



High Throughput Array Screening System Operation and Assembly Manual



OPERATION and ASSEMBLY MANUAL

VERSION 1.0

ArrayPGstat Operation and Assembly Manual

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Crown Point, Indiana

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GLOSSARY

Listed below are the symbols used to identify windows and actions in the ArrayPGware software.

SYMBOL	WINDOW/ACTION	EXAMPLE
[Square brackets]	Window	[Run Arraystat]
Bold	Button / Button Header	START
<u>Bold underline</u>	Tab or Header	<u>Aux. TC/MFC</u>
<u>Underline</u>	End-user input	<u>Number TC</u>
“Quotes”	End-user input options	“Ramp to”

SPECIFICATIONS

The ArrayPGstat is an automated 25-channel potentiostat for high-throughput characterization of electrode components. The ArrayPGstat can switch out (i.e. open circuit) any number of electrodes while others are tested either in parallel or sequentially. The operator can evaluate electrodes individually, or in separate rows or columns. The ArrayPGstat can apply complex sequences of test parameters to any number of the array electrodes in parallel or sequentially.

Cell Control	
Compliance Voltage	±15V
Max Output Current	±1.625 A over 25 channels / 65mA max per Ch.
Rise Time	45 uS for 1k Ohm load (0%-100% signal)
Slew Rate	0.2 V/μs
Bandwidth	8 kHz (-3 dB, 1k Ohm load)
Applied DC Potential Ranges	1 (±10 V)
Applied Potential Resolution	0.3 mV
Applied Potential Accuracy	< 0.04% FSR
Current Autoranging	Yes
Applied DC Current Ranges	2 (±250μA , ±65mA)
Best Applied Current Resolution	3 nA, 0.003% of FSR
Applied Current Accuracy	0.03% of FSR
Input Bias Current	500 pA
Input Impedance	250 GΩ parallel to 3 pf
Maximum Update Rate	4 μs
Maximum Scan Rate	100V/sec
IR Compensation	N/A
External Control	N/A
Potential Measurement	
Measured DC Potential Ranges	1(±10 V)
Resolution	3 μV, 300 μV (0.006%, 0.003% of FSR)
Accuracy	0.08 or 0.03% of FSR
Current Measurement	
Measured Current Ranges	Potentiostat: 2 (± 250uA, ± 65 mA)
Potentiostat Min to Max	80nA to 65mA
Best Resolution	Galvanostat: 80 nA (0.003% of FSR) Potentiostat: 80 nA - 20 μA (0.06 - 0.003% of FSR In Order of Range)
Accuracy	Galvanostat: 0.03% of FSR; Potentiostat: 0.3 - 0.03% of FSR Depending on Range
Data Acquisition	
Acquisition Speed	250 k samples/s (Aggregate) 9.25 k samples/s/ch. (min 27 channels)
DAC Resolution	16 bits
FSR: Full Scale Range	

SAFETY

Computer Control and System Safety

Precautions should be taken to ensure sustained operation of the PC controlling the ArrayPGstat. A failure of the PC or a crash in the software for any reason may leave the load in an 'ON' but uncontrolled state that may damage the ArrayPGstat.

Grounding

For the safety of the equipment and the operator, the ArrayPGstat must be connected by means of a protective conductor to earth ground. Under no circumstances should the ground be bypassed. It is advisable not to unplug the instrument when not in use. This protection must not be disabled by the use of two-conductor extension cord or adapter, or any other type of connection that does not maintain earth ground continuity.

ESD Protection

Electrostatic Discharge (ESD) can cause immediate or latent damage to electronic circuits. NuVant products are adequately protected against ESD for their intended use. However, it is possible to damage the product by delivering electrostatic discharges when touching, moving the product, or connecting leads. It is advisable to ground and discharge yourself before touching the product. It is also advisable to ground and discharge any leads and the fuel cell before connecting it to the array potentiostat.

AC Supply Voltage

Before first connecting the power supply to the equipment, make sure that the line voltage is nominally 120 volts. Never operate the equipment from a line voltage or frequency in excess of that specified. *The equipment may be damaged by the application of incorrect line voltage. Voltage and current conditions inside that equipment described in this manual are sufficient to cause injury and possible death. Only qualified technicians should be permitted to remove the cover or attempt repairs.*

Fuses

Upon blowing a fuse make sure that the replacement fuse, accessible from the exterior of the equipment on the rear panel, is of the correct rating. The rating of the ac line fuse must correspond to the values stated in the specifications. Refer to the rear panel information in the Hardware Installation section.

Live Conductors

The opening of covers or removal of parts of this equipment could expose live conductors. The equipment must be disconnected from all power and signal sources prior to the onset of any adjustment, replacement, maintenance, or repair. Only qualified personnel must do adjustments, maintenance or repair.

HARDWARE CONVENTIONS

- Physical switches and connectors on the front panel relevant to operation are designated as **Bold Blue** text. For example, **Main Power Switch** and **Cell Enable Switch**.
- Physical switches and connectors on the back panel relevant to operation are designated as **Bold Red** text. For example **Data Communication Port**.

HARDWARE INSTALLATION

Description of Controls and Connections

This section provides a general description of the controls and indicators of the panels on the ArrayPGstat potentiostat.

Potentiostat Front and Back Panel

The following are the parts on the front panel of the ArrayPGstat as shown in the diagram below.



Figure 1 ArrayPGstat Front Panel

1. Electrode channel connectors – Each of the five electrode channel connectors corresponds to five electrode channels. Figure 2 shows one electrode cable connected to the first electrode channel connector. The electrode cable has fifteen electrodes, providing the working, counter and reference electrodes for five channels.

