Precision Voltammetry System
For Ussing Chamber or Three-Electrode Use
with Excel® Template for Control and Data Handling

Lawson Labs, Inc.
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Stack of three Precision Voltammetry systems

INTRODUCTION

Our ultra-high impedance electrode interfacing hardware and software are here joined to a precision current source-sink to enable flexible voltammetry. With a four-electrode system, current and voltage are completely independent. With three electrodes, behavior is still close to ideal. Excel® handles the control of the system. Voltage can be set to a desired point, or current can be set. In either voltage or current control mode the setpoint is entirely programmable. Linear ramp, stepped ramp, alternate polarity steps, cyclic controls, or other waveform control can be done in either voltage or current mode. The voltage range is +/- 5 volts and the current injection range is +/-100 uA. Current is resolved to the nanoamp and voltage is resolved to within a few microvolts.

The Excel® template provided can be run in manual mode or the voltage or current can be controlled automatically. Up to ten systems can run concurrently in one workbook. The flexibility and power of Excel® enable customization without the need for programming. The user can modify the spreadsheet template for their desired application, or, changes can be requested from Lawson Labs. In either event, generous support is provided to get started

A) Software Installation

On a computer with Excel® 2012 or newer installed, make a new folder for your project. Unzip the software archive and place the files in the new the folder. You can rename the Voltammetry.xlsm file as desired.

Make a backup copy of the spreadsheet before continuing. Run the .xlsm file to begin. It should invoke Excel® when starting.

B) Connections

The Voltammetry Interface is built around a customized Model 302 24-Bit Data Acquisition System. Model 302 documentation regarding generalized analog inputs and outputs applies here also.

The four electrodes connecting to an Ussing chamber will be referred to here as Vin plus and minus, and Iout plus and minus. Iout minus is connected to system ground at the interface. (See the Block Diagram, Section G.) For three-electrode systems, Vin minus and Iout minus connect to the common electrode. The interface is optically isolated from the host computer. At the least, the chassis post of the interface should be connected to an earth ground. The optimum connection for system ground should be determined experimentally. It may or may not prefer to connect to earth ground. That choice will depend on ambient noise, and any other galvanic connections to the apparatus. A ground switch is provided for convenience. Start with the switch in the SAFE position.

Iout can be set over the range of +/-100 uA. If the impedance of the output electrodes is much above 50 Kohms, the extremes of the current range will be reduced. As the impedance of the system falls below 50 Kohms, the maximum voltage obtainable for the set point will fall below the 5 volt maximum, following Ohm's Law. The 100 uA limit for the current output will then be the limiting factor.

The electrodes connect to the centers of the BNC connectors. The outer ring in all cases is connected to the chassis ground. The grounding switch has three positions DIF, SAFE, and COM. For DIF, no internal connection is made between system ground and chassis ground. For SAFE, a 10 Kohm internal connection is made. In the Common position, system ground and chassis ground are directly connected.

For now, the interfaces use a repurposed Model EMF6 case, so the labeling is not ideal. Vin+ is labeled "1", Vin- is labeled "2", Iout+ is labeled "5", and Iout- is labeled "6".

Otherwise the connections are the same as the standard Model 302. The power supply connects at the barrel connector and the USB cable connects the interface to the host computer. The host computer should be on line when the Interface is first plugged in so that it will automatically find and install the correct device driver.

When not connected to electrodes for extended periods, and for transport or storage, the voltage inputs should be protected by terminators. These are placed on the two left-hand BNC connections with the ground switch in the SAFE or COM position. The terminators then ensure that static charge does not build on the inputs. In extreme cases, static charge can cause damage to the input buffers.

C) Operation

The spreadsheet has some areas intended for general purpose use. In some cases, we require certain settings for Ussing chamber operation, and in others, flexibility is left available. We color code cell background to identify the category. Yellow background is for cells that require user input. These include an entry for the Device ID number, the control loop gain, the voltage setpoint (+/-5 volts maximum), and the amount of averaging. It may be desirable to change the update interval from 1 second, so that cell is also yellow. Tan cells are active inputs and can be changed, but are normally set once at initial configuration. These include the internal device rate (60 Hz for line frequency rejection, set to 50Hz for 50Hz line frequency), Block Average to reduce the amount of data, the number of channels to scan, 3 for the voltammetry system, and the scan type, normally 6 for a multichannel scan with digital inputs enabled and self-calibration. Pink background cells are calibration values- see below.

