NLEIS Starting Guide

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Hardware Component

This LabView program is made specifically to work with:

1. Solartron 1287 Potentiostat/Galvanostat
2. PXI 5421 AWG (Arbitrary Waveform Generator)
3. PXI 5922 Oscilloscope

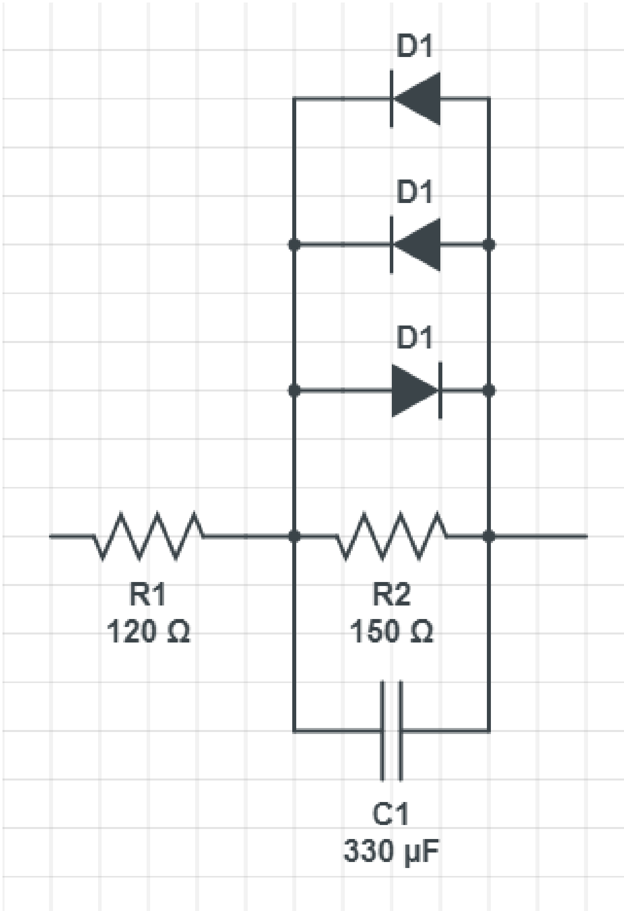
Using different potentiostat or PXI modules likely require modification of the source code.

Make sure to establish the connection between your computer where the LabView code will be run on with the hardware.

NLEIS Experiment Workflow

1. Workflow diagram to conduct a NLEIS experiment:

From this point on, we will go into details how to perform these steps along with an example of our in-house diode test circuit. The diode circuit test circuit is shown below:



The diodes are of type VFT2060C 14176

EIS Experiment

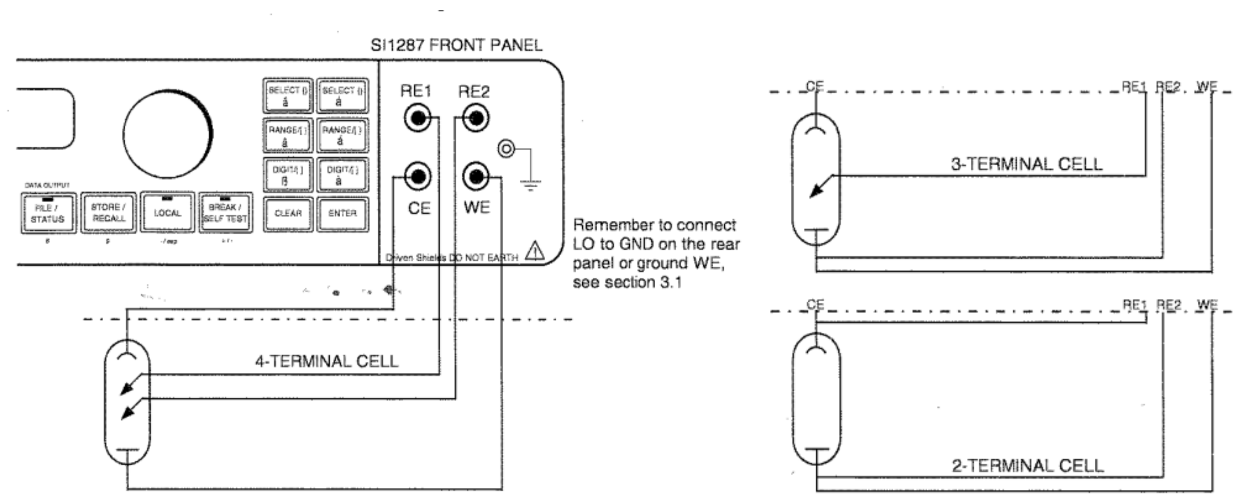
Before making the NLEIS experiment, you need to perform an EIS experiment. This later determines what current perturbation amplitudes you would be using for each perturbation frequency. We typically determine this by performing an EIS experiment and use EIS data with the spreadsheet “Template.xls” to appropriately create a data file of scaling factor for each frequency.

1. EIS Experiment with Solartron 1287 + Solartron 1252 FRA

We use Solartron 1287 and Solartron 1252 FRA to do EIS experiment in our lab. This section describes the bare-bone instructions to make an EIS experiment with Scribner ZPlot & ZView.

Electrical connections.

- *Electrochemical device to Solartron 1287*. Make the following electrical connection from the test model (fuel cell, battery, circuit, etc.) to the Solartron 1287 Potentiostat:



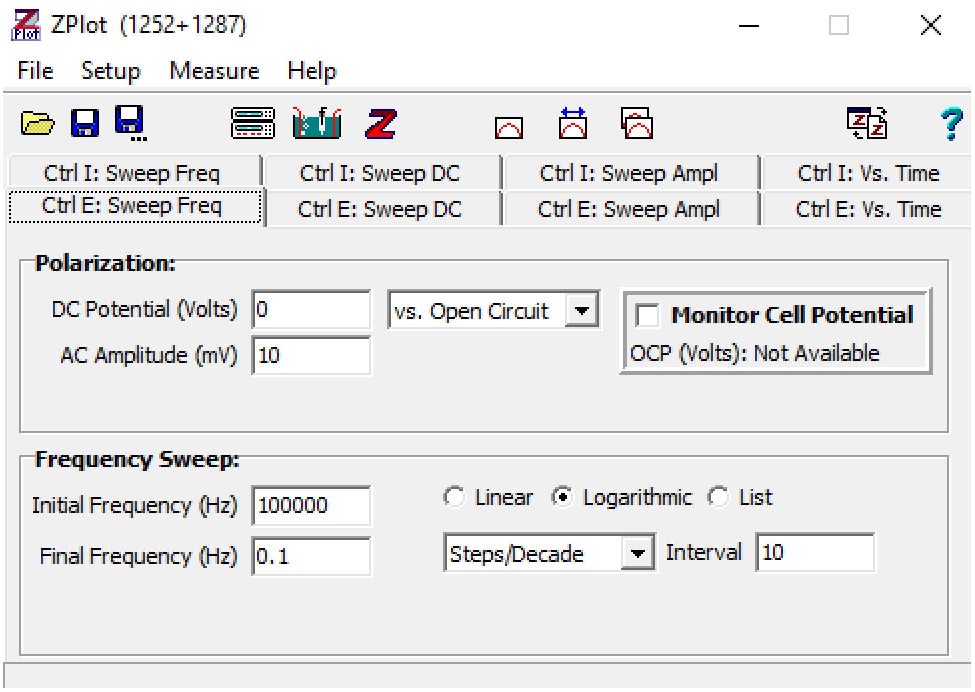
- Solartron 1287 to Solartron 1252.
  - **V OUT** (1287) to **V2 HI** (1252)
  - **I OUT** (1287) to **V1 HI** (1252)
  - **POL IN** (1287) to **GEN OUTPUT** (1252)

GPIB connections.