2. . Main Power Switch – This switch is used to turn the ArrayPGstat system on or off. Pressing this switch on will allow the main AC voltage to be applied to the transformers of the power supplies. Pressing this switch to the off position will disconnect the AC voltage from the transformers of the power supplies.



Figure 2 Electrode Cables connected to ArrayPGstat

The following are the parts on the rear panel of the unit.



Figure 3 Electrode Cables connected to ArrayPGstat

1. AC Power Input – The ArrayPGstat operates on both 115 and 230 VAC. These voltages can be selected on the mains input module located on the back of the instrument. Fuse rating for this instrument are also located on the decal along with additional information for the user.

2. AC Main Power Switch – AC main power switch. This must be on for the front panel **Main Power Switch** to illuminate.

3. Main Power Voltage Selector Switch and AC Fuse Holder – External AC fuse holder. 4 A at 250 VAC Slow blow fuse for voltage selections of 115 VAC or 2 A at 250 VAC Slow blow fuse for voltage selection of 230 VAC 50~60Hz.

4. USB Data Communication Cable – Do not connect this cable to the computer until the software is installed. Ensure this cable is inserted completely into the instrument and computer during use. Do not connect a USB extension cable to this cable. Do not replace this cable without contacting NuVant Systems to obtain an appropriate cable.

5. Serial Number Plate – Removal of or defacing the serial number plate will void the warranty.

6. Cooling fans – Allow at least 2 inches of space behind and to the sides of the cooling fans. Periodically inspect the fan ports to make sure debris does not obstruct air flow.

Procedure for Connection of Electrode Cables to an Electrochemical Device

Electrodes should never be connected or disconnected from the Arraystat when the cell is enabled. If the software is running, disable the cell using the software AND by putting the **Cell Enable Switch** in the out position, whether or not it is illuminated. For operator safety, and protection of the Arraystat electronics, always connect and disconnect electrodes in the following order (NOTE: counter, reference and working electrodes must ALL be connected to the electrochemical device):

1) Electrode connection;

- a) With the cell disabled connect the White Reference electrode lead;
- b) With the cell disabled connect the Red Counter electrode lead
- c) With the cell disabled connect the Black Working electrode lead.

2) Electrode disconnection;

- a) With the cell disabled disconnect the Black Working electrode lead;
- b) With the cell disabled disconnect the Red Counter electrode lead;
- c) With the cell disabled disconnect the White Reference electrode lead;
- d) Do not enable the cell unless all electrodes are connected correctly.

Model ArrayPGstat Fuel Cell Load Precautions and Connection Information

System Connections

The ArrayPGstat's data acquisition system has the following electrical characteristics that **MUST** be observed.

1. The assembled high throughput device must be placed on a non-conducting and heat resistant surface so that it is not grounded.
2. Any heater attached to the fuel cell body or end plate must be completely isolated from the heater power source, such as the AC line. Leakage current from the AC line into the fuel cell electrode circuits may result in incorrect and noisy data readings and may cause damage to the ArrayPGstat and its electronics.
3. Connections for the reactant supply on the Counter side are on the counter electrode and for the array side it is on the end plate. All reactant supply lines, and liquid cooling lines if used, **must** be non-conductive or have a non-conducting section, to provide for electrical isolation from the ground.
4. Care is to be taken in connecting the electrical leads between the pin-jacks on the array block and pin-jacks on the front panel of the ArrayPGstat so that the numbering corresponds to the appropriate positions on the array, lest a wrong ranking of catalyst performances result.

Input Precautions

The maximum current of the ArrayPGstat is 2.5 AMPERES when all the channels are active. The current per channel **should not exceed 65 mA** when all channels are active.

Software installation and operation

This chapter provides an overview of the NuVant ArrayPGstat software used to control the ArrayPGstat, acquire data traces and provide Graphic User Interface. Included in this section is the initial start-up of the fuel cell test system and software, application of reactant gases, electrical and mechanical connections and precautions, applying AC power to the test system hardware and, finally, starting the control program to test the system, as well as collecting and saving the data.

Software and Hardware Installation Instructions

The ArrayPGware installation DVD includes the LabVIEW Runtime Engine, the NI-DAQmx driver, the NI-Serial driver and the ArrayPGware software. Version specific installation instructions are provided with each ArrayPGware installation DVD. *Users are urged to read the instructions before installing the software and connecting hardware. Please note that the recommended pattern is to install the software prior to*

connecting the hardware so all hardware is automatically recognized by both the Windows and National Instruments New Hardware Wizards.

- 1.0 Install the LabVIEW Runtime Engine using the default installation options. Restart the computer after installation is complete.
- 2.0 Install NI-DAQmx with the default installation options. Restart the computer after installation is complete.
- 3.0 If the temperature and/or mass flow controller options are ordered, the NI-Serial driver on the installation DVD must also be installed. Installation and COM port configuration instructions are provided under separate cover when these options are ordered.
- 4.0 Install the ArrayPGware software. A re-boot is not required after installing the ArrayPGware software.
- 5.0 Connect the USB cable provided to the back of the ArrayPGstat but do not plug it into the computer yet.
- 6.0 Apply power to the ArrayPGstat by connecting and plugging in the external power supply. Ensure the physical Cell Enable switch is in the out (off) position. Press the power switch on the front panel. This power switch should illuminate green and you should hear the sound of the cooling fan when the instrument is on.
- 7.0 Connect the USB cable to your computer and wait to see the new hardware installed twice. The hardware must be recognized first by Windows and then by the National Instruments Measurement & Automation Explorer (MAX).

