of each step.

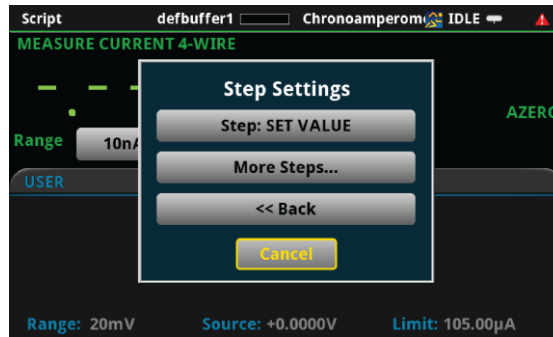
Figure 107: Possible double-step chronoamperometry (DSCA) plot



Step Settings

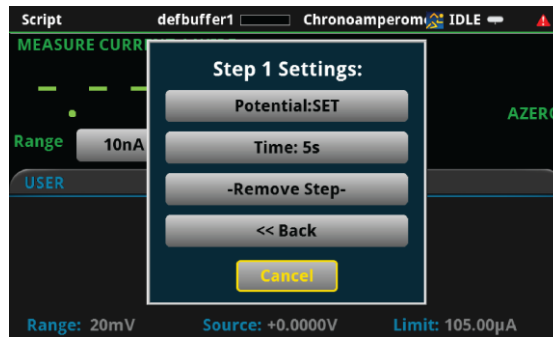
In chronoamperometry, the potential will be stepped to a specific value and remain there for a specific duration of time. From the Main Menu, you can select **Step Settings** to bring up a menu that allows you to set the number of potential steps to be sequentially executed, and the potential and duration for each measurement. Press **<< Back** or **Cancel** to go back to the Main Menu.

Figure 108: Step Settings



If you select the setting **Step**, then you will pull up a menu for that specific step.

Figure 109: Specific Step Settings



From this menu, you can set the specific step Potential or Time, or you can select -Remove Step- to delete the step.

Figure 110: Set Specific Step Potential

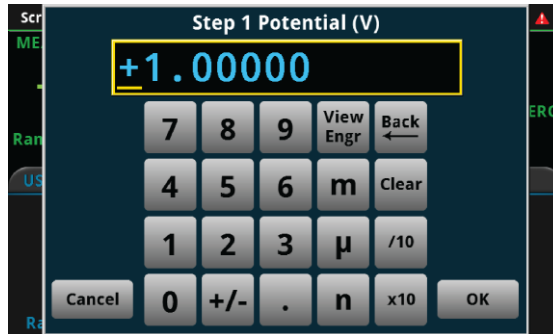
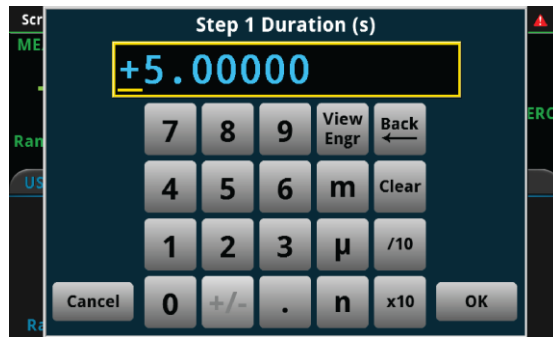
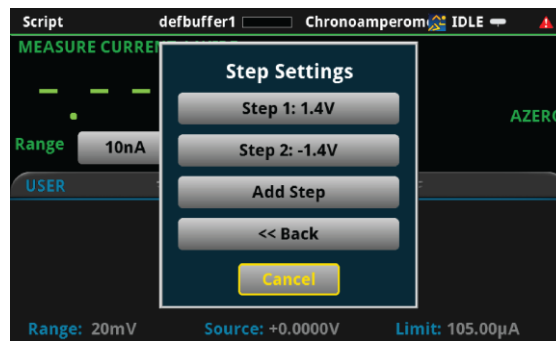


Figure 111: Set Specific Step Duration



Keithley Instruments' Chronopotentiometry and Chronoamperometry programs give you the flexibility to add up to eight steps, though such multi-step procedures are not common. For chronoamperometry, the most common protocols use a single step. To add more than one step, click **More Steps...** in the Step Settings menu. This immediately adds a Step 2, while providing the option to add even more steps.

Figure 112: Double-Step Chronoamperometry



Click **Add Step** to add another step. You can set as many as eight total steps.

Figure 113: Multi-Step Chronoamperometry



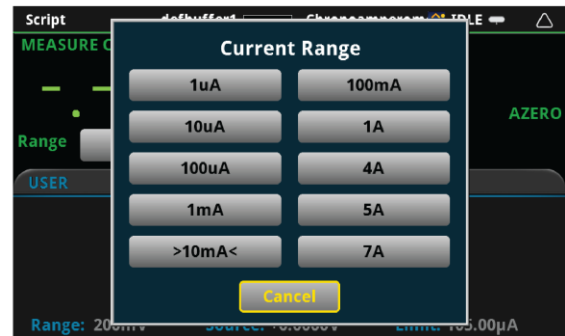
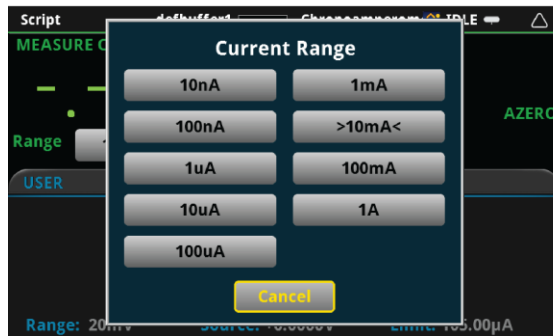
Current Range

From the Main Menu, select **Current Range** to set the current range for the experiment. This range constrains the currents that the instrument is capable of sourcing through the device under test, so you should choose a range above what you expect the maximum current to be. The available current ranges for the 2450 and 2460 are shown in the following figure.

Figure 114: Maximum current measurement range

Model 2450

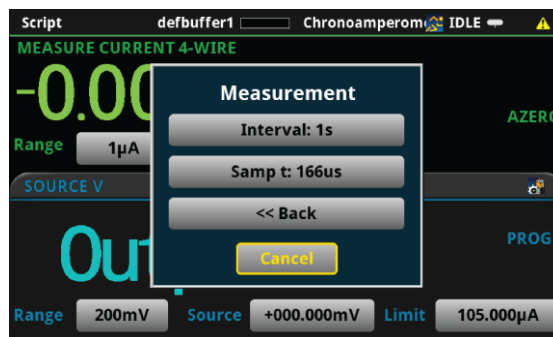
Model 2460



Measurement Settings

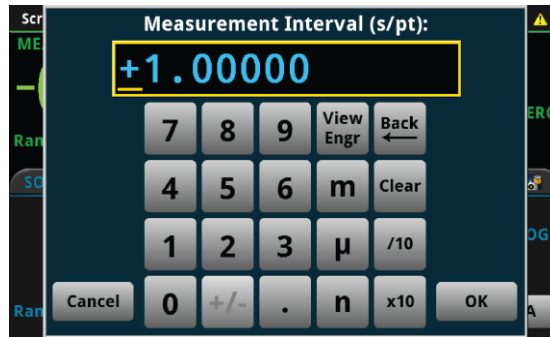
From the Main Menu, select Measurement to change the settings for the measurement **Interval** and the **Sample Time**.