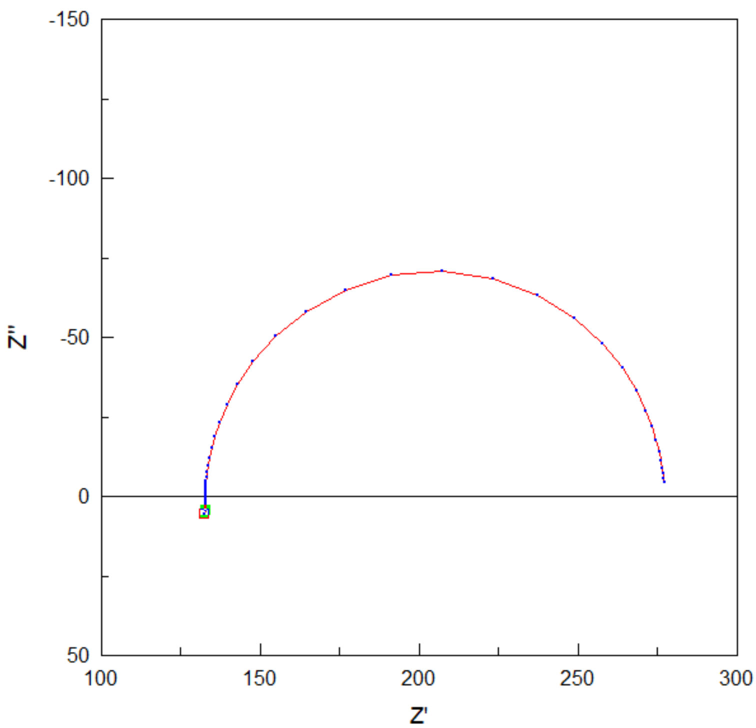
- Connect the Solartron 1287 GPIB gate to Solartron 1252 GPIB gate to the computer GPIB receiver.
- Check the GPIB addresses for each Solartron Instruments by looking at the indicator on the back of the Solartron locates near the GPIB outlet.
- When Scribner ZPlot, make sure to correctly enter the GPIB address of these Solartron Instruments and use the device self-test option on ZPlot software.

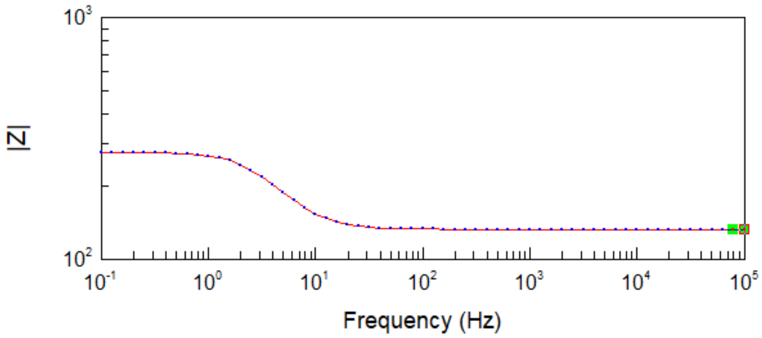
Scribner ZPlot EIS experiment.

We can use the default EIS experiment setup from the program ZPlot to collect a potentiodynamic EIS.

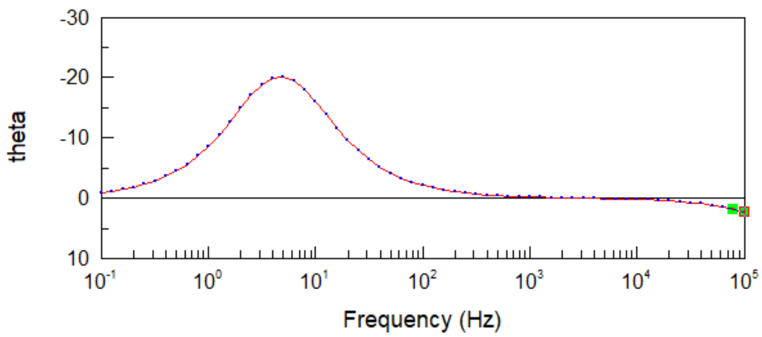


In the Example folder, you should find a EIS data file of a diode-circuit. Diode has a non-linear voltage-current relationship, which is why we use them to make a test cell for NLEIS experiment. You can pull this up to view in ZView complex plane and bode plot. It would look like this.





10^3
10^2
|Z|
10^{-1} 10^0 10^1 10^2 10^3 10^4 10^5
Frequency (Hz)



-30
-20
-10
0
10
theta
10^{-1} 10^0 10^1 10^2 10^3 10^4 10^5
Frequency (Hz)

Determine Scaling Factor

Use "Template.xls" to determine scaling factor.

Following these steps:

1. Open the Scribner ".z" file or any other type of text file used to save EIS data. Copy the frequency, Z real and Z imaginary into the right columns in "Template.xls".
2. Enter the following information into the EIS Results box (cell K8:K10):
    a. Cell K8: Peak frequency (Fp) (frequency where Z imaginary magnitude is largest). This should also be highlighted with green in the frequency column (column A).
    b. Cell K9: Z real value at the peak frequency (Rtotal at Fp).
    c. Cell K10: the electrode impedance contribution (Rchem) which is the width of the half circle observed in EIS.
3. Select the appropriate Solartron 1287 gain setting (cell K13) (either 1 or 0.01). The polarization input from PXI 5421 will be multiplied by this gain.
4. NLEIS test setting (cell K16:K22) – this calculate the current perturbation amplitude to run a NLEIS test run before running the full frequency and amplitude spectrum.
    a. Cell K16: enter the smallest voltage perturbation amplitude wanted (should be in the linear behavior region, e.g. 10 mV)
    b. Cell K17: enter the appropriate selection of Solartron internal resistor* (footnote).
    c. Cell K20: enter the largest voltage perturbation amplitude wanted (should be in the non-linear region, e.g. 150 mV or 200 mV)
    d. Cell K21: enter the appropriate selection of Solartron internal resistor* (footnote).
5. This should provide the two test amplitudes in cell K18 & cell K22 respectively to use in NLEIS_Run_PXI.vi program).
6. NLEIS Sweep cell block (K25:K30) to calculate total run time. Enter the internal resistor information and crossover frequencies (if needed) as required.
7. Copy the scaling factor (values only) from Column F into a new txt file for later use.

*Solartron 1287 comes with 0.1, 1, 10, 100, 1k, 10k, 100k or 1M Ω resistors. Each has a specific current range (Solartron manual). Select the resistor that compatible with the galvanodynamic NLEIS experiment current range.

For the diode example, you can find in the example folder the saved "Template_filled.xls" and scaling factor text file, named "scaling_factor.txt"

Data from Scribner diode z file – 090921_diode_test_cell_EIS.z

```
090921_diode_test_cell_EIS.z - Notepad
File  Edit  Format  View  Help
    Density:                  0
    Weight:                   0
    Polarity:                 0
    PolarityI:                0
    Corrosion Unit Type:      0
    Reference Type:           0
    Reference Potential:      0
    User-Defined Potential:   0
    Stern-Geary:              26
  End Information:            Cell Information
  Null Configuration:         0,3,0,1,100000,0.1
  Data Points:                61
  Freq(Hz)      Ampl    Bias    Time(Sec)       Z'(a)   Z''(b)  Aux     Err     Range
End Comments
1.000000E+05    1.0000E-02      0.0000E+00      4.380000E+00    1.3246E+02      5.3787E+00      0.0000E+00      0       4
7.943282E+04    1.0000E-02      0.0000E+00      5.130000E+00    1.3249E+02      4.2652E+00      0.0000E+00      0       4
6.309573E+04    1.0000E-02      0.0000E+00      9.130000E+00    1.3261E+02      3.4059E+00      0.0000E+00      0       4
5.011872E+04    1.0000E-02      0.0000E+00      9.870000E+00    1.3270E+02      2.6908E+00      0.0000E+00      0       4
3.981072E+04    1.0000E-02      0.0000E+00      1.062000E+01    1.3281E+02      2.1196E+00      0.0000E+00      0       4
3.162278E+04    1.0000E-02      0.0000E+00      1.137000E+01    1.3288E+02      1.6766E+00      0.0000E+00      0       4
2.511886E+04    1.0000E-02      0.0000E+00      1.212000E+01    1.3287E+02      1.3306E+00      0.0000E+00      0       4
1.995262E+04    1.0000E-02      0.0000E+00      1.287000E+01    1.3290E+02      9.8572E-01      0.0000E+00      0       4
1.584893E+04    1.0000E-02      0.0000E+00      1.362000E+01    1.3276E+02      8.3684E-01      0.0000E+00      0       4
1.258925E+04    1.0000E-02      0.0000E+00      1.437000E+01    1.3277E+02      6.5604E-01      0.0000E+00      0       4
1.000000E+04    1.0000E-02      0.0000E+00      1.512000E+01    1.3285E+02      5.0257E-01      0.0000E+00      0       4
7.943282E+03    1.0000E-02      0.0000E+00      1.587000E+01    1.3290E+02      3.6685E-01      0.0000E+00      0       4
6.309573E+03    1.0000E-02      0.0000E+00      1.662000E+01    1.3285E+02      2.5662E-01      0.0000E+00      0       4
5.011872E+03    1.0000E-02      0.0000E+00      1.735000E+01    1.3276E+02      1.6614E-01      0.0000E+00      0       4
3.981072E+03    1.0000E-02      0.0000E+00      1.813000E+01    1.3273E+02      7.5299E-02      0.0000E+00      0       4
3.162278E+03    1.0000E-02      0.0000E+00      1.888000E+01    1.3264E+02      1.4835E-03      0.0000E+00      0       4
2.511886E+03    1.0000E-02      0.0000E+00      1.959000E+01    1.3263E+02      -7.4737E-02     0.0000E+00      0       4
1.995262E+03    1.0000E-02      0.0000E+00      2.036000E+01    1.3264E+02      -1.5517E-01     0.0000E+00      0       4
1.584893E+03    1.0000E-02      0.0000E+00      2.111000E+01    1.3257E+02      -2.4085E-01     0.0000E+00      0       4
1.258925E+03    1.0000E-02      0.0000E+00      2.184000E+01    1.3255E+02      -3.5284E-01     0.0000E+00      0       4
1.000000E+03    1.0000E-02      0.0000E+00      2.263000E+01    1.3259E+02      -4.5533E-01     0.0000E+00      0       4
7.943282E+02    1.0000E-02      0.0000E+00      2.337000E+01    1.3264E+02      -6.1409E-01     0.0000E+00      0       4
6.309573E+02    1.0000E-02      0.0000E+00      2.585000E+01    1.3255E+02      -7.9416E-01     0.0000E+00      0       4
5.011872E+02    1.0000E-02      0.0000E+00      2.662000E+01    1.3254E+02      -9.8004E-01     0.0000E+00      0       4
3.981072E+02    1.0000E-02      0.0000E+00      2.738000E+01    1.3254E+02      -1.2372E+00     0.0000E+00      0       4
3.162278E+02    1.0000E-02      0.0000E+00      2.810000E+01    1.3259E+02      -1.5314E+00     0.0000E+00      0       4
Ln 20, Col 22    100%    Windows (CRLF)    UTF-8
```