NuVant provides periodic software upgrades to ensure that instruments are working at the highest efficiency. NuVant will be in contact when a software upgrade becomes available.

The NI-DAQmx driver and LabVIEW Runtime Engine may be downloaded from the National Instruments website www.ni.com. Direct links to the drivers can be found at <http://nuvant.com/download-software/software/>.

RUNNING SOFTWARE

Note: It is recommended that you disable any screen saver when the control and data acquisition software is running so that it is possible to monitor the system to ensure proper functioning in the initial stages.

Start Screen

1. Go to **Start-->All Programs-->ArrayPGware vx.x.xx.xxxx-->ArrayPGware** to run the program.

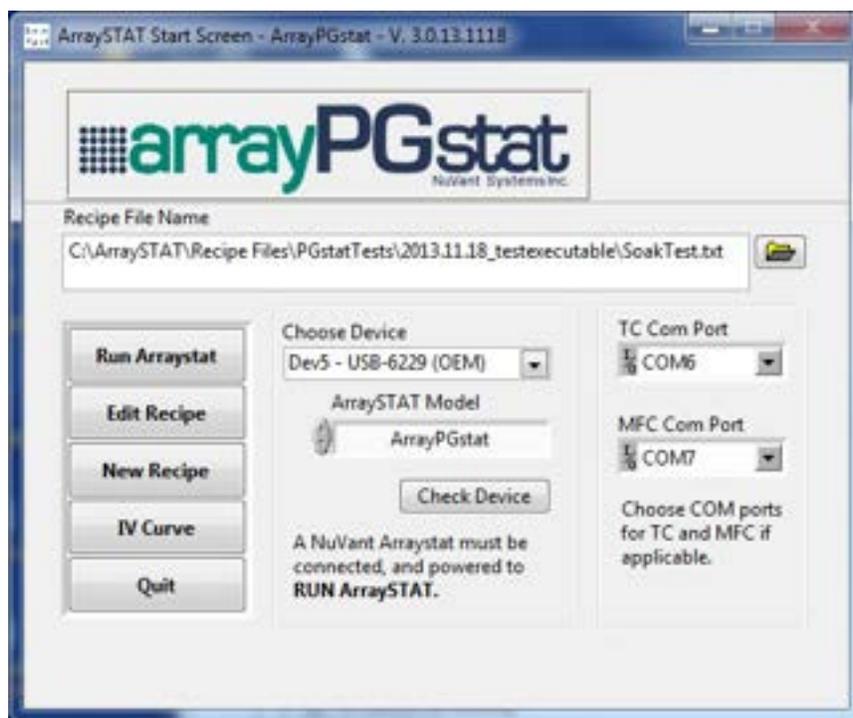


Figure 4 ArrayPGstat [Start Screen]

2. Ensure that the software sees the device in Choose Device. If the field is empty, click **Check Device**. Once the device is seen by the software, select “ArrayPGstat” from the Arraystat Model dropdown menu.
3. Recipe files may be loaded from the [Start Screen] by clicking on the folder symbol  next to the **Recipe File Name** entry field.
4. There are five main options on the [Start Screen]: **Run Arraystat**, **Edit Recipe**, **New Recipe**, **IV Curve** and **Quit**.

Edit Recipe: opens [Recipe Editor], where the recipe can be viewed and edited

New Recipe: creates a new recipe file. Name the file and then click **Edit Recipe**

to start editing.

Run Arraystat: opens [Run Arraystat].

IV Curve: [IV Curve], where a data file can be viewed.

Quit closes all running screens

Recipe Editor

1. Recipe files can be loaded by clicking on the folder symbol  next to the Recipe File Name in the [Start Screen]. Recipe files may also be loaded through File → Open on the menu bar in [Recipe Editor]. Files loaded through the [Recipe Editor] menu bar do not automatically load into the [Start Screen] Recipe File Name control.