Do not move cells around because some cells are referred to by absolute addressing. That may change in the future.

Start with a single device to begin. Enter the device ID number on Sheet 1 in the upper left. Click Initialize in the control box. If the control box is hidden, click the button labeled show/hide device control. Wait a few seconds for the "Ready" message in the Device Control box. Once initialized, you can start a scan. Do not attempt to change cell values while scanning. A Do not Edit message in the Device Control box reminds you whether editing is allowed. If you need to edit the spreadsheet, stop all scans first, then edit, then restart the scan or scans. When scanning, data appears in columns, starting with the scan number in column H, followed by each scan's data, one scan per row.

Once scanning, in mode 1 the control loop will attempt to servo the current to obtain the voltage setpoint found in cell A17. In mode 2 the control loop will attempt to servo the current to obtain the current setpoint in cell A17. In mode 2, the Gain (cell A14) should be set to about 0.028 for a one second control interval, or about 0.006 for a 0.1 second control interval. In mode 1 the Gain setting is determined by the user. Gain depends on impedances. If the voltage input oscillates, the Gain should be reduced. If the voltage input takes too long to settle, the Gain should be increased. If the membrane resistance is too high, the voltage setpoint will not be obtainable. In that case, the current output will rise to one or the other limit, depending on polarity.

You can hide or retrieve the Device Control box by clicking the button at the top of the spreadsheet. You may need to click twice to view a hidden Control box.

Channel 1 is the voltage input, shown in column I. Columns J and K show the measured current source and sink. The total of the scaled source and sink currents is the current applied to the I+ electrode. That current shows in column L, and the most recent value also appears in cell D27. For convenience, row 31 shows the most recent scan data in a fixed location at the left margin.

Each Voltammetry interface has calibrations written on the bottom of the base. We supply an offset value for Channel 1, and gain values for the source and sink current outputs. We have left provision for source and sink offsets, but have not found them necessary, so leave them at zero for now. The channel 1 offset value is applied to the reading in cell A31 and to the voltage readings in column I.

A sample chart sheet is included for each device. Use the Excel® charting capabilities to obtain the presentation you want. The device sheets must be named Sheet1, Sheet2, etc. The chart sheets can be named at will, but must follow the device sheets in the tabs. The template provided includes extra sheets and charts for additional devices by way of example. Extra tabs can be deleted if not needed. Tabs can be added later, as described in Section D, below.

The controls in the Device Control box are normally used when initialized but not scanning. Digital I/O is active, but not connected to anything in this application. If the control interval is set to zero (cell E8), analog outputs may be controlled manually during scanning from the Device Control box. The control loop will not act whenever the DAC interval (for Analog Outputs) is set to zero.

The DAC settings at the start of a scan will be whatever they were at the end of the last scan. If you want particular starting conditions, set the DACs manually before starting a scan.

There is one other control in the Device Control box that is active during scanning. The Pause button stops the data from being written into the spreadsheet, but does not stop the scan or the control loop. Pause is useful for reducing the volume of data when periods of time of no experimental interest are included in a test sequence. Pause applies on a device by device basis. If, for example, you Pause while a control ramp is running, the graph will stop, and will show a discontinuity when the scan is unpaused. Remember that spreadsheet editing is not allowed when paused.

Editing when not allowed will stop all scans immediately. The change will be noted after a wait of 30 seconds. It should be possible to restart by clicking start/stop scan once on each stopped device sheet. (Remember to save any data first, since restart clears the previous data.) The status should then show as scanning. If you cannot restart all the scans, stop any running scans and click Initialize. You may need to repeat that step. Worst case, you may need to quit and restart the software before continuing.

D) Additional Devices

A sheet can be copied to add a device. First, stop any scanning devices. Right click on the tab, Select move or copy, check the create a copy box, and chose the location. The Device tabs must be in the first 1 to 10 locations, and must be labeled Sheet1, Sheet2, etc. (This limitation is an expedient that could be lifted with more programming.) When prompted, yes, you wish to copy everything over. Then, enter the new device ID and rename the second sheet to Sheet2 or third sheet to Sheet3 or fourth sheet to Sheet4 (case sensitive), etc. You will want to manually enter any calibrations for the added device.