Pasting the frequency range of interest into Template.xls **(Step 1)**. In this example, we remove the high frequency range (>1 kHz) from NLEIS experiment because they are much faster than the main process.

	A	B	C
1	**Frequency (Hz)**	**Re (Z)**	**Im (Z)**
2	1.00E+03	1.46E+02	-0.46419
3	7.94E+02	1.46E+02	-0.60323
4	6.31E+02	1.46E+02	-0.80122
5	5.01E+02	1.46E+02	-0.98556
6	3.98E+02	1.46E+02	-1.2549
7	3.16E+02	1.46E+02	-1.5555
8	2.51E+02	1.46E+02	-1.971
9	2.00E+02	1.46E+02	-2.4918
10	1.58E+02	1.46E+02	-3.1534
11	1.26E+02	1.47E+02	-3.9369
12	1.00E+02	1.47E+02	-4.9309
13	7.94E+01	1.47E+02	-6.1859
14	6.31E+01	1.47E+02	-7.8057
15	5.01E+01	1.47E+02	-9.7055
16	3.98E+01	1.48E+02	-12.16
17	3.16E+01	1.48E+02	-15.191
18	2.51E+01	1.49E+02	-18.914
19	2.00E+01	1.51E+02	-23.453
20	1.58E+01	1.53E+02	-28.937
21	1.26E+01	1.57E+02	-35.387
22	1.00E+01	1.62E+02	-42.72
23	7.94E+00	1.69E+02	-50.643
24	6.31E+00	1.78E+02	-58.495
25	5.01E+00	1.90E+02	-65.276
26	3.98E+00	2.05E+02	-69.809
27	3.16E+00	2.21E+02	-71.087
28	2.51E+00	2.37E+02	-68.832
29	2.00E+00	2.51E+02	-63.509
30	1.58E+00	2.63E+02	-56.3
31	1.26E+00	2.72E+02	-48.312
32	1.00E+00	2.78E+02	-40.512
33	7.94E-01	2.83E+02	-33.325
34	6.31E-01	2.86E+02	-27.217
35	5.01E-01	2.88E+02	-21.987
36	3.98E-01	2.89E+02	-17.694
37	3.16E-01	2.90E+02	-14.241
38	2.51E-01	2.90E+02	-11.321
39	2.00E-01	2.90E+02	-9.015
40	1.58E-01	2.91E+02	-7.2059
41	1.26E-01	2.91E+02	-5.8345
42	1.00E-01	2.91E+02	-4.5941

Entering the EIS result summary box **(Step 2)**

EIS Results	
Fp	3.2
Rtotal at Fp	221.31
Rchem	144.27

Enter Solartron polarization gain setting & the NLEIS testing values. Adjust the values as needed until the NLEIS sweep value cleared for all frequency. **(Step 3 & 4)**
1287 Settings & NLEIS testing:

1287 Settings	
gain	1

NLEIS Testing		
Vrd (Smallest Amplitude) (mV)	30	
IR (Ω)	100	
Testing Amplitude #1 (mA)	0.136	OK
Vrd (Largest Amplitude) (mV)	150	
IR (Ω)	100	
Testing Amplitude #2 (mA)	0.678	OK

All frequency cleared for NLEIS experiment:

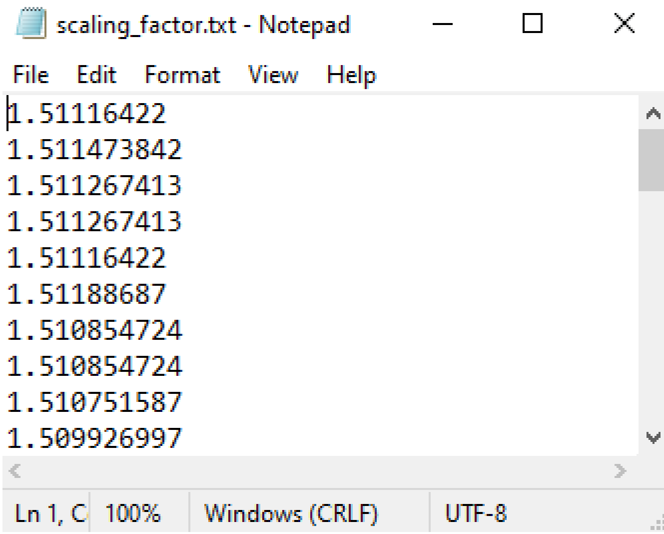
Scaling Factor	IR (Ω)	NLEIS Sweep
1.51116422	100	OK
1.511473842	100	OK
1.511267413	100	OK
1.511267413	100	OK
1.51116422	100	OK
1.51188687	100	OK
1.510854724	100	OK
1.510854724	100	OK
1.510751587	100	OK
1.509926997	100	OK
1.508794655	100	OK
1.507253286	100	OK
1.505817514	100	OK
1.502138057	100	OK
1.498273644	100	OK
1.491407777	100	OK
1.480928801	100	OK
1.466600398	100	OK
1.444299419	100	OK
1.413218391	100	OK
1.370170877	100	OK
1.313256587	100	OK
1.242686282	100	OK
1.161915262	100	OK
1.078351118	100	OK
1	100	OK
0.93328554	100	OK
0.880905943	100	OK
0.841899038	100	OK
0.814538093	100	OK
0.795821497	100	OK
0.783204162	100	OK
0.775112076	100	OK
0.76945275	100	OK
0.766202742	100	OK
0.763849101	100	OK
0.762375555	100	OK
0.761981821	100	OK
0.761405078	100	OK
0.761378883	100	OK
0.761247936	100	OK

Enter the NLEIS sweep information as calculated from the NLEIS testing data:

NLEIS Sweep

Smallest IR used (Ω)	100
Crossover Frequencies	
Smallest Amplitude (mA)	0.136
Largest Amplitude (mA)	0.678
Number of Amps to use	20
Estimated Time (hr)	9.78

Copy the scaling factor into a new text file, named "scaling_factor.txt" **(Step 7)**