Figure 115: Measurement dialog



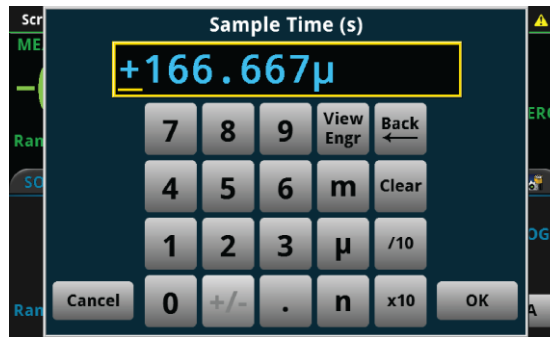
Select **Interval** to set the measurement interval.

Figure 116: Measurement interval



From the Measurement menu, you can also change the **Sample Time**. The instrument will average the measurement over this duration. Longer sample times will reduce the effect of noise on the measurement.

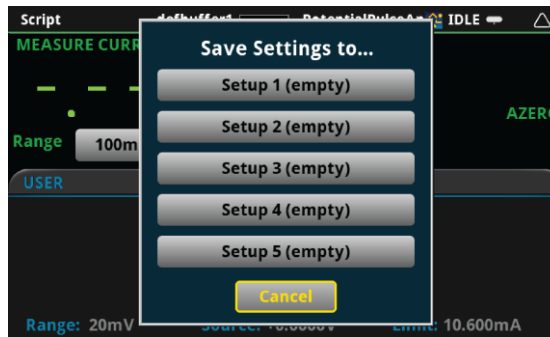
Figure 117: Sample time



Save Settings

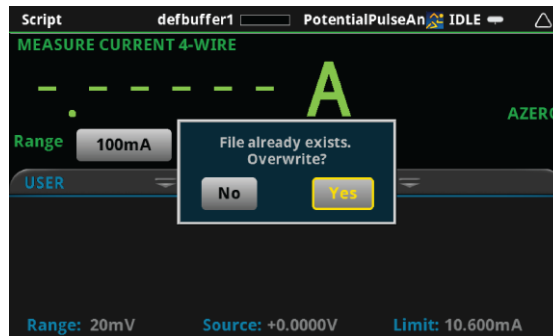
From the Main Menu, you can select **Save Settings** if you want to save the present settings to a USB drive for use or reference later. The settings will be saved in a .csv file. Many spreadsheet programs can open and read .csv files legibly. When a USB drive has been successfully recognized by the instrument, the **Save Settings** option will prompt you to choose from 1 of 5 setup files. All files labeled as **(empty)** have not yet been saved on your USB drive.

Figure 118: Save settings



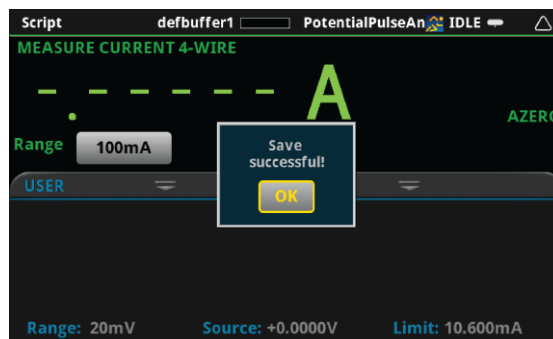
If you attempt to save the setup to a file that already exists on the USB drive—one that is not "empty"—the program will ask if you want to overwrite the existing file.

Figure 119: Overwrite dialog



You will be notified if the saving was successful.

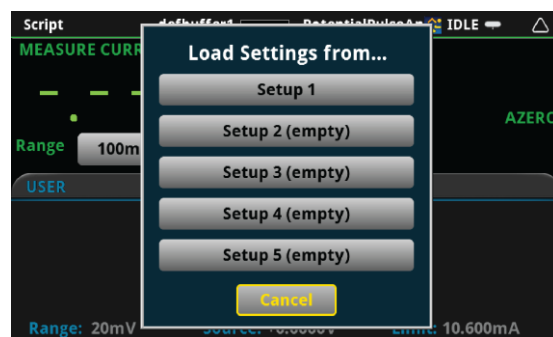
Figure 120: Save Successful



Load Settings

From the Main Menu, you can select **Load Settings** if you want to load settings from a USB drive that have been previously saved via the Chronoamperometry program. If the file for the settings has been altered, the program may not be able to load the settings. Numbers may be altered, but altering any text may cause an error while loading. When a USB drive has been successfully recognized by the instrument, the **Load Settings** option will prompt you to choose from one of five setups to load. Setups labeled as **(empty)** do not have setups associated with them, and will not be able to be loaded.

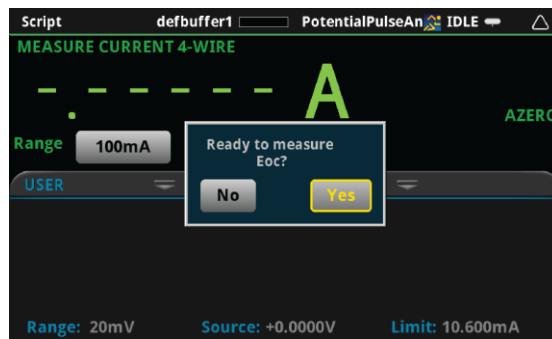
Figure 121: Load settings



Measure open circuit potential

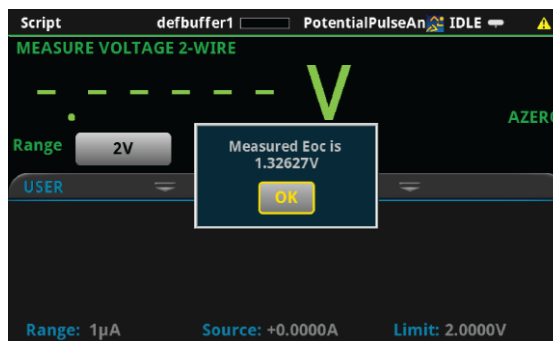
From the Main Menu, you can select **Measure Eoc** to measure the open-circuit potential of the device under test. The open-circuit potential is the potential across the terminals under a no-load condition. You will first be prompted whether you are ready to measure the open-circuit potential. Please ensure that the device under test is connected to the instrument in a proper 4-wire configuration before pressing **Yes**. You may also press **No** to return to the Main Menu.