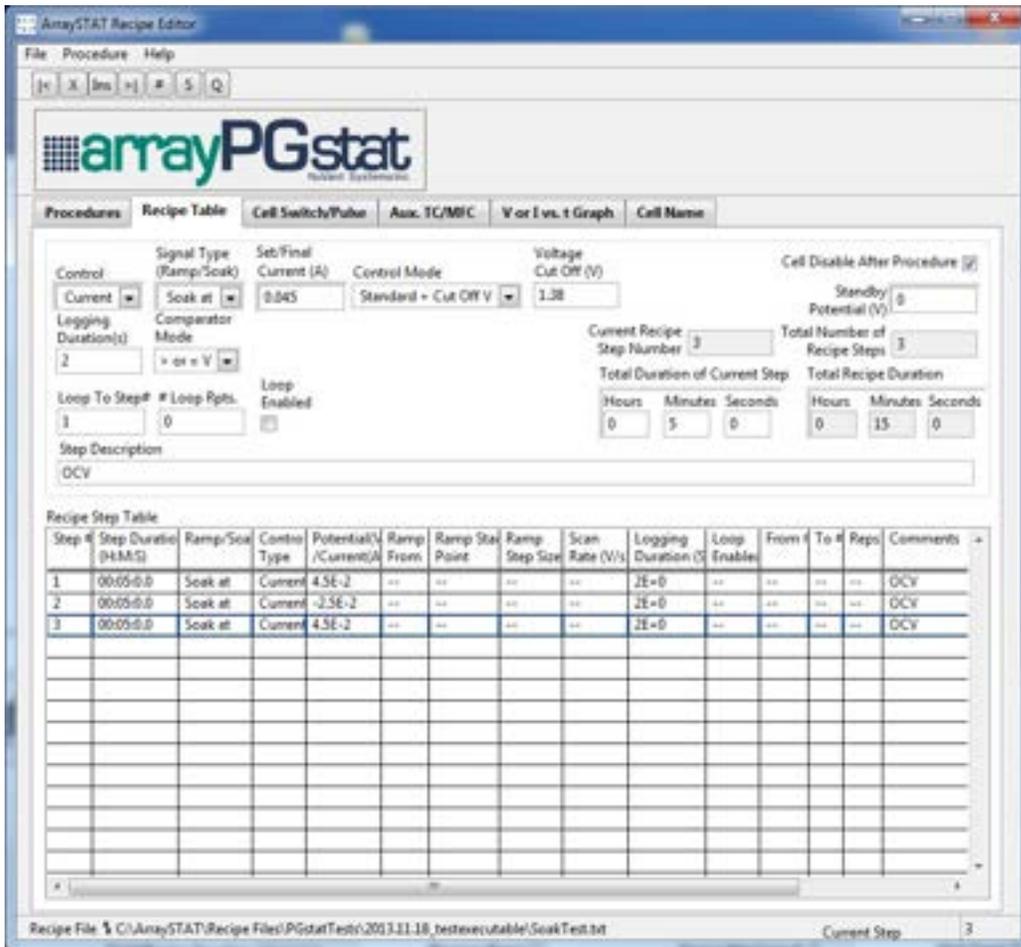


Figure 5 Recipe Editor Window

2. The entire recipe is displayed in Recipe Table. The current step being edited is highlighted by the blue border. The input values are located above the Recipe Step Table.
3. To move between steps, use the buttons in Editing Controls field
4.  -- **Go To Prev Step**: moves the blue border up to the previous step for it to be edited.
-  -- **Go To Next Step**: moves the blue border down to the next step for it to be edited.

-- **Go To Step #:** a window asks users to enter the desired recipe step to edit. The blue border will be moved to the desired step.



Figure 6 Go to Step #

X -- **Delete This Step:** deletes the selected step (blue border).

Ins -- **Insert A New Step:** copies the current selected step and inserts it at the current position.

- Recipe editor controls:** controls the potential output type (soak at, ramp to, or OVC)



Figure 7 Recipe editor controls

Control Type can be set to either “Voltage” or “Current”. If set to “Voltage”, Signal Type (Ramp/Soak) has six options: “Ramp to”, “Soak at”, “OCV”, “CV Scan”, “Potential Step” and “Pulse Voltammetry”. If set to “Current”, only “Ramp to” and “Soak at” are available. “Ramp to” ramps the potential/current from either “E ref.” or “E prev.” (selected in Ramp From) to the value specified in Set/Final Potential/Current. If the Ramp From is set to “E ref.”, the user can specify the starting ramp value in Initial Ramp Potential/Current. The user can also select the Step Size and Scan Rate. “Soak at” holds the potential/current at the value specified in Set/Final Potential/Current for a time specified in Total Duration of Current Step. The user can also set the Logging Duration (s), which

is the sample recording interval. Selecting Reverse ramp runs a reverse scan of the step being edited. Loop Enabled loops the recipe from the step being edited to the step specified in Loop to Step # for a number of loops input in # Loop Rpts. **Total Recipe Duration** field computes and displays the total running time of entire recipe.

6. ArrayPGstat allows the user to turn on/off every cell or run different pulse trains individually in **Cell Switch/Pulse Settings**.

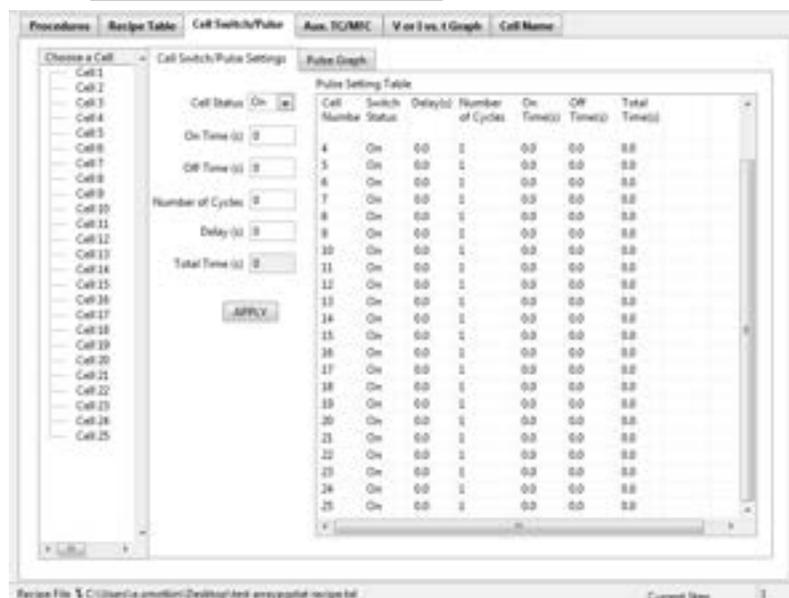


Figure 8 Cell switch and pulse settings

The user can turn on/off each cell or apply a pulse train to each cell in **Cell Switch/Pulse** panel. Cell Status changes the cell to “On”, “Off” or “Pulse”. When a cell is set to “Pulse”, “On Time (s)”, “Off Time (s)”, “Number of Cycles” and “Delay (s)” need to be input as these values control a pulse train. “Delay(s)” is the offset time that a pulse train has and refers to the beginning of the current step. “On Time (s)” and “Off Time (s)” are the time the cell is on or off in a single cycle. “Number of Cycles” is the number of cycles of the pulse train. “Total Time (s)” is the total running time of the pulse train.