Initialize the new device. All active devices will be initialized together, then, set up your added experiment. Thereafter, scans are started or stopped independently on the various sheets. Still, any spreadsheet editing, anywhere, will stop all running scans, so be sure to set everything up in advance before beginning any long-running tests. If a scan is running on another sheet, you will see an "editing not allowed" notice in the Device Control box. That notice is a warning not to edit or you will stop all other scans in progress. If you do edit and stop all scans, you will see that the status of stopped devices is still shown as "Scanning". Remember, to recover, click start/stop in the Device Control box to first stop, and then again to restart the scan.

To temporarily disable a device, enter 0 for the device ID number. To re-enable, enter the device ID number and click Initialize.

The limit of how many devices can be run at once depends on the power of the computer and the various scanning rates. If the computer begins to fall behind, the control loop will suffer. For now, there is an arbitrary maximum limit of ten devices.

Analog outputs may be changed manually whenever a device is initialized, even during scanning. DAC1 controls current sourcing. It is scaled for 5 volts equals 100 uA, or 20 uA per volt. DAC2 controls current sinking. It is scaled for 5 volts equals -100 uA. DAC3 is for fine-tuning of the current. It is scaled for 5 volts equals -2 uA. If you start DAC3 at half scale, 2.5 volts, DAC3 then provides +/- 1 uA fine tuning. Manual changes will disturb the control loop, if it is running at the same time. The most recent DAC output values show in Cells A32-34, whether or not those values were set manually.

E) Tuning the Control Loop

The control has two modes, 1 for voltage and 2 for current. Mode 0 is for manual control.

Local Averaging can be set from 1 to 128. Increased averaging will provide steadier readings, but will slow down the response of the servo loop. Increased Local Averaging reduces the optimum gain because of the delay incurred. For best dynamic loop behavior, set the Local Average to one.
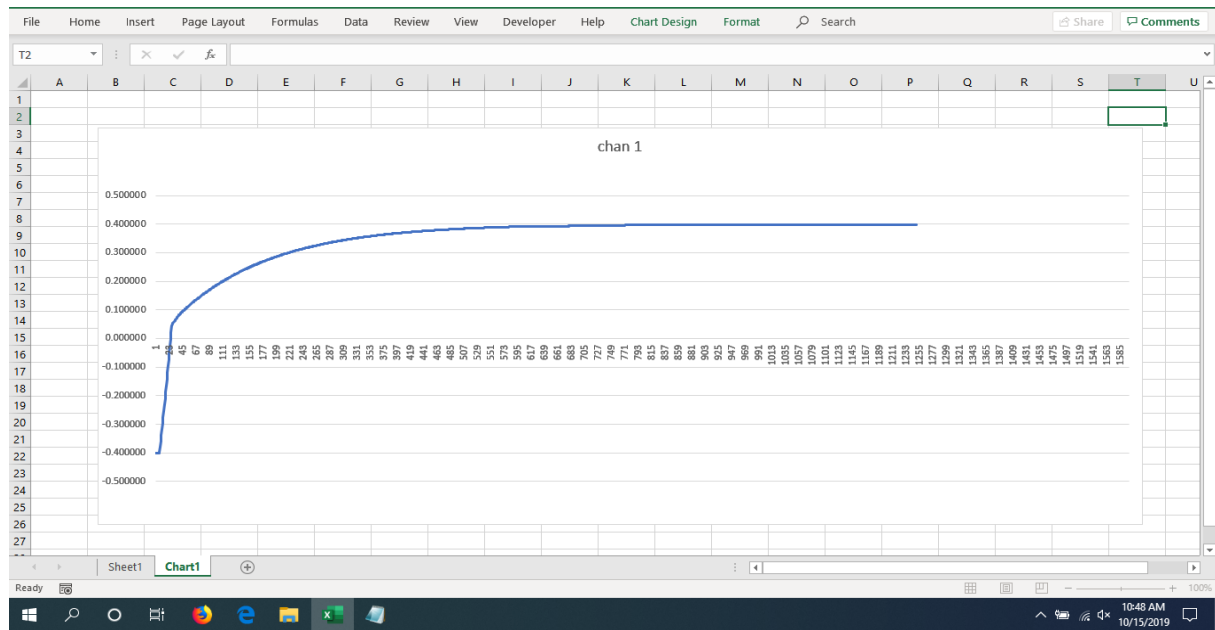
The control mode is set in cell G6.
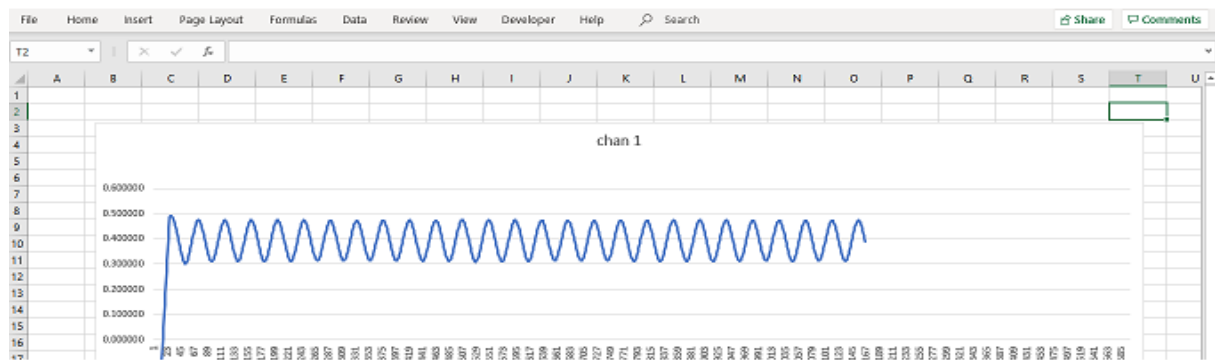
Voltage control, mode 1