Figure 122: Measure Eoc



A prompt will return to you the measured open circuit potential.

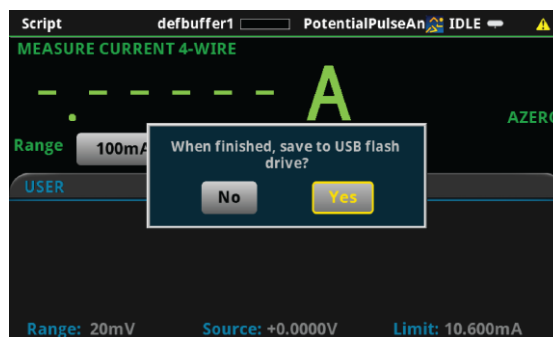
Figure 123: Measured Eoc



Step 7: Save the data to the USB drive on the front panel

After defining the test parameters, you may select **Continue >>** from the Main Menu to start the experiment. The program will first ask you if you want to save the results of the experiment.

Figure 124: Save results



If you choose **Yes**, a `.CSV` file will be created with a name containing the date and time of its creation, and results will be saved to it at the end of the experiment. Note that if there are more than 10,000 measurements made, the process of saving may take several minutes.

Step 8: Start the experiment

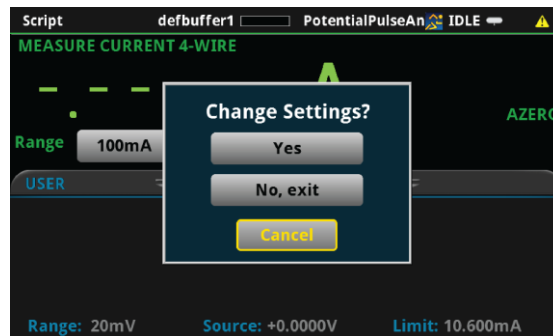
After the prompt to save your measurements, you are prompted to begin the experiment. If you want to begin, select **Yes**; the test will start immediately.

Figure 125: Start the Experiment



If you select **No** for the previous question, you will have the option to exit or change settings. If you select **Yes**, the program will take you back to the Main Menu. If you select **No, exit**, the program will terminate and any unsaved settings will be lost.

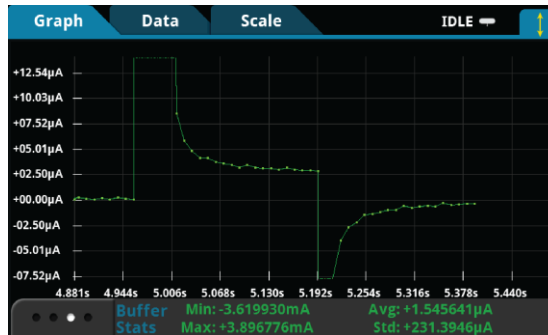
Figure 126: Start the Experiment



Step 9: View the graph

As the test begins an x-y plot of the measured current as a function of time will be displayed as shown below. When the test completes, you can revisit the graph by pressing the MENU key, then Graph.

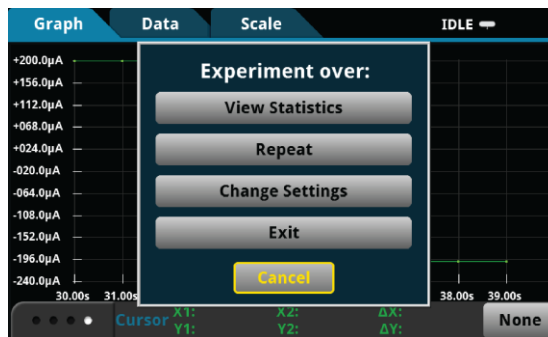
Figure 127: Real time graph of results



Step 10: View the data

After the experiment is finished, you will be prompted whether to **View Statistics** for the test data, **Repeat** the experiment using the same settings, **Change Settings** in the Main Menu, or **Exit** the program.

Figure 128: Experiment over dialog



View Statistics

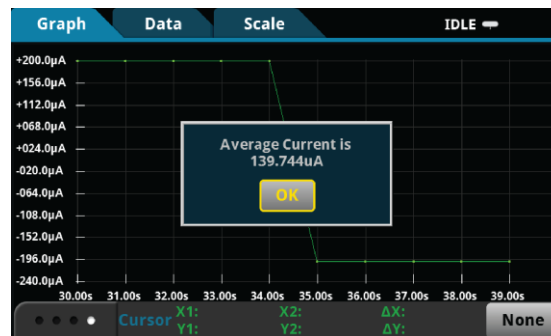
If you select View Statistics, you will see information about data as shown in the figure below. This includes the total number of points in the test (**NumPts**), the minimum measured current (**MinI**), the median measured current (**MedI**), the maximum measured current (**MaxI**), the average measured current (**AvgI**), and the standard deviation of the measured current (**StdevI**), and the duration of the test (**Test t**).

Figure 129: Statistics



You may select any one of the statistics to view a higher resolution view of the number displayed, or you can select <<<Return or Cancel to go back to the **Experiment over** menu. If you have set up more than one step you can select **Next Step>>** to view statistics for the next step.

Figure 130: High Resolution Statistics View



If you have set up multiple steps, you will also the option to navigate from the statistics of one step to another by selecting the **Next Step>>** and <<<Previous Step options.

Figure 131: Statistics for multi-step test



Repeat

If you select **Repeat**, the test will begin again using the same settings as originally set. You will be prompted to save the data.

Change Settings

If you select Change Settings, you will return to the Main Menu to start the test again.

Exit

You will be asked if you want to exit the program.

Step 11: View the results saved to the USB drive

If you choose to save the data to the USB drive, the readings will be saved in a .csv file after the test is finished.

To view the readings, open the file in a spreadsheet program on a computer. The figure below is an example of how the data appears (only a few readings from the test are shown). Current, potential, relative timestamps and general parameters and statistics for the test are included in the file.