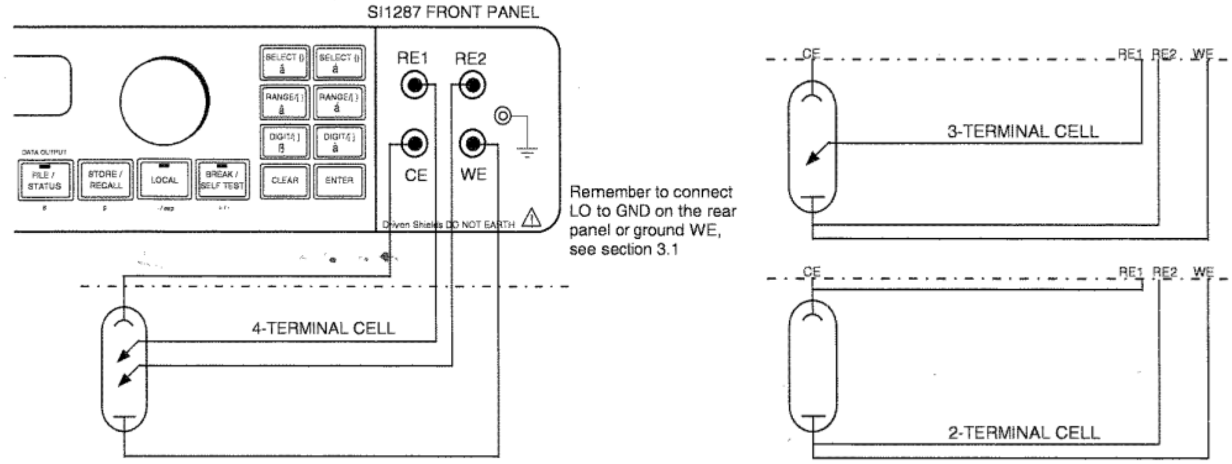


Run NLEIS Experiment

1. Physical setup.

Electrical connections:

Make the following electrical connection from the test model (fuel cell, battery, circuit, etc.) to the Solartron 1287 Potentiostat:



Make the following connection with the correct coaxial cable between the Solartron 1287 back panel to the PXI modules (5421 & 5922):

- **V OUT** (1287) to **CH 1** (NI PXI-5922 Oscilloscope)
- **I OUT** (1287) to **CH 0** (NI PXI-5922 Oscilloscope)
- **POL IN** (1287) to **CH 0** (NI PXI-5421 AWG)

NI PXI signal connection:

- Connect the Solartron GPIB gate to the PXI system computer GPIB receiver or to PXI system computer USB using a GPIB-USB-HS+ cable.
- Use NI MAX software to make sure the PXI system recognizes and can receive & send signal to the Solartron 1287.
- Also make sure to test/self-check the 5922 & 5421 modules with NI MAX.

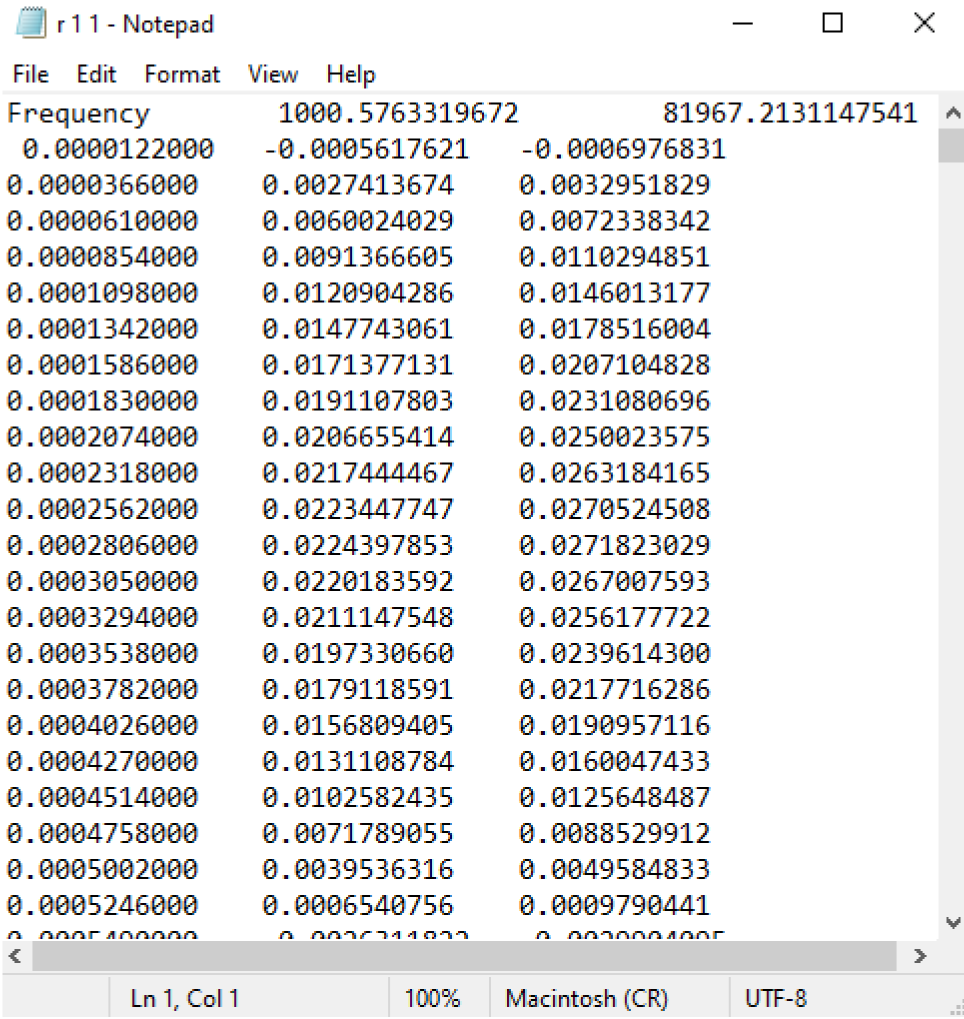
2. Perform test runs

Open NLEIS_Run_PXI.vi. Make sure to follow these steps:

1. Toggle the correct run mode:
   a. Test (NOT sweep)
   b. Galvanostatic (OR Potentiostatic). Galvanostatic is typically recommended to protect the test cell.
2. Choose the Solartron internal resistor
3. Pick the Overload Action. Cutout is recommended. This means the potentiostat and the whole experiment will stop if the potentiostat detects overloading current.
4. Pick potentiostat control bandwidth. GB0-2 is used for galvanostatic mode. PB0-9 is for potentiostatic mode.
5. Enter the peak frequency from “Template.xls” or from EIS.
6. Enter the smallest/largest test current/voltage amplitude calculated from “Template.xls”.
7. In Gain setup, select the Perturbation Input/ Voltage Output/ Current Output gain as appropriate.

8. Pick the file path to save the time files (voltage & current collected over time at the test frequency and amplitude).

If the test run is performed successfully, you should be able to find a data file in the location you chose to save it. It should look something like this:



r 1 1 - Notepad

File Edit Format View Help

Frequency	1000.5763319672	81967.2131147541
0.0000122000	-0.0005617621	-0.0006976831
0.0000366000	0.0027413674	0.0032951829
0.0000610000	0.0060024029	0.0072338342
0.0000854000	0.0091366605	0.0110294851
0.0001098000	0.0120904286	0.0146013177
0.0001342000	0.0147743061	0.0178516004
0.0001586000	0.0171377131	0.0207104828
0.0001830000	0.0191107803	0.0231080696
0.0002074000	0.0206655414	0.0250023575
0.0002318000	0.0217444467	0.0263184165
0.0002562000	0.0223447747	0.0270524508
0.0002806000	0.0224397853	0.0271823029
0.0003050000	0.0220183592	0.0267007593
0.0003294000	0.0211147548	0.0256177722
0.0003538000	0.0197330660	0.0239614300
0.0003782000	0.0179118591	0.0217716286
0.0004026000	0.0156809405	0.0190957116
0.0004270000	0.0131108784	0.0160047433
0.0004514000	0.0102582435	0.0125648487
0.0004758000	0.0071789055	0.0088529912
0.0005002000	0.0039536316	0.0049584833
0.0005246000	0.0006540756	0.0009790441