7. The temperature controller (TC) settings of up to 8 channels can be controlled in **Aux. TC/MFC**. Specify the Number TC used in the system. Name the TC in Temp. Cont. Channel Names. Temp. Standby Set Point (C) is the set temperature of the controller when the recipe is not running. Under Temp. Ramp/Soak the temperature of each channel can be ramped or soaked. If “Soak at” is selected, the soaking temperature is specified in Temp. Set Point (C). If “Ramp to” is selected, the final temperature is specified in Temp. Set Point (C) and the Ramp Rate (C/min) is specified. The gas flow of up to 8 channels is also controlled in this tab under in this tab.

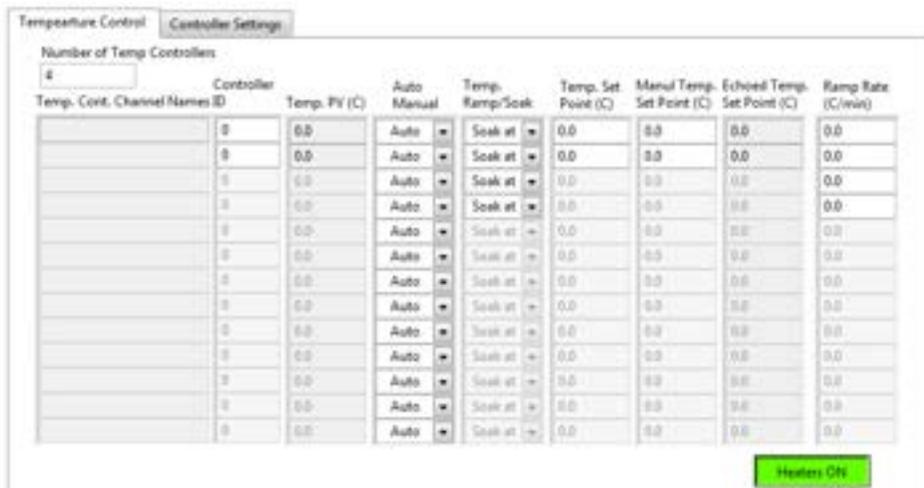


Figure 9 Temperature control

- To leave the editor just click **Quit Editor**. If you have not saved your edits you will be asked to do so. You must click **Save Recipe** button to save the file before any changes will be effective in [Run Arraystat].

Recipe Editor Example – Battery Cycling with Voltage Target

To use a selected Voltage Target to initiate current reversal through an automated step change, knowledge of the standard cell potential and voltage limits of the storage device is required. This example uses an AA nickel metal hydride (NiMH) battery. The standard potential is 1.2 volts. The battery may be charged up to 1.6 volts. NiMH may be discharged to about 0.9 volts and typically loses 2% of charge per day. The mAhr rating of the NiMH battery is about 1,000 mAhr. The positive terminal of the battery is connected to the Working electrode and both the Counter and Reference electrodes are connected to the negative battery terminal.

1. First, create a new recipe file and click **Edit Recipe**. Create an Open Circuit step as the first step. Set Control Type to “Voltage”, and Signal Type (Ramp/Soak) as “OCV”. Use the standard potential of the battery, here "1.2 V", as the Standby Potential (V). Enter a value appropriate for the internal capacitance of the cell into Total Duration of Current Step. This might be 5 times the product of the capacitance and resistance of the cell (5RC).
2. Save your recipe.
3. Insert an OCV step. Edit the step to discharge the battery to 1.1 V at 0.05 Amps. With the electrode connections and battery in this example, discharging entails setting the direction of the current negative (-0.05 A). Set Control Type to "Current" and Signal Type (Ramp/Soak) to "Soak at". Enter “-0.05” to Set/Final Current (A). Set Control Mode to "Battery". Enter the Battery Voltage Target (V), here "1.1" V. Set the Comparator Mode to end the step when the voltage is "< or = V" where V is the Battery Voltage Target (V). The Total Duration of Current Step entry field is not visible. This is because the Battery Voltage Target (V) provides feedback control, with the time for current reversal determined only by the state of the battery.
4. In the [**Recipe Editor**] insert two more steps. Go back to step 3 and change this to an OCV step with the same duration as OCV step 1. Then, go to step 4 and edit this to create a charging step. Set Control Type to “Current” and Signal Type (Ramp/Soak) as “Soak at”. Enter the current "0.05" into Set/Final Current (A). Since this is a charge step the current is positive "0.05" A. Set Control Mode to “Battery”. Enter the Battery Voltage Target (V), here "1.4" V. Set the appropriate Comparator Mode. During charging, the potential increases with time. End this step when the voltage is greater than or equal to V where V is the Battery Voltage Target (V). Therefore, the appropriate Comparator Mode is "> or = V".
5. Save your recipe.
6. If desired, create a loop of the entire cycle for long term battery performance testing. *Note: More than one loop can exist in a recipe but loops cannot be embedded.* While at the last step of loop creation (here step #4), enter the step # to loop back to (here step #"1") into Loop To Step #. Enter the desired number of

loops, here "3", into # Loop Rpts. Activate looping by placing a "check mark" in Loop Enabled.

7. Save your Recipe.
8. Make sure the recipe (path) is loaded into [**Start Screen**] Recipe File Name. Click **Run Arraystat.**

Running ArrayPGstat

Warning: It is strongly recommended that the “Cell Enable Switch” be set to the OFF state before loading and running a recipe. “Cell Enable Switch” must be set to the ON state before running the recipe.

After editing a recipe for use, load it into the [Start Screen]. Click **Run Arraystat** to open the [Run Arraystat].