Figure 132: Example data as it appears in a .csv file

| Keithley Instruments: Chronoamperometry | | | |
|---|--------------------------|-------------------------------|-------------|
| System Parameters | | | |
| Date/Time | Tue Nov 10 11:12:29 2015 | | |
| Model | 2460 | | |
| Serial Number | 4054493 | | |
| Firmware | 1.3.0s | | |
| Program Version | v1.0 | | |
| Measurement Interval: | 0.01 | s/pt | |
| Current Range: | 10mA | | |
| Step #1 Settings | | | |
| Potential | 1 | V | |
| Duration | 5 | s | |
| Step #1 Quick Stats | | | |
| 493 points recorded | | 1.02E-06 A average | |
| -7.45E-08 A minimum | | 2.00E-05 A standard deviation | |
| 1.06E-07 A median | | 5.00006 s to run experiment | |
| 0.000444092 A maximum | | | |
| Raw Data | | | |
| Step #1 | | | |
| Data Pt | Times (s) | Potential | Current (A) |
| | 1 0 | 1 | 0.000444 |
| | 2 0.011266 | 1 | 5.72E-07 |
| | 3 0.021338 | 1 | 4.15E-07 |
| | 4 0.031413 | 1 | 3.17E-07 |
| | 5 0.04149 | 1 | 1.94E-07 |

Chronopotentiometry

In Chronopotentiometry, the 2450 or 2460 steps the supplied current to a user-selected value where it is held constant for a user-specified period of time. While this current is held constant, the instrument will measure the resulting potential at user defined time intervals. The instrument can repeat this process for up to eight user defined steps.

Device connections

Connections are made from the instrument to the electrochemical cell using the Electrochemistry Translation Cable Accessory Kit cable as described in the Electrochemistry Translation Cable Accessory Kit Quick Start Guide.

Test procedure overview

The following list outlines the basic steps to perform Chronopotentiometry measurements and save the data to a USB flash drive using the Model 2450 or 2460. Details of each step are described more thoroughly in the following sections.

1. Select the TSP command set on the Model 2450 or 2460.
2. Before making physical connections to the cell, set the output of the 2450 or 2460 to the high-impedance output-off state.
3. Prepare the analyte and assemble the electrochemical cell.
4. Connect the instrument output terminals to the electrochemical cell.
5. Execute the test script.
6. Select and adjust each settings group in the Main Menu:
 - a. Set the step settings
 - b. Set the potential range
 - c. Set the measurement setting
7. After you are finished changing the settings, choose whether to save results.
8. Start the experiment and view the measurement results.
9. View the data on the graph.
10. After the experiment, choose whether to view experiment statistics, repeat the experiment, change settings, or exit.
11. View the results saved to the USB flash drive.

Test script parameters

The Chronopotentiometry test script (Chronopotentiometry.tsp) prompts the user to adjust certain test parameters as listed below:

Step current: $V_{\text{step}} \leq \pm 20 \text{ V}$

- 2450: $I_{\text{step}} \leq \pm 1.05 \text{ A}$
- 2460: $I_{\text{step}} \leq \pm 7.35 \text{ A}$

Step Duration: $0.01 \text{ s} \leq \text{time} \leq 99,999 \text{ s}$

Potential Range:

- 2450: 20 mV, 200 mV, 2 V, 20 V
- 2460: 200 mV, 2 V, 7 V, 10 V, 20 V

Measure Interval: $0.01 \text{ s} \leq \text{measurement interval} \leq 100 \text{ s}$

Sample Time:

- $0.01 \text{ PLC} \leq \text{sample time} \leq 10 \text{ PLC}$
- $\text{sample time} \leq (\text{measurement interval} - 0.05)\text{s}$
- $\text{sample time} \leq (\text{step duration} - 0.005)\text{s}$

Step 1: Select the TSP command set

To configure the Model 2450 or 2460 to use the TSP command set:

1. Press the **MENU** key.
2. Under System, select **Settings**.
3. Select the button next to Command Set and select **TSP**.
4. When prompted to reboot, select **Yes**.

Step 2: Place the output in the high-impedance output-off state

Before making physical connections from the instrument to the cell, set the output of the instrument to the high-impedance output-off state. When the high impedance output-off state is selected, the output relay opens, disconnecting the instrument from the load.

To set the output of the Model 2450 or 2460 to the high-impedance output-off state:

1. Press the **MENU** key.
2. Under Source, select **Settings**.
3. Select the button next to Output Off State and select **High Impedance**.
4. Press the **HOME** key to return to the home screen.

Step 3: Prepare the analyte and assemble the electrochemical cell

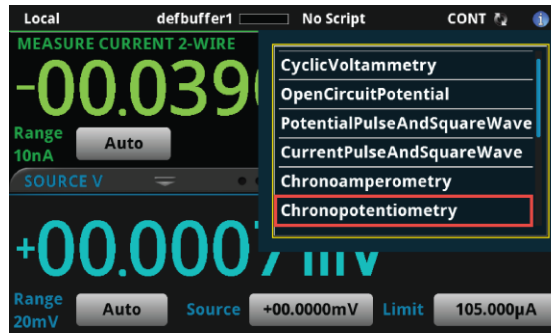
Step 4: Connect the four output terminals of the instrument to the electrochemical cell

Specific information on making connections from the instrument to the device is described in the Model 2450/60-EC Electrochemistry Lab System Quick Start Guide.

Step 5: Execute the Chronopotentiometry test script

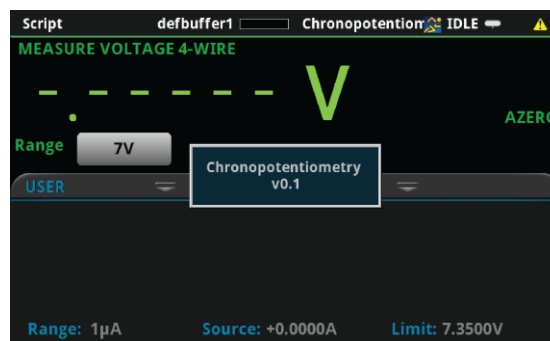
1. At the top of the home screen, touch the active script indicator. A list of available scripts is displayed

Figure 133: Selecting the Chronopotentiometry test script



2. Select **Chronopotentiometry**. The script begins to execute immediately and the following screen will be displayed for one second

Figure 134: Start of Chronopotentiometry test script

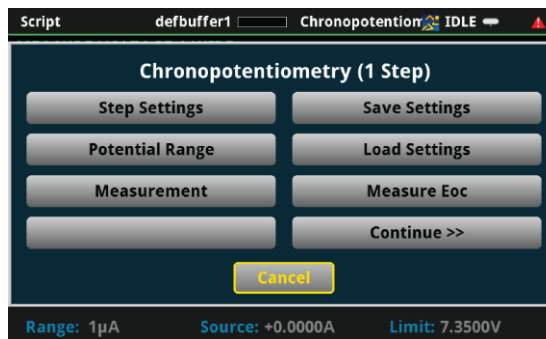


Step 6: Select and adjust each settings group in the Main Menu

After the program begins, the parameters must be defined from the Main Menu. The option **Step Settings** allows you to change the current value and duration of the current step, and to add up to 8 total steps in sequence. **Potential Range** allows you to constrain the potential that the instrument is capable of sourcing through the device under test. **Measurement** allows you to set the measurement interval and the sample time.