The optimal gain setting depends on the impedance of the system. As a place to begin, if the resistance is on the order of 500K, use a gain of 0.1. For 50K, use a gain of 1, for 5K, use a gain of 10, and for 500 ohms, use a gain of 100. Excess gain will result in oscillation. A little extra gain causes a damped oscillation that will eventually settle out. A lot of extra gain will cause an oscillation that will continue indefinitely. Too little gain will cause the output to be slow to reach the setpoint, but is otherwise fine.

As averaging is increased, the gain is reduced. Gcor on the spreadsheet shows that gain reduction.

The screenshots to follow show characteristic plots for aid in setting the voltage mode gain.
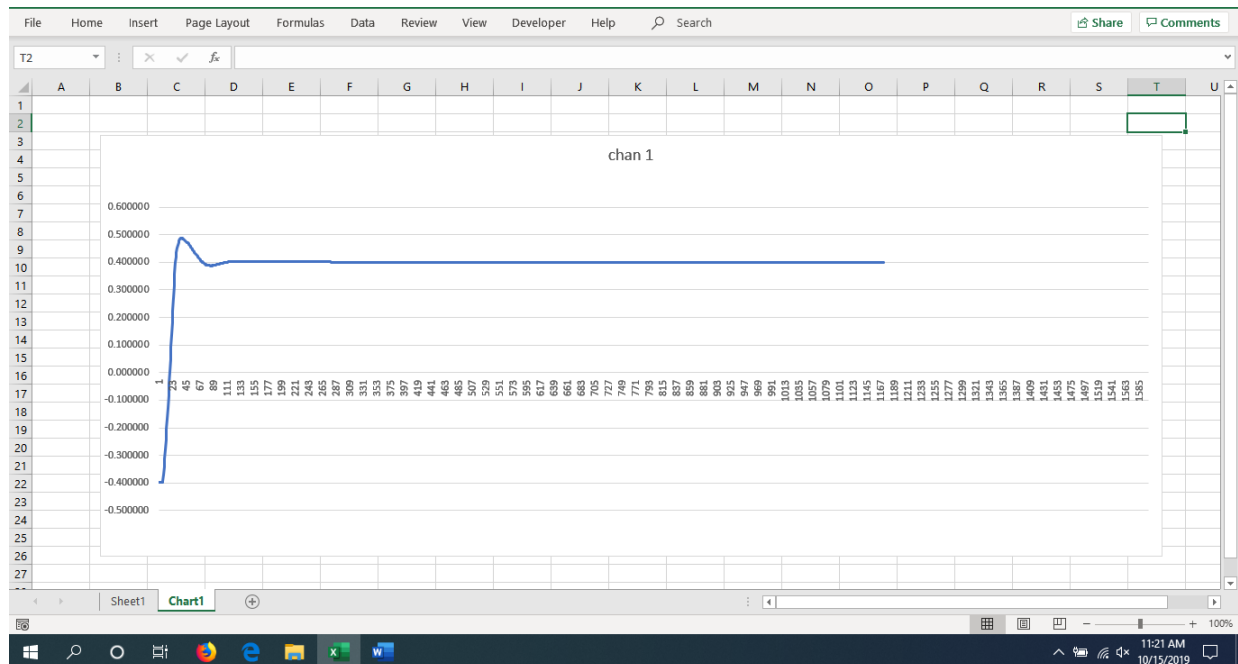
Too little gain (Gain =1, at 5 Kohms) means slow settling

Too much gain (Gain = 40). At a Gain of 35, the oscillation will fade out.