1. The Run Screen will now appear as shown in the following figure.

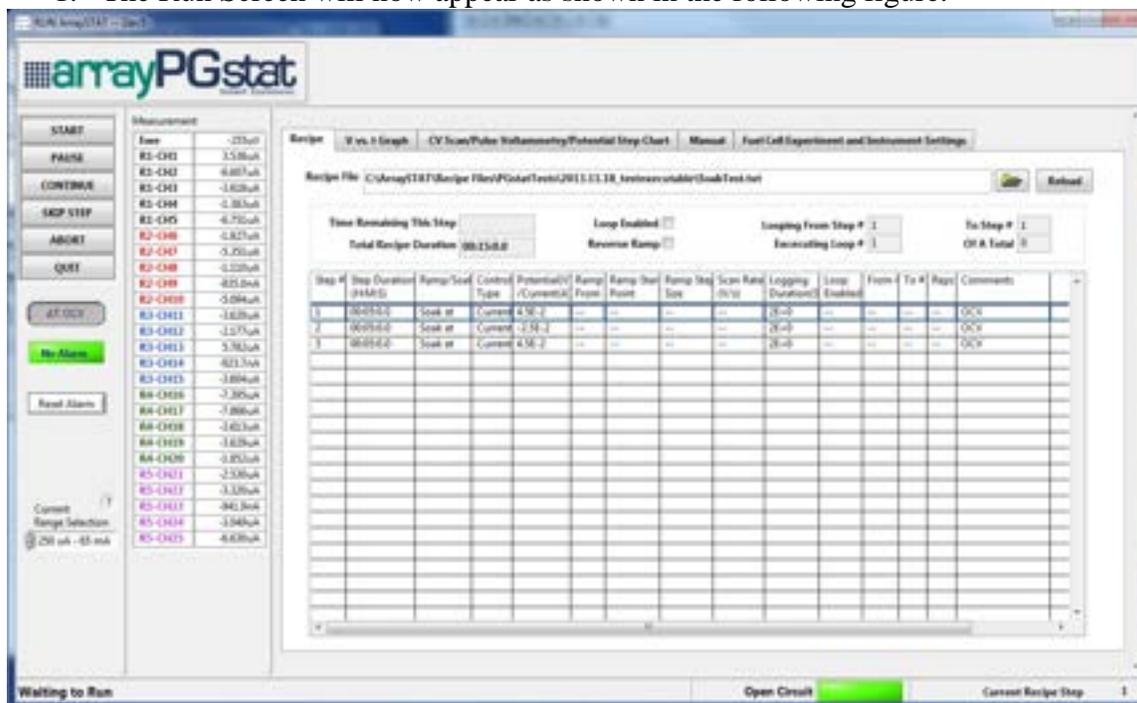


Figure 10 Run ArrayPGstat Screen

2. Recipe files can be loaded directly by clicking on the folder symbol  next to the **Recipe File** entry field.
3. If a recipe file of the same name as that presently loaded in the [Run Screen] has been edited, it must be reloaded by clicking **Reload** for the changes to be loaded.
4. Current Range Selection has two range options. Select the one that best fits the expected current output. Selecting the wrong current range can affect the data collection.
5. Clicking **START** runs the loaded recipe. A window appears asking to save the data. If the user chooses to create a data file, the default file name is the present date and time. Users may change the file name and the save path from the default. The file created is a *.csv comma separated file.

6. Clicking **PAUSE** will pause the execution of the recipe and hold your recipe at its present state and defer writing to your data file until the **CONTINUE** button is pressed.
7. Users may then click **Manual** to view the recipe override controls. Pressing **Output Auto/Manual** disengages the ArrayPGstat from the recipe and allows users to select **Control Type** as either “Voltage” or “Current” and enter a specified potential/current into **Manual Potential/Current**. Each cell can be turned on/off by clicking the appropriate circles in the **Cell Switch** space. If on, the circles will be green. If off, they will be grey. If the user plans to **CONTINUE** with the recipe, he/she must go back to **Auto** position of the **Output Auto/Manual** control.