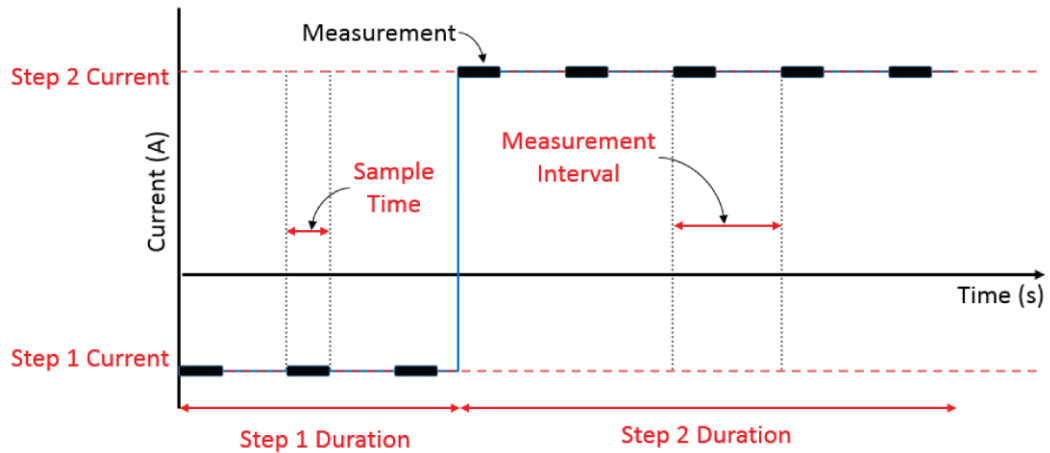
The option **Save Settings** allows you to save the present settings to a USB drive, while **Load Settings** allows you to load settings you have previously saved to a USB drive from this program. **Measure Eoc** enables you to measure the open-circuit voltage. Press **Continue** when you have initialized all of the settings needed.

Figure 135: Chronopotentiometry Main Menu



Several user-defined parameters are displayed in the figure below. Notice that there will always be a measurement at the beginning of each step.

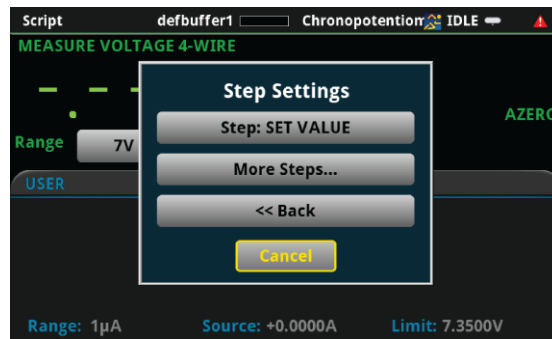
Figure 136: Possible double-step Chronopotentiometry (DSCP) plot



Step Settings

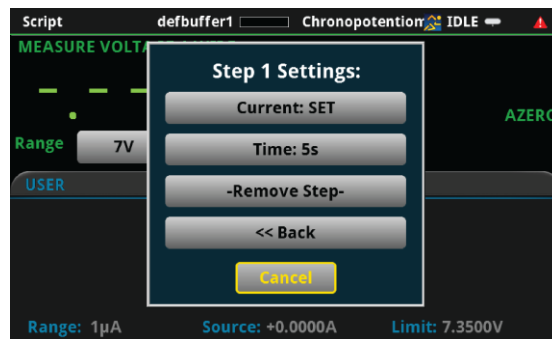
In Chronopotentiometry, the current will be stepped to a specific value and remain there for a specific duration of time. From the Main Menu, you can select **Step Settings** to bring up a menu that allows you to set the number of current steps to be sequentially executed as well as the current and duration of each step. Press **<< Back** or **Cancel** to go back to the Main Menu.

Figure 137: Step Settings



If you select the setting **Step**, then you will receive a menu for that specific step.

Figure 138: Specific Step Settings



From this menu, you can set the specific step **Current** or **Time**, or you can select **-Remove Step-** to delete the step.

Figure 139: Set Specific Step Current

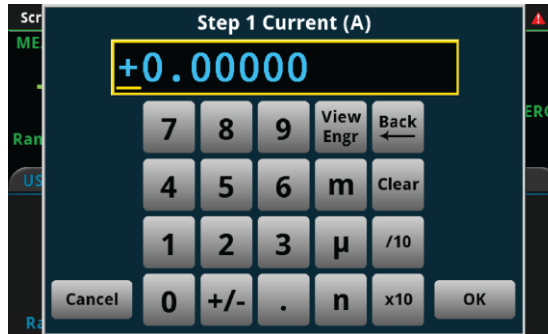
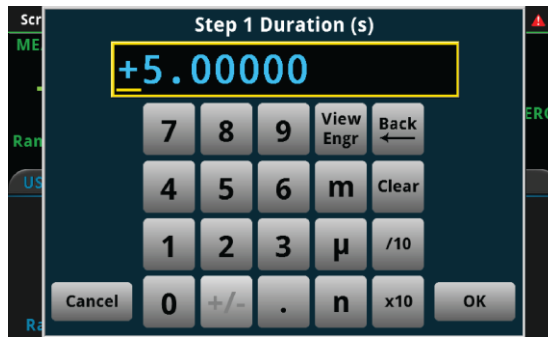
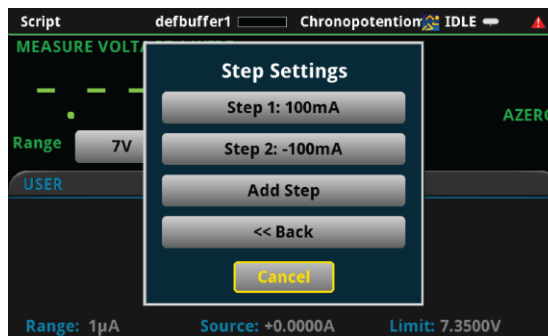


Figure 140: Set Specific Step Duration



The Keithley Instruments Chronopotentiometry and Chronoamperometry programs give you the flexibility to add up to eight steps, though such multi-step procedures are not common. For Chronopotentiometry, the most common protocols use a single step. To add more than one step, click **More Steps...** in the Step Settings menu. This immediately adds a Step 2, while providing the option to add even more steps.

Figure 141: Step Settings



Click **Add Step** to add another step. You can set as many as eight total steps.

Figure 142: Adding Another Step



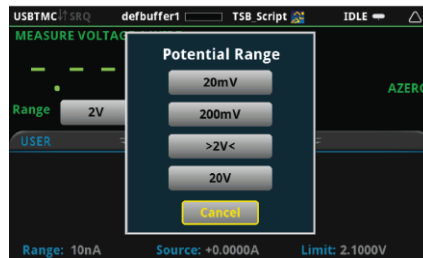
Potential Range

From the Main Menu, select **Potential Range** to set the potential range for the experiment. This range constrains the potential that the instrument is capable of sourcing through the device under test. You should choose a range above what you expect the maximum potential to be. The available potential ranges for the 2450 and 2460 are shown in the following figure.