Fastest settling is obtained when the gain is set for slight overshoot. If overshoot is followed by undershoot, marginally reduce the gain.

Gain is close to optimal (Gain = 10)

If you have difficulty finding a good gain setting, try increasing the control interval in cell E8. Longer control intervals have much the same effect as reducing the gain, which can bring higher stability.

Maximum and minimum current limits are provided to protect fragile membranes. Cells E16 & F16 are the current limits in uA. The Max must be positive and the Min is always negative, with or without an explicit minus sign. If the voltage setpoint is not attainable within those limits, the current will sit at the limit. These limits apply only in voltage control, mode 1. Setting both limits at 100 allows operation over the full range of the system. The limits are accurate to within 2 uA because the trim DAC, DAC3, is not clamped.

Current control, mode 2

Mode 2 controls the current, and the voltage follows. In Mode 2, the optimal gain setting is usually around 0.028 for a one second control interval, or about 0.006 for a 0.1 second control interval.

The set point can be static or dynamic. Two sample formulas are included below the set point cell (A17), a ramp (A19) and a stepped waveform (A20). Move one of these formulas, or one of your own, into the set point cell to invoke the dynamic behavior. These formulas are based on the scan number, so scanning rate changes will, for example, change the slope of a ramp.
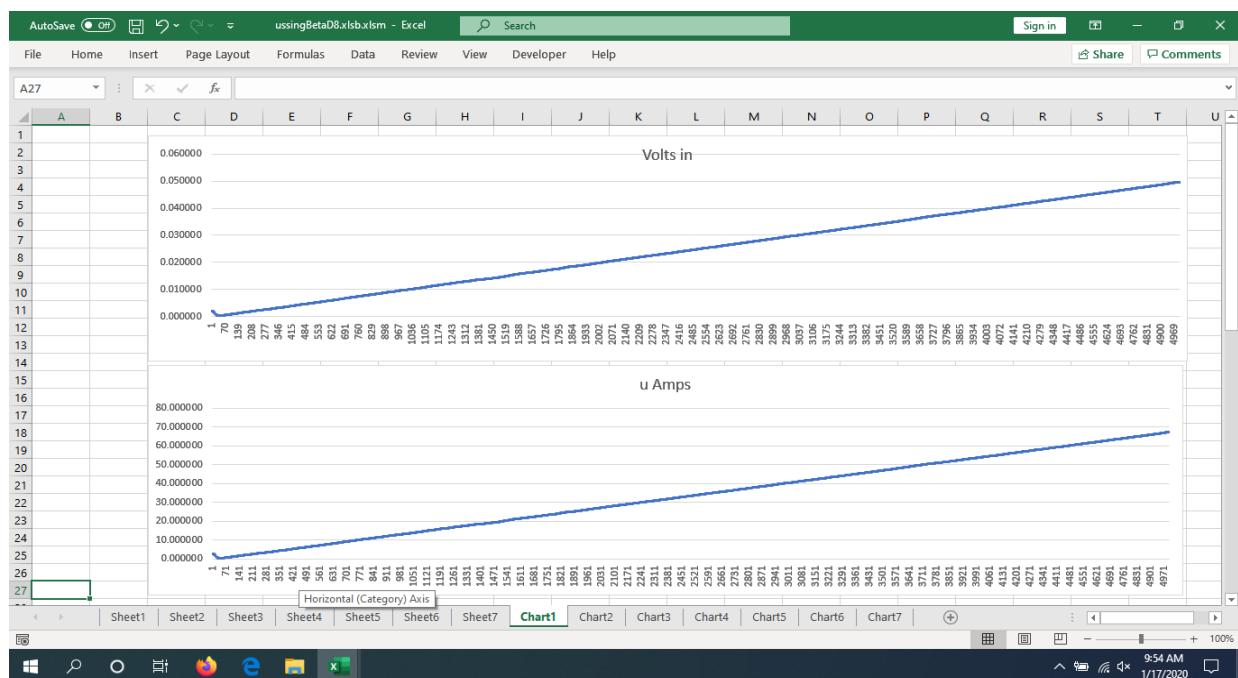
Tune should be 0 when you use a dynamic set point, or when in Mode 2. Entering 0 in Cell E19 disables self-tuning, and 1 allows most aggressive tuning. There is a continuous scale between for fractional values. For now, self-tuning is only appropriate under near-static conditions. The Max and Min Gain cells, E & F14, constrain the possible gain settings. These are needed for auto-tune, but do apply at any time.
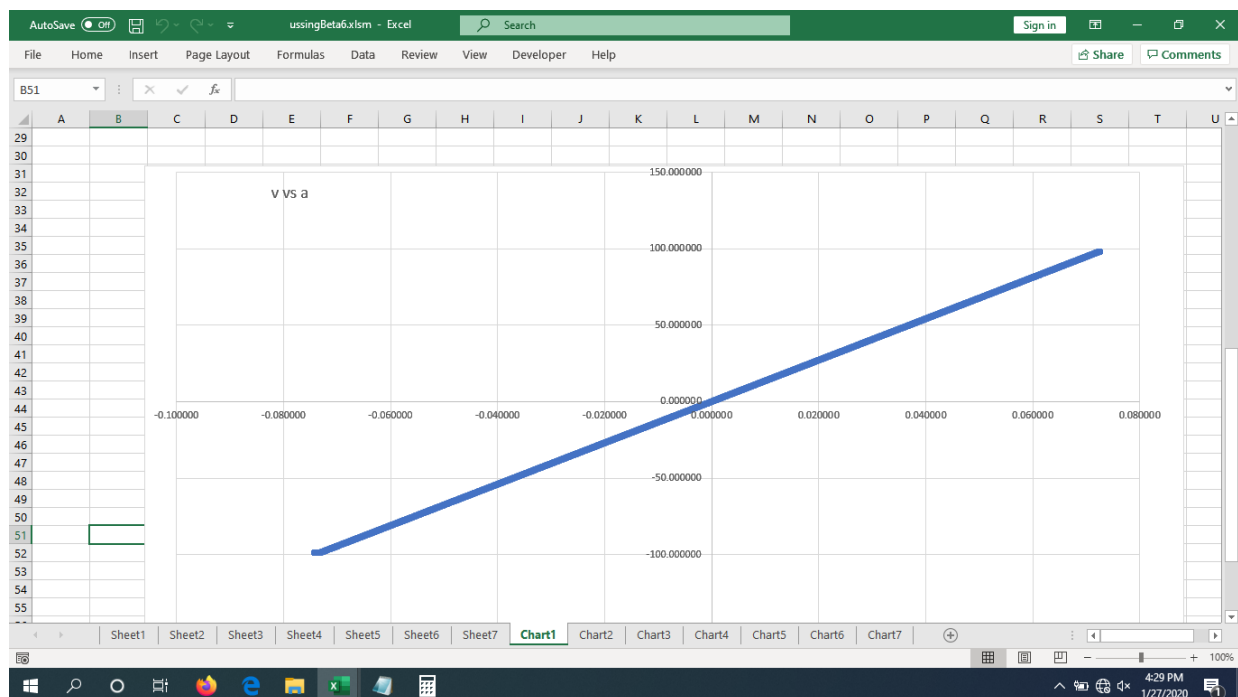
F) Charting

Three sample charts are included on the chart sheets, Chart1, Chart2, Chart3, etc. The first is voltage (in volts) vs time. Next is current (in uA) vs time. These are provided by way of example only. Use your Excel® skills to produce publication quality graphing.