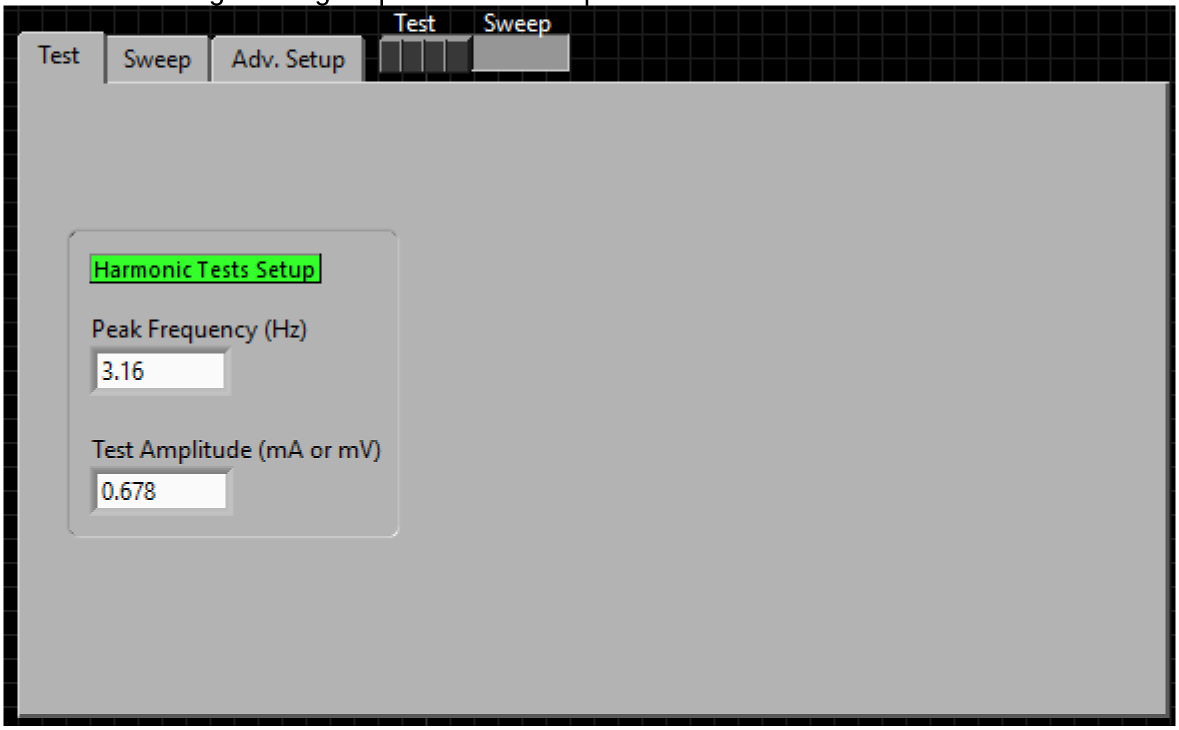
Ln 1, Col 1 | 100% | Macintosh (CR) | UTF-8

If no such file was created, make sure to check the error indicators to find out what has gone wrong with the experiment.

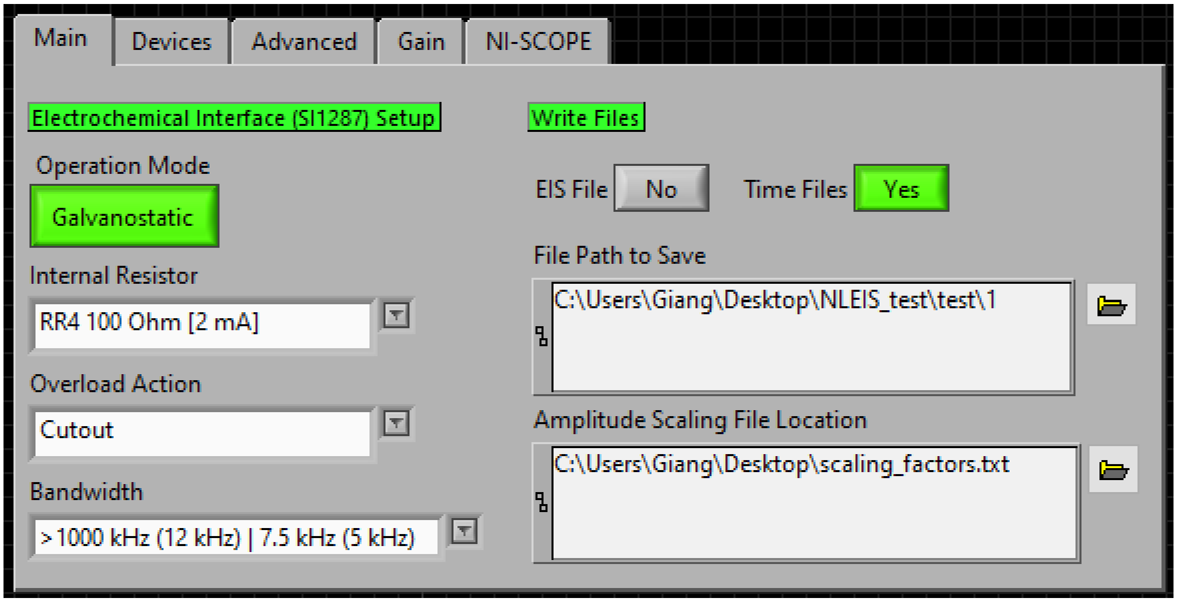
For the diode 2 test runs, I provide here the appropriate settings:

1st test run using the lowest perturbation amplitude:

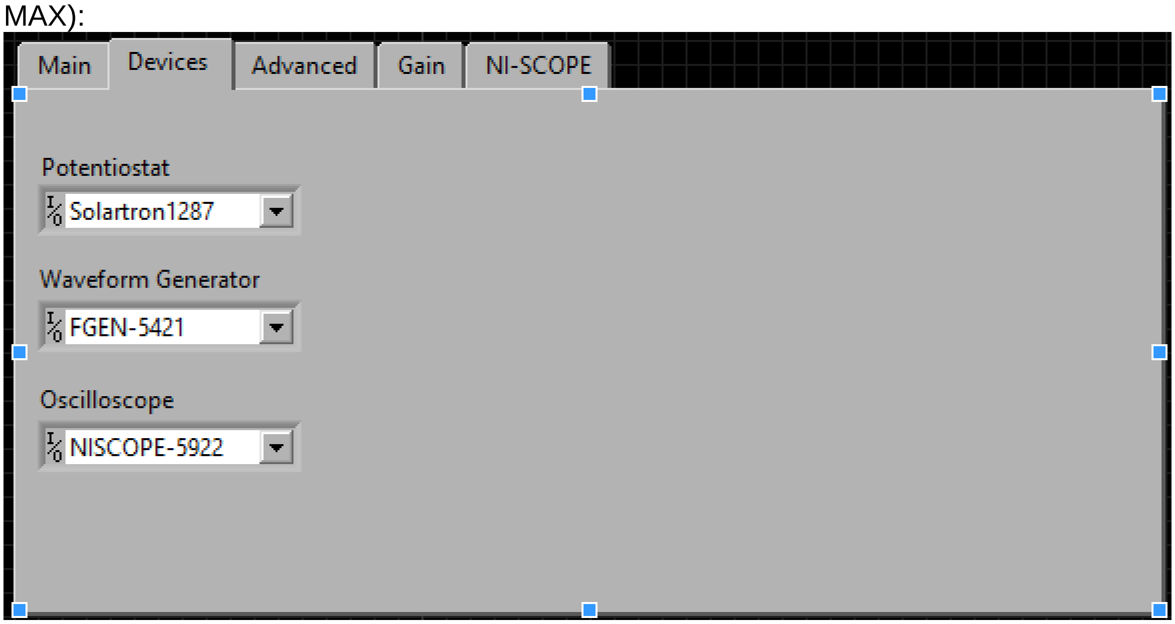
2nd test run using the largest perturbation amplitude:



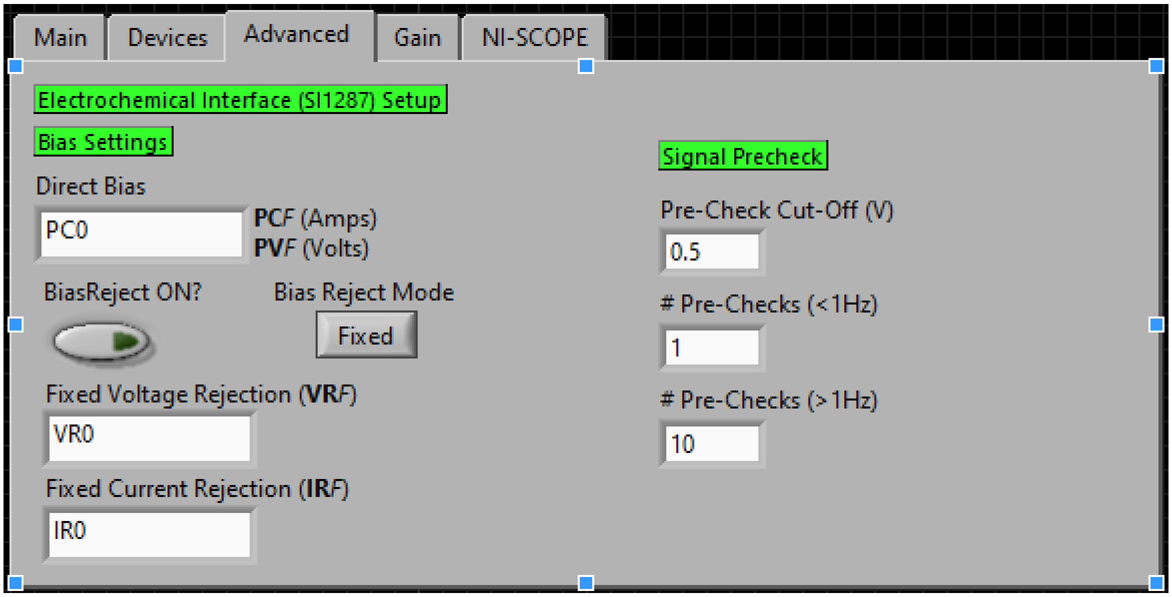
Then the main experiment setting panel:



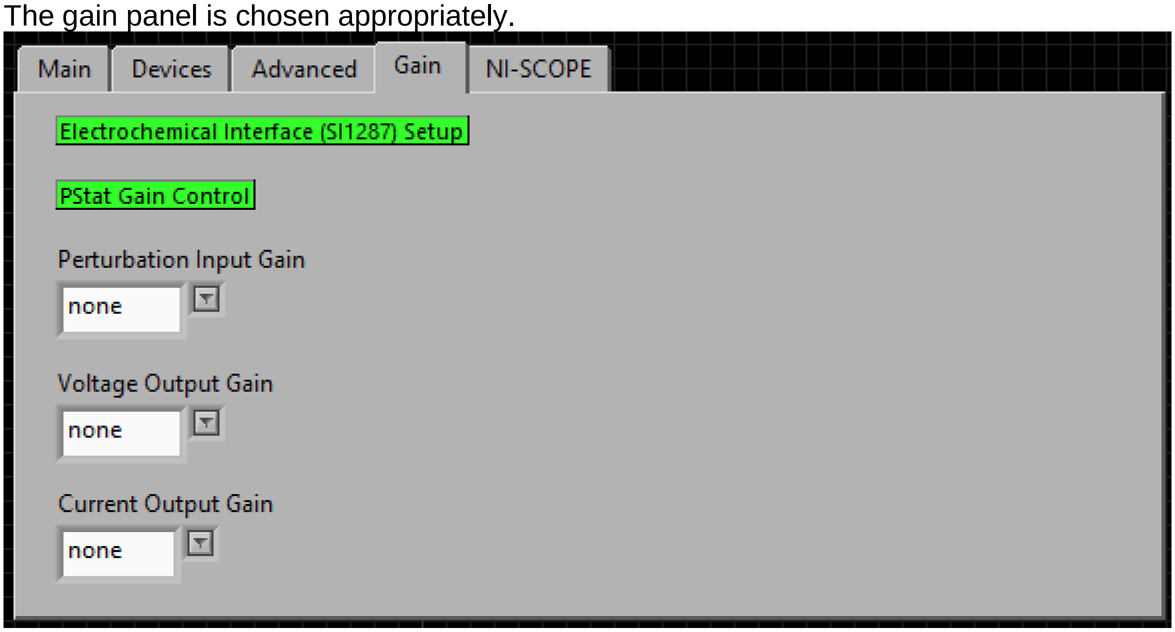
Then the device setting panel (the device is selected as appropriate as identified through NI MAX):



The advanced panel can be left as is:

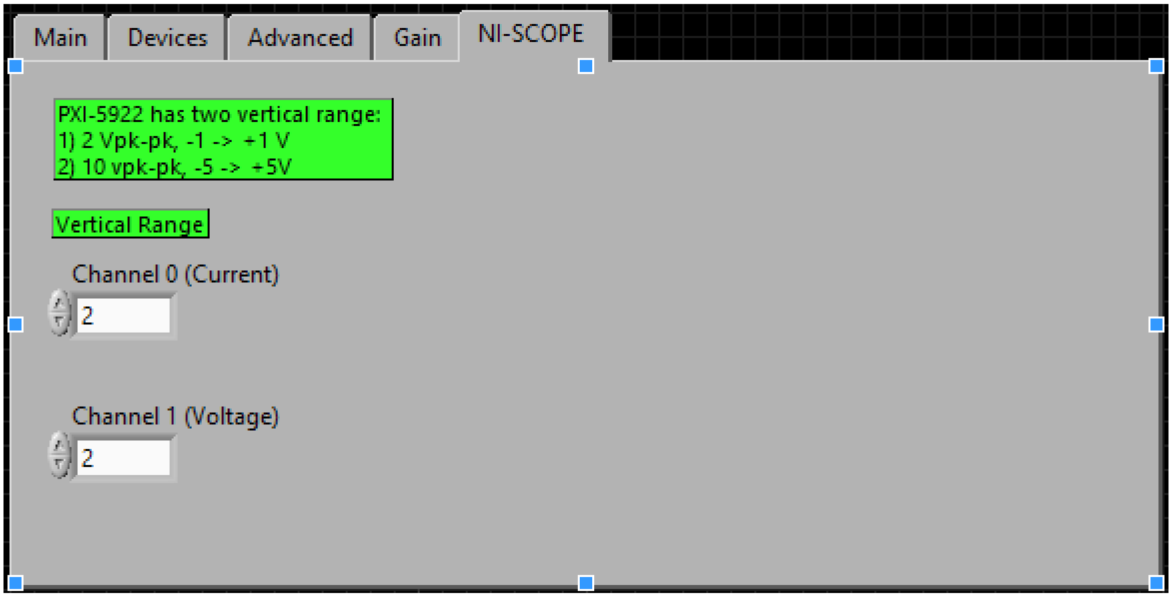


The gain panel is chosen appropriately.

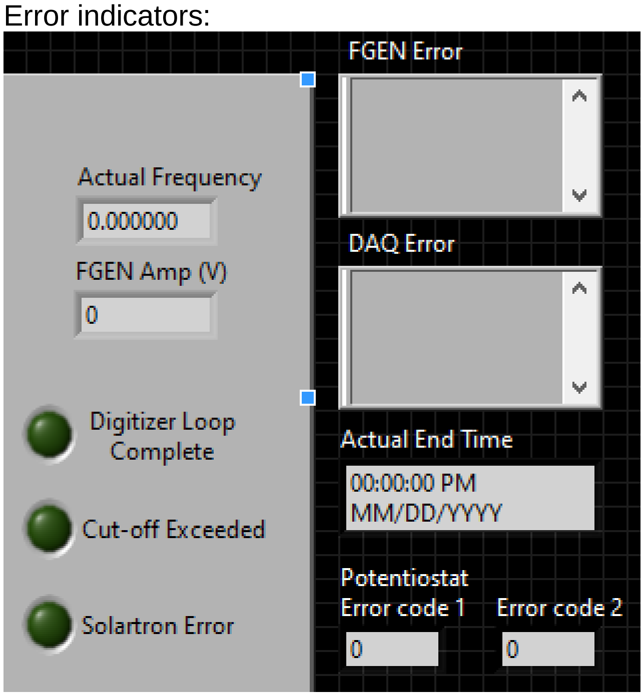


For the diode experiment, the perturbation gain is as determine from the “Determine scaling factor” step is 1 (None). The voltage magnitude and the current gain can be changed to “x 10” instead of “none” to get more precision if needed but you need to make sure the voltage will not exceed the oscilloscope chosen range (NI 5922 has 2 ranges ± 1 V or ± 5 V).

The NI-SCOPE setting:



Error indicators:



3. Perform full NLEIS experiment

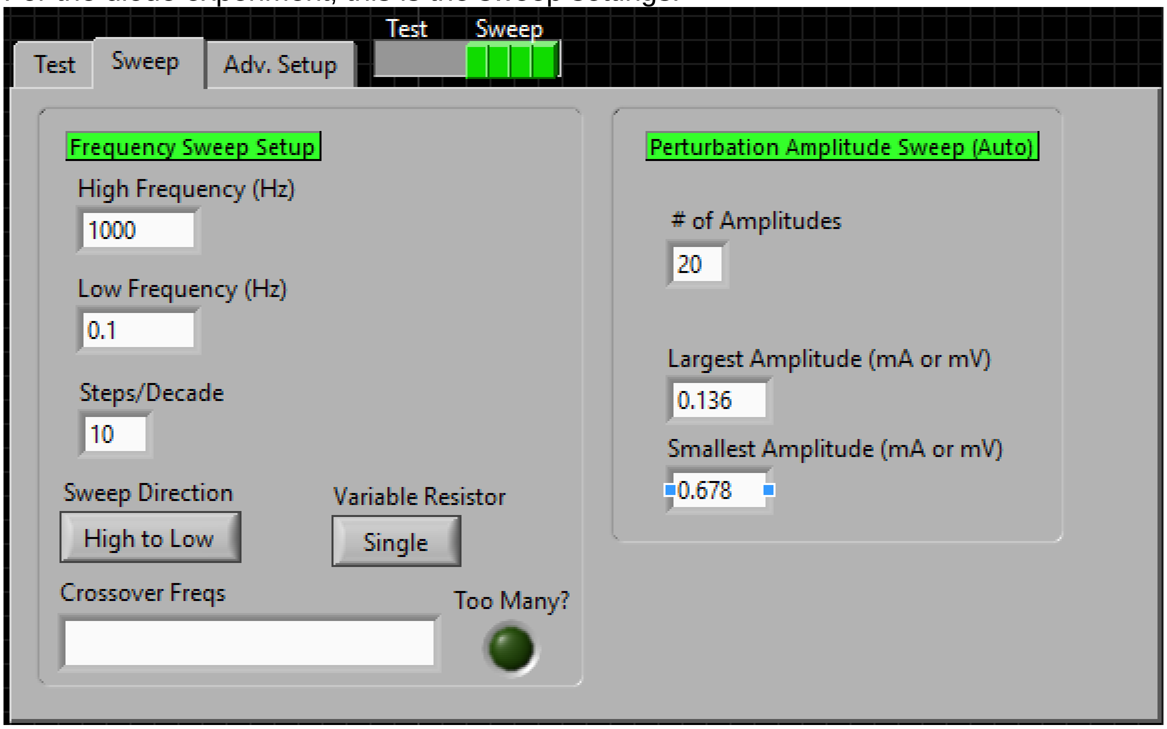
When test runs clear without problem, you are ready to perform a full NLEIS experiment. Follow these steps:

1. Toggle the correct run mode:
    a. Sweep (NOT test)
    b. Galvanostatic (OR Potentiostatic). Galvanostatic is typically recommended to protect the test cell.
2. Enter sweep experiment information:
    a. High frequency
    b. Low frequency
    c. Steps/Decade

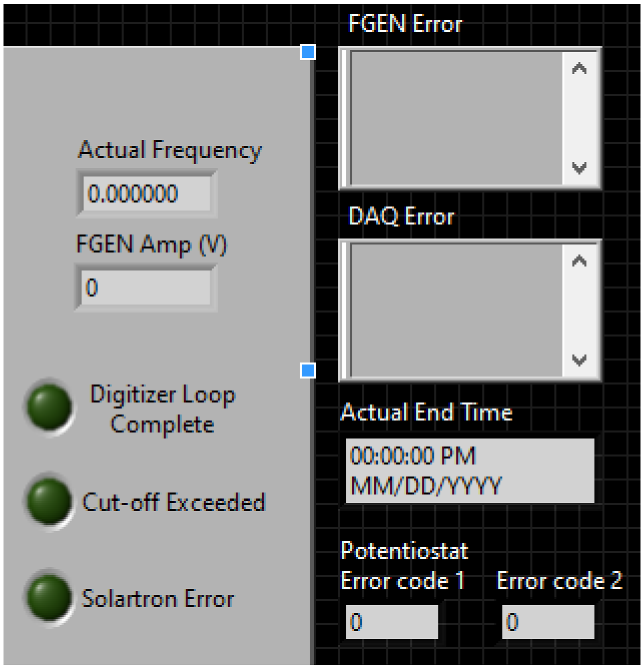
d. If the potentiostat need to change resistor pass a certain frequency, toggle the Variable Resistor option from Single to Multiple, AND add the crossover freqs, separated by comma.
e. Number of perturbation amplitudes
f. Largest amplitude
g. Smallest amplitude

3. If not done already, change the other hardware settings as needed.

For the diode experiment, this is the sweep settings:



Observe error indicators:


Actual Frequency
0.000000
FGEN Amp (V)
0
Digitizer Loop Complete
Cut-off Exceeded
Solartron Error
FGEN Error
DAQ Error
Actual End Time
00:00:00 PM
MM/DD/YYYY
Potentiostat
Error code 1
Error code 2
0
0

Process NLEIS Data

2. Process NLEIS Data with LabView

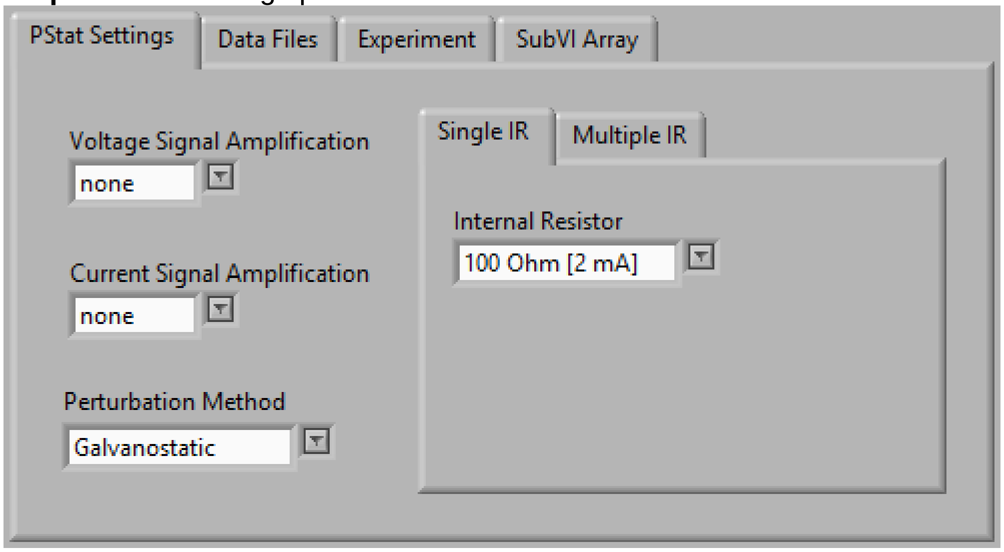
Open the two LabView vi files: NLEISView_v15.vi and NLEISView_Subvi_v15.vi. To process the NLEIS raw data, follow these steps:

1. Go to the bottom panel & select the "PStat settings" used in the NLEIS experiment:
    a. Select the correct "Voltage Signal Amplification"
    b. Select the correct "Current Signal Amplification"
    c. Select the correct "Perturbation Method" (Galvanostatic/Potentiostatic)
    d. Select either "Single IR" (internal resistor) and select the IR used in the NLEIS experiment
    e. OR select "Multiple IR" if multiple IR was used in the experiment and enter the frequency information at which the IR was switched.
2. Go to the "Data Files" panel and enter the index number of the amplitude and frequency number.
3. Go to the "Experiment" panel. In here you have the option of non-dimensionalize the processed NLEIS data.
4. Let the program know the path directory to the raw data. Do this by browsing your directory select one of the many raw data files and remove the index number in its name.
5. Enter the path to write the processed NLEIS data.
6. Now that you entered the minimum information required, hit run and you can start fitting harmonic data. See section 2 right below for recommendation on how to best fit harmonic data.
7. When you are happy with the fit at the frequency you are analyzing, hit the "NEXT" button underneath the "Graphs Updated" indicator to move to the next frequency.
8. When all frequency data have been fitted, the NLEIS data will be saved to the provided save path (in "File Path Write NLEISVIEW Data").
9. The NLEIS data is now can be view in Excel for example.

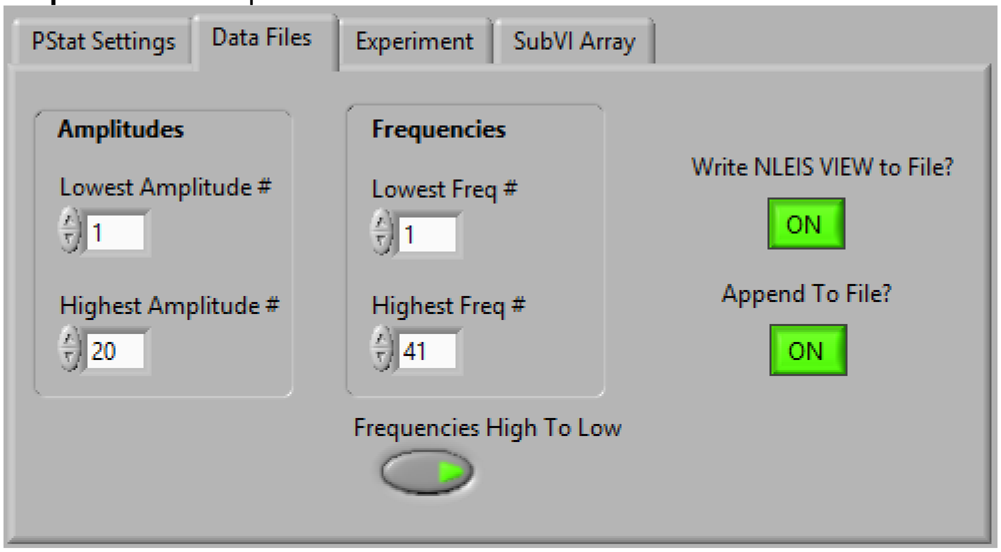
Follow these following steps to fit NLEIS response at each frequency:

You can consult the visual guide below that follows the diode test circuit example.