Figure 143: Maximum potential measurement ranges

Model 2450

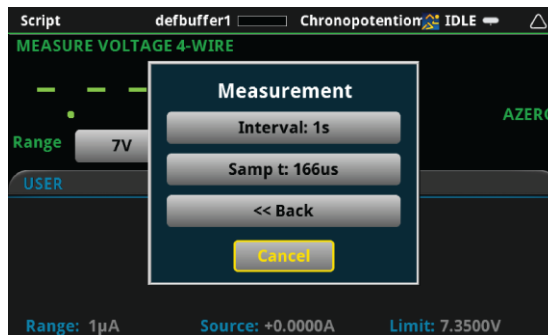
Model 2460



Measurement Settings

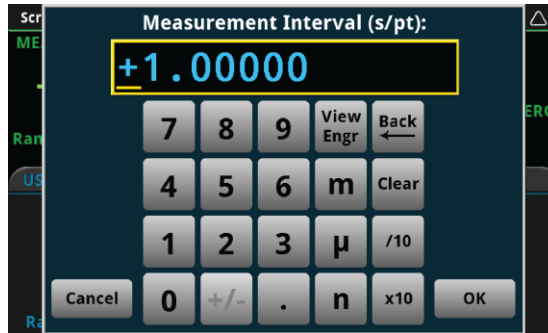
From the Main Menu, select **Measurement** to change the settings for the measurement **Interval** and the **Sample Time**.

Figure 144: Measurement



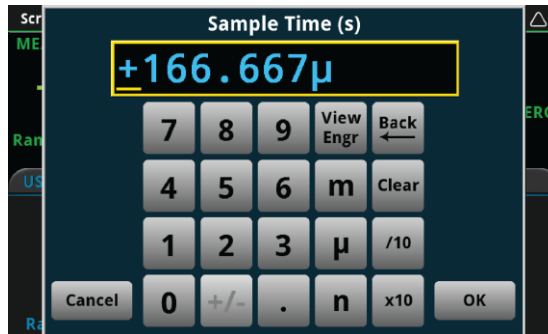
Select **Interval** to set the measurement interval.

Figure 145: Measurement Interval



From the Measurement menu, you can also change the **Sample Time**. The instrument will average the measurement over this duration. Longer sample times will reduce the effect of noise on the measurement.

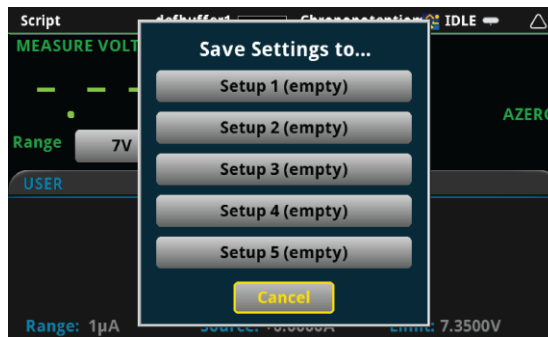
Figure 146: Sample Time



Save Settings

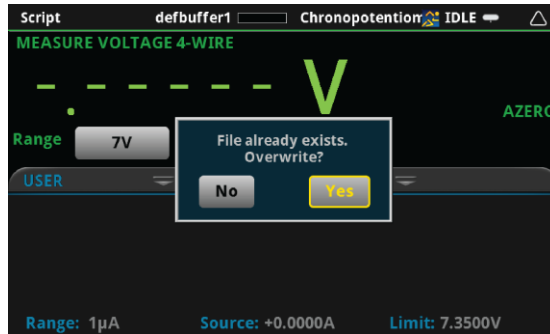
From the Main Menu, you can select **Save Settings** if you want to save the present settings to a USB drive for use or reference later. The settings will be saved in a .csv file. Many spreadsheet programs can open and read .csv files legibly. When the instrument recognizes the USB drive, the **Save Settings** option will prompt you to choose from 1 of 5 setup files. All files labeled as **(empty)** have not yet been saved on your USB.

Figure 147: Save Settings



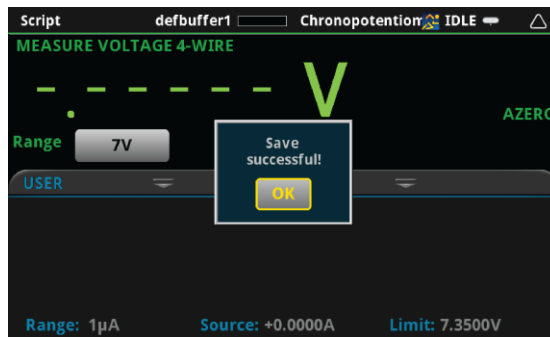
If you attempt to save the setup to a file that already exists on the USB drive—one that is not empty—the program will ask if you want to overwrite the existing file.

Figure 148: File already exists, Overwrite?



You will be notified if the saving was successful.

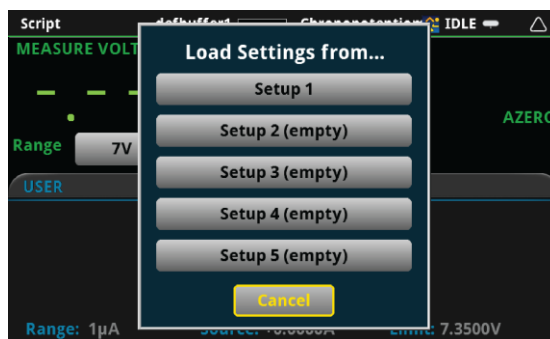
Figure 149: Save successful



Load Settings

From the Main Menu, you can select **Load Settings** if you want to load settings from a USB drive that have been previously saved via the Chronopotentiometry program. If the file for the settings has been altered, the program may not be able to load the settings. Numbers may be altered, but altering any text may cause an error while loading. When a USB drive has been successfully recognized by the instrument, the **Load Settings** option will prompt you to choose from one of five setups to load. Setups labeled as **(empty)** do not have setups associated with them, and will not be able to be loaded.