The third chart is below the voltage and current charts. It shows voltage vs current. With constant resistance, all points will fall on a straight line dictated by Ohms law. If the system runs out of range, you will see the line bend at the positive or negative extreme. Otherwise, non-linearities in the voltage vs current plot represent changes in the electrochemistry.
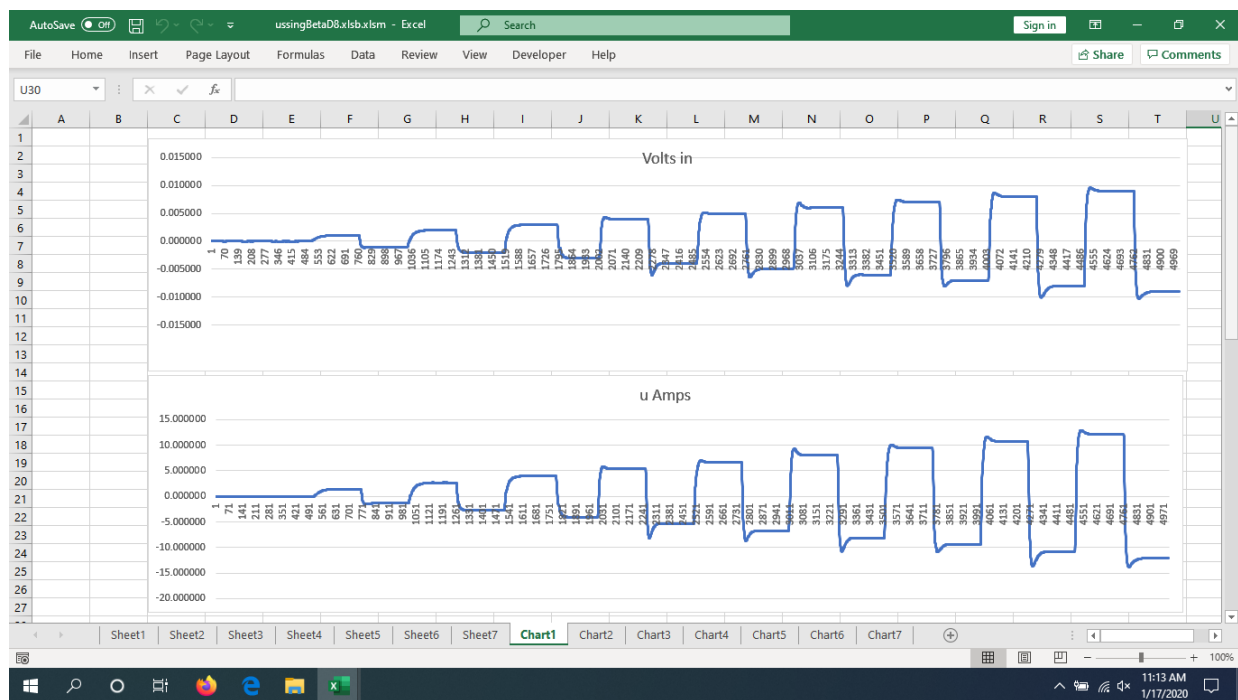
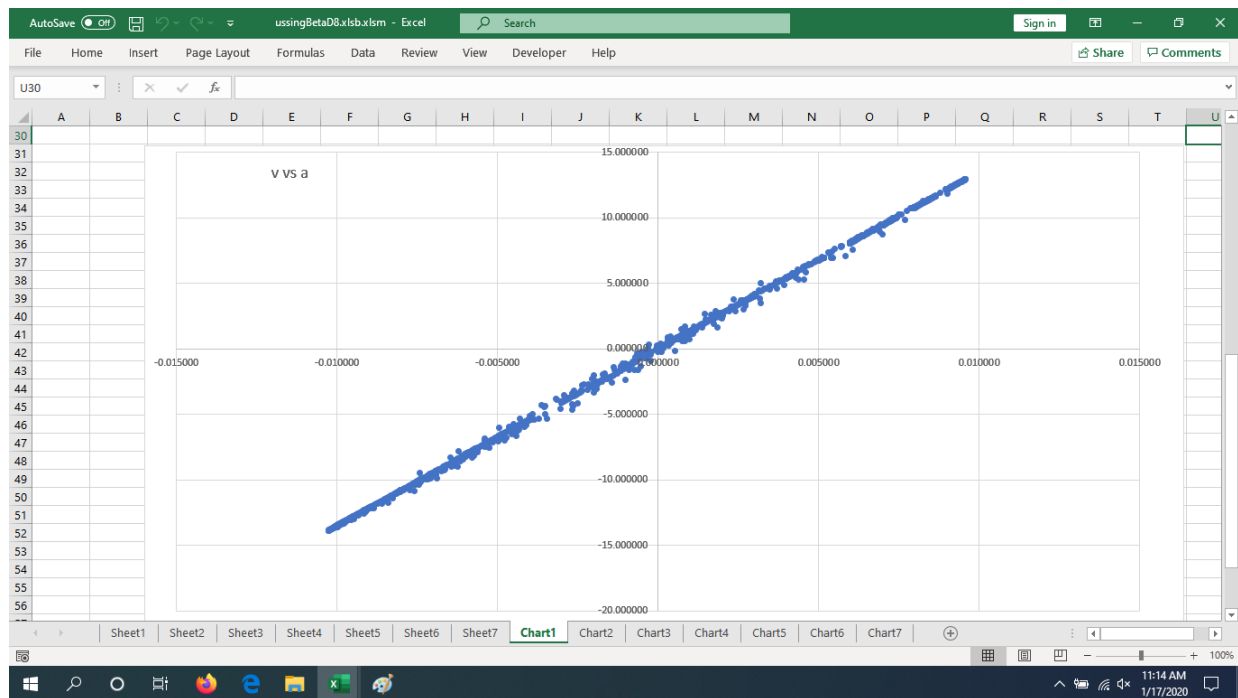Here are sample plots for various setpoint formulas.