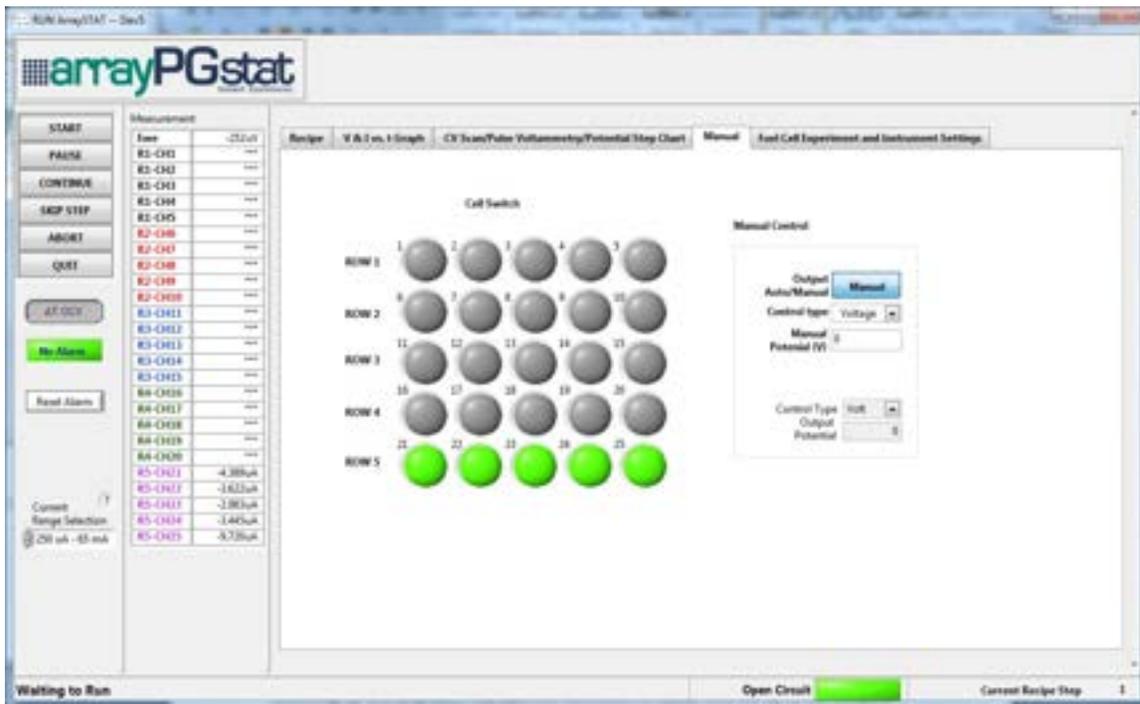


Figure 8 ArrayPGstat manual control

8. Clicking **SKIP STEP** will advance to next step of a running recipe. If no recipe is running or it is aborted, a window will prompt up to ask users to enter the desired step for the program. When **START** is pressed, it will jump from the present step to that step and begin execution.

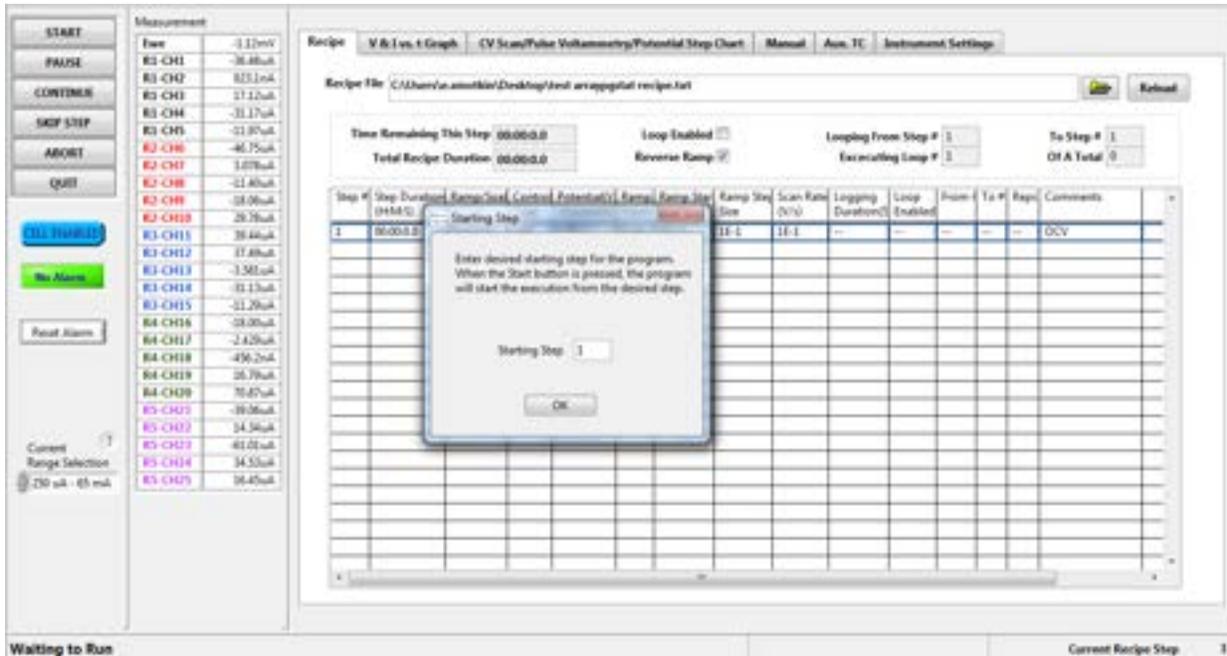


Figure 11 Skip step window

- The **Instrument Settings** tab allows users to set a few features. First, if Cell Disable at end is selected, the cell will disable at the end of the recipe. If Cell Disable at end is NOT selected, the cell potential/current will go to the value specified in Standby Potential/Current (depending on which Control Type is selected).