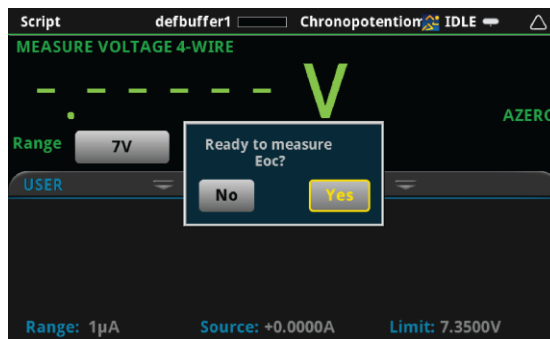
Figure 150: Load settings



Measure open circuit potential

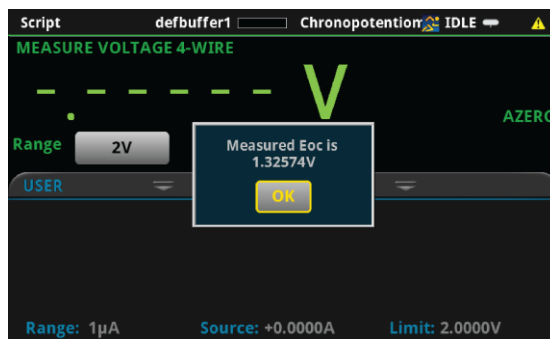
From the Main Menu, you can select **Measure Eoc** to measure the open-circuit potential of the device under test. The open-circuit potential is the potential across the terminals under a no-load condition. You will first be prompted whether you are ready to measure the open-circuit potential. Please ensure that the device under test is connected to the instrument in a proper 4-wire configuration before pressing **Yes**. You may also press **No** to return to the Main Menu.

Figure 151: Measure Eoc



A prompt will return the measured open circuit potential.

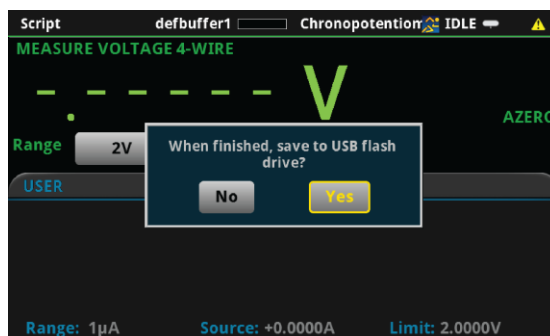
Figure 152: Measured Eoc



Step 7: Save the data to the USB drive on the front panel

After defining the test parameters, you may select **Continue >>** from the Main Menu to start the experiment. The program will first ask you if you want to save the results of the experiment.

Figure 153: Save results

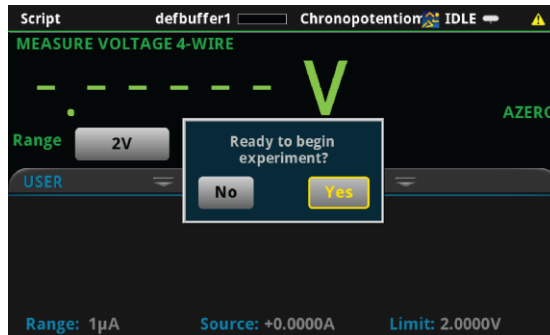


If you choose **Yes**, a `.CSV` file will be created with a name containing the date and time of its creation, and results will be saved to it at the end of the experiment. Note that if there are more than 10,000 measurements made, the process of saving may take several minutes.

Step 8: Start the experiment

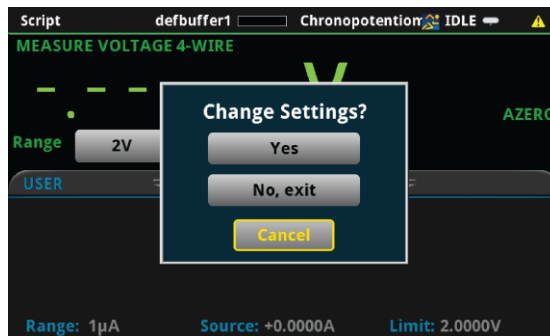
After the prompt to save your measurements, you are prompted to begin the experiment. If you want to begin, select **Yes**; the test will start immediately.

Figure 154: Start the experiment



If you select **No** to the previous question, you will have the option to exit or change settings. If you select **Yes**, the program will take you back to the Main Menu. If you select **No, exit**, the program will terminate and any unsaved settings will be lost.

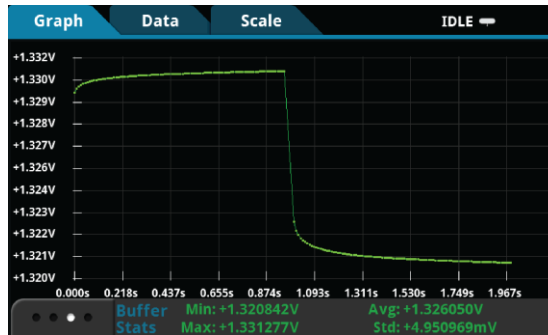
Figure 155: Change Settings?



Step 9: View the graph

As the test begins an x-y plot of the measured current as a function of time will be displayed as shown below. After exiting the script, the graph can be viewed by pressing the MENU key, then Graph.

Figure 156: Real time graph of results



Step 10: View the data

After the experiment is finished, you will be prompted whether to **View Statistics** for the test data, **Repeat** the experiment using the same settings, **Change Settings** in the Main Menu, or **Exit** the program.

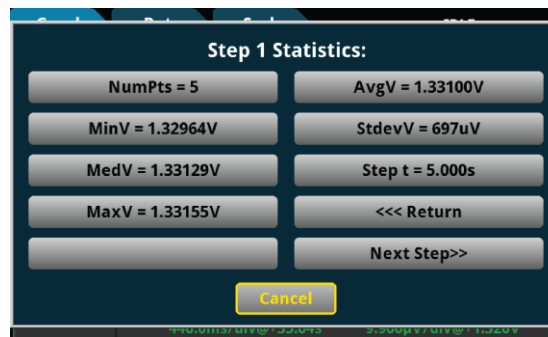
Figure 157: Choose to view statistics, repeat test, change settings and repeat, or exit



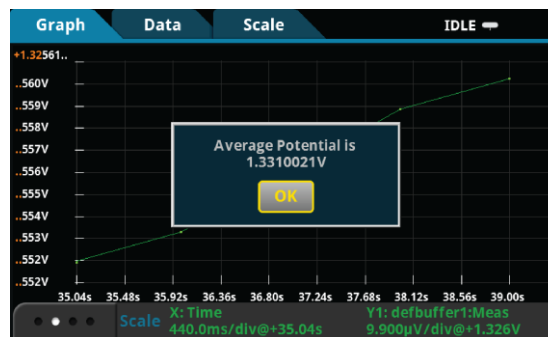
View Statistics

If you select **View Statistics**, you will see information about data as shown in the figure below. This includes the total number of points in the test (**NumPts**), the minimum measured potential (**MinV**), the median measured potential (**MedV**), the maximum measured potential (**MaxV**), the average measured potential (**AvgV**), and the standard deviation of the measured potential (**StdevV**), and the duration of the test (**Test t**).