Mode 1 or 2 Linear ramp, starting at 0

Mode 1 Linear Ramp scatter plot with a fixed resistor in place of a membrane

Mode 1 increasing step with alternate polarity

Scatter plot from step function, above. Note slight non-ideality due to fast step edges.

G) Other Displayed Information and User Input Details

During scanning, cell C3 shows the number of scans being processed. Because scans are buffered, if the spreadsheet falls behind the data, that number will rise. If the spreadsheet is temporarily engaged in some other task, the C3 number may flick up when you return to the sheet. That is normal. If, on the other hand, you see larger in-process numbers cycling on a regular basis, that means you are falling behind. The control loop will not regulate well if you are behind. Either reduce the scanning rate, or reduce the calculation load on the computer.

Normally, the internal device data rate, cell B5, is set to 50 or 60 Hz, to match the line frequency. If you want fastest response, you can change the internal device data rate to 800 with a block average (Cell B4) of 4. That will provide 10 scans per second, and good rejection of both 50 and 60 Hz noise. For 5 scans per second, use a block average of 8. Using other rates and block averages, with a few exceptions, will destroy the system's rejection of line frequency noise. The scan rate is shown in cell B6. Note that single channel scanning is supported, but only in manual mode. The number of channels, cell B8, should be set to one if single channel scanning is selected in cell B14.

The DAC interval, cell E8, can be set in the range of 0.1 to tens of seconds. The loop will never be updated more frequently than the scan interval, so if you are running at line frequency, the smallest update interval would be 0.33 seconds. Normally, 1 second is fine for 50 and60 Hz, and 0.1 for 800 Hz with a block average of 4. Remember that changing the control update interval may change the optimum gain.

Block Average reduces the scanning rate. Local Average, cell B17, does not. It is simply a running average of the last n points. If your system is dynamic, and you are using control mode 1 or 2, we recommend Local Avg to be left at 1 because local averaging will slow the loop response.

Note: The Max and Min current limits apply only in mode 1.

H) Troubleshooting

If you find the Start scan button unresponsive, check first that the focus is not on an edited cell. Press enter, or select another cell in that case. Next, check the messaging in the Device Control box for clues.

This software is being continually improved. There may be occasional issues. For example, cell locking during scanning is not implemented. It is possible to crash Excel® if worksheets are edited during scanning. If you have difficulty restarting after a crash, unplug each USB cable for a second. That should allow Excel® to fully unload prior to a new start. If not, look in the task manager for Excel®, and End Task for any Excel® remnant still loaded.

If you should inadvertently have two copies of the spreadsheet open at once, nonsensical behavior will result. In that case, close all Excel®, and check the task manager, as above, for any Excel® remnants to unload.

Keep a backup copy of your work, and do not rely solely on the Excel® document recovery features.

In order to run long-term tests, you will need to turn off any power saving features that would stop the scanning. In Win10, you need to specifically turn off power saving on the USB ports. You should also turn off automatic updates.

Installation issues are covered extensively at www.lawsonlabs.com.
Call or email Lawson Labs for help with noise or stability issues, 610 725-8800, or
lawsnlab@lawsonlabs.com.

I) Block Diagram

V in +

V in -

I out +
(Bipolar)

I out -

Lawson Labs
Model 302
with Precision
Current Output
Option

USB TO HOST

+

POWER IN

-

SAFE

DIF   Ground Switch

COM

Chassis Ground