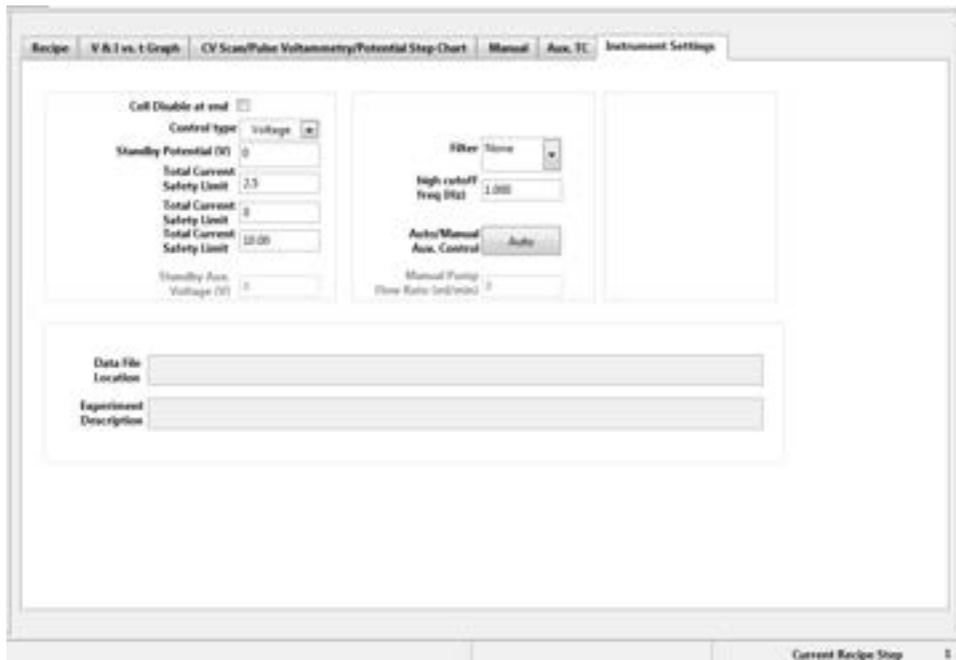


Figure 12 Instrument settings

10. **ABORT** quits the running recipe. A window asks to go to **OCV** or **Standby**. **OCV** sets the cell to the open circuit voltage. **Standby** sets the cell to the potential specified in Standby Potential.



Figure 13 Choose an ABORT mode

11. If the recipe runs to completion, the user will be indicated when the file is closed.

IV Curve

Click the **IV Curve** on the [Start Screen] to open the [IV Curve] window. Load a file into the data display by browsing through the file browser window at the bottom [IV Curve]. The **Select Channels** pane allows selection of specific channels. After selecting the channel(s), specific steps in the recipe can be selected in the **Select Step** pane. More than one channel can be viewed at the same time. In the file browser window at the bottom of [IV Curve], find the first file of interest. Click **ADD FILE**. Find the second file of interest and click **ADD FILE**. Once all the files have been added, click **File Selected**. This will change the browser window to show only the added files. Now the channels and steps can be viewed and added from the selected files. Click one of the added files and select a channel. Click **ADD CHART** to add this to the data chart. This can be repeated for another channel from the same file or the other files. The added channels will be displayed in the column on the right of [IV Curve] with a box indicating the line style. Line styles can be changed by clicking on this indicator. **CLEAR CHART** (located under Plot in the top of the window) removes the data from the chart. **REMOVE FILE** removes the selected data file from the file list.

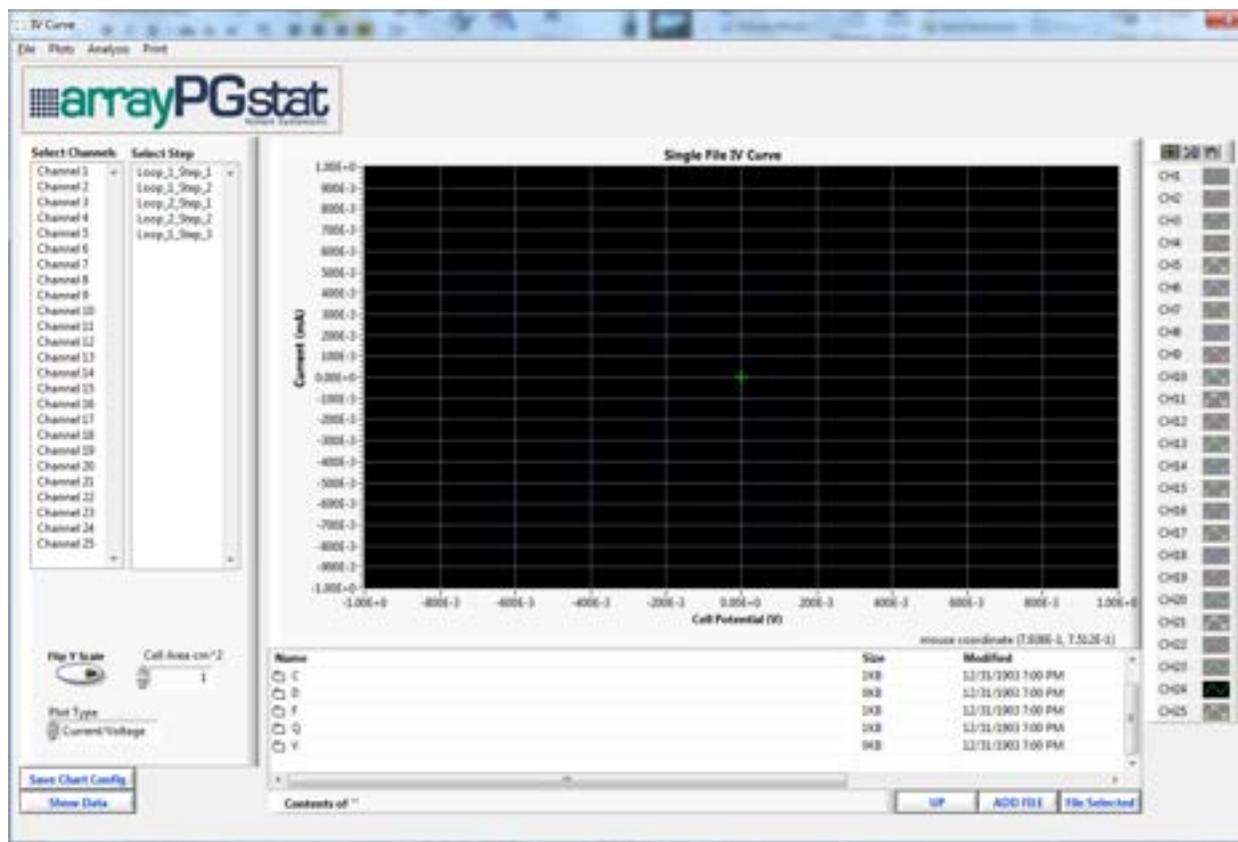


Figure 14 IV Curve