Figure 158: Statistics

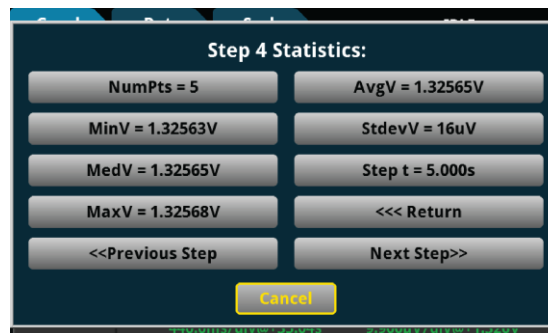


You may select any one of the statistics to view a higher resolution view of the number displayed, or you can select **<<<Return** or **Cancel** to go back to the **Experiment over** menu. If you have set up more than one step you can select **Next Step>>** to view statistics for the next step.



If you have set up multiple steps, you can navigate from the statistics of one step to another by selecting the **Next Step>>** and **<<Previous Step** options.

Figure 159: Statistics



Repeat

If you select **Repeat**, the test will begin again using the same settings as originally set. You will be prompted to save the data.

Change Settings

If you select **Change Settings**, you will return to the Main Menu to start the test again.

Exit

You will be asked if you want to exit the program.

Step 11: View the results saved to the USB drive

If you choose to save the data to the USB flash drive, the readings will be saved in a `.csv` file after the test is finished.

To view the readings, open the file in a spreadsheet program on a computer. The figure below is an example of how the data appears (only a few readings from the test are shown). Current, potential, relative timestamps and general parameters and statistics for the test are included in the file.

Figure 160: Example of data as it appears in a .csv file

| Keithley Instruments: Chronopotentiometry | | | |
|---|--------------------------|-------------|-------------------------------|
| System Parameters | | | |
| Date/Time | Tue Nov 10 11:47:25 2015 | | |
| Model | 2460 | | |
| Serial Number | 4054493 | | |
| Firmware | 1.3.0s | | |
| Program Version | v1.0 | | |
| Measurement Interval: | 0.01 s/pt | | |
| VRange | 2V | | |
| Step #1 Settings | | | |
| Current | 0.1 A | | |
| Duration | 1 s | | |
| Step #1 Quick Stats | | | |
| | 96 points recorded | | 1.331054 A average |
| | 1.330080509 V minimum | | 0.000223 A standard deviation |
| | 1.331124306 V median | | 1.000066 s to run experiment |
| | 1.331276894 V maximum | | |
| Raw Data | | | |
| Step #1 | | | |
| Data Pt | Times (s) | Current (A) | Potential (V) |
| 1 | 0 | 0.1 | 1.330081 |
| 2 | 0.010288 | 0.1 | 1.330311 |
| 3 | 0.020366 | 0.1 | 1.330431 |
| 4 | 0.030442 | 0.1 | 1.330516 |
| 5 | 0.040499 | 0.1 | 1.330572 |

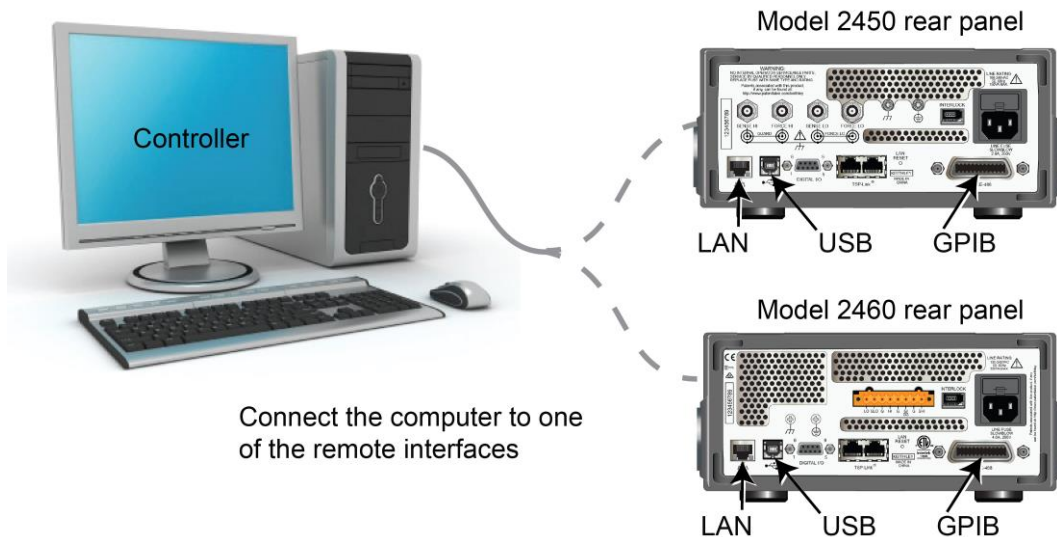
Remote operation

The code for the test scripts was created using Keithley Test Script Processor (TSP®) code. TSP technology embeds complete test programs inside the instrument. The SourceMeter® instruments have an internal processor that makes them capable of executing test programs (scripts) without the use of an external computer. A script is a text file that can be edited in any text editor such as Notepad. This allows you to make changes to the program easily, if necessary.

Set up remote communications

If you choose to run the test scripts from Keithley Test Script Builder (TSB), you will need to configure the 2450 or 2460 to run using a computer over a remote interface. The following figure shows the rear-panel connections for the remote communications interfaces on the 2450 and 2460. The 2450 and 2460 automatically detect which remote interface is connected to the computer.

Figure 161: Models 2450 and 2460 remote interface connections



Editing the test script

You can edit the test scripts in Keithley's full-featured editor, Test Script Builder (TSB). TSB is a software tool that can be downloaded from the [Keithley Instruments support website](http://www.tektronix.com/keithley) (<http://www.tektronix.com/keithley>). This editing tool has built-in debugging capability and enables code execution before you download the script to the instrument. More information about Test Script Processor and Test Script Builder can be found in the "Introduction to TSP operation" and "TSP command reference" sections of the Model 2450 and 2460 Reference Manuals.

⚠ CAUTION

The test scripts were written with very tight voltage source and current measurement timing controls. If you edit the test script, you run the risk of losing the synchronization of the test. Keithley Instruments is unable to support edits to the test script.

To edit the TSP code:

1. Insert the USB flash drive into a computer and save the `.tsp` file to the computer.

NOTE

To keep the original test script, make a backup copy of it before making any changes to the script.

2. Open the `.tsp` code file with a text editor or TSB. TSB allows you to make changes to the code and execute the code to verify the operation.

NOTE

Before executing the TSP code in TSB, the 2450 or 2460 must be set to use the TSP command set.

3. If you are using TSB to edit the code, select the TSP command set:
 - a. Press the **MENU** key.
 - b. Under System, select **Settings**.
 - c. Select the button next to Command Set and select **TSP**. The instrument will prompt you to reboot.
 - d. To execute the test automatically in TSB, use any of the supported communication interfaces for the instruments (GPIB, USB, or ethernet).
4. After making changes to the TSP code, save the script to the USB flash drive as a `.tsp` file with a different name. The code then can be executed by:
 - Using the active script indicator on the front panel
 - Downloading it to the 2450 or 2460
 - Using the TSB program

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