Step 1: PStat settings panel

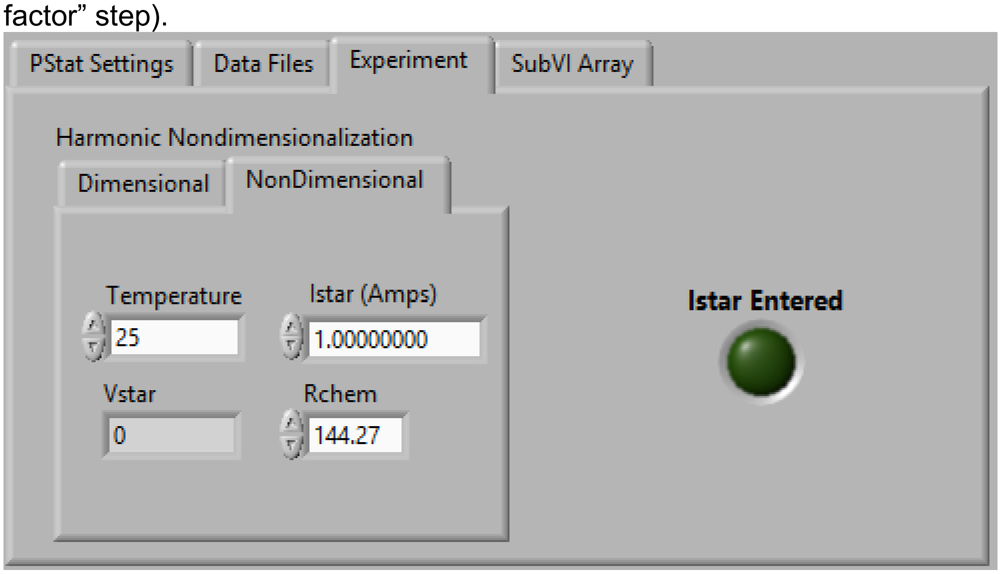


Step 2: Data Files panel

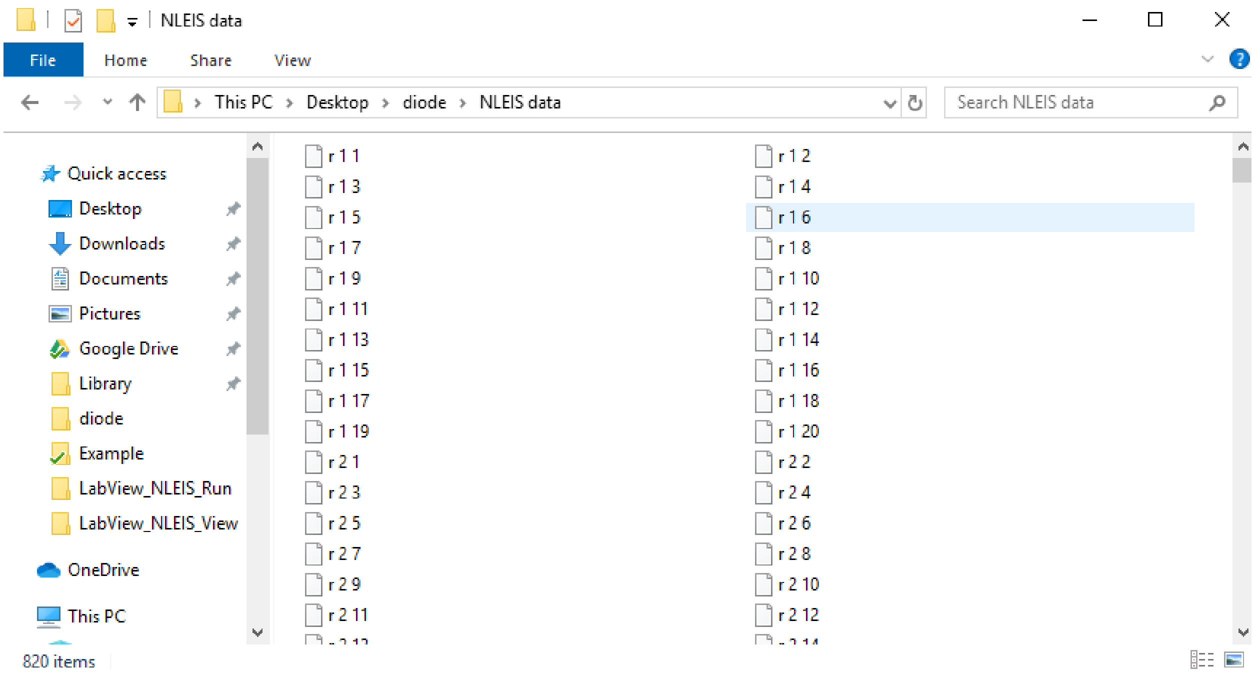


This is because we used 20 amplitudes and 41 frequencies (1 kHz – 0.1 Hz, 10 points per decade).

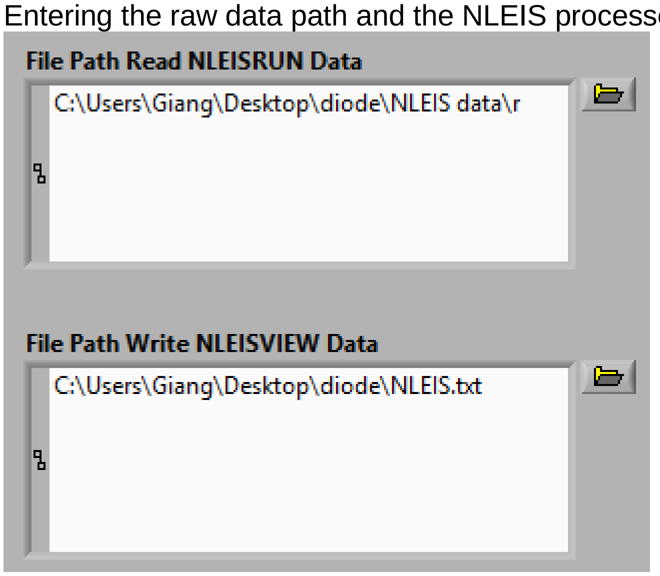
Step 3: It is optional to non-dimensionalized the harmonic data. If you choose to, enter the temperature and the Rchem value (which we already determined in the "Determine scaling factor" step).



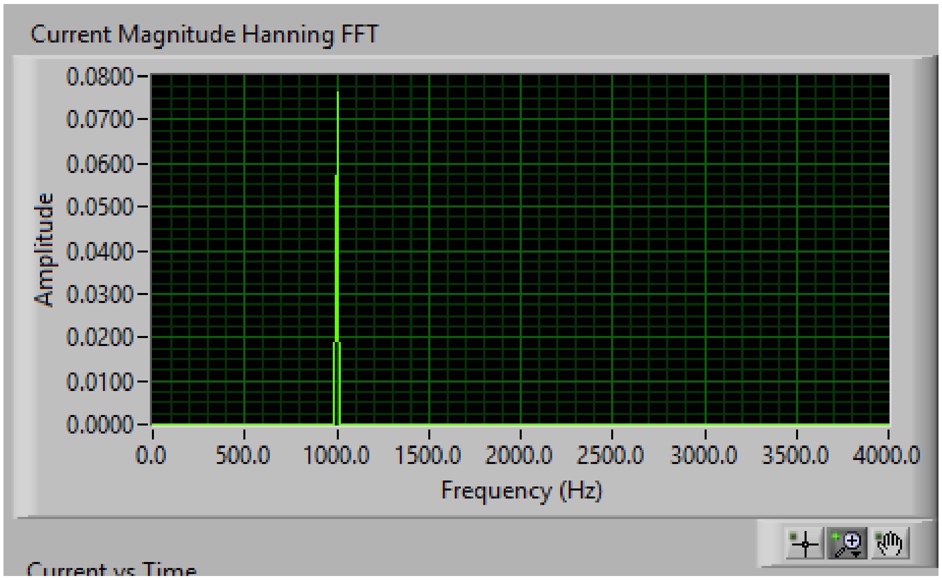
Step 4 – 5: Raw data directory:
The NLEIS raw data from the experiment is included in the Zip file in the example folder – "NLEIS data.zip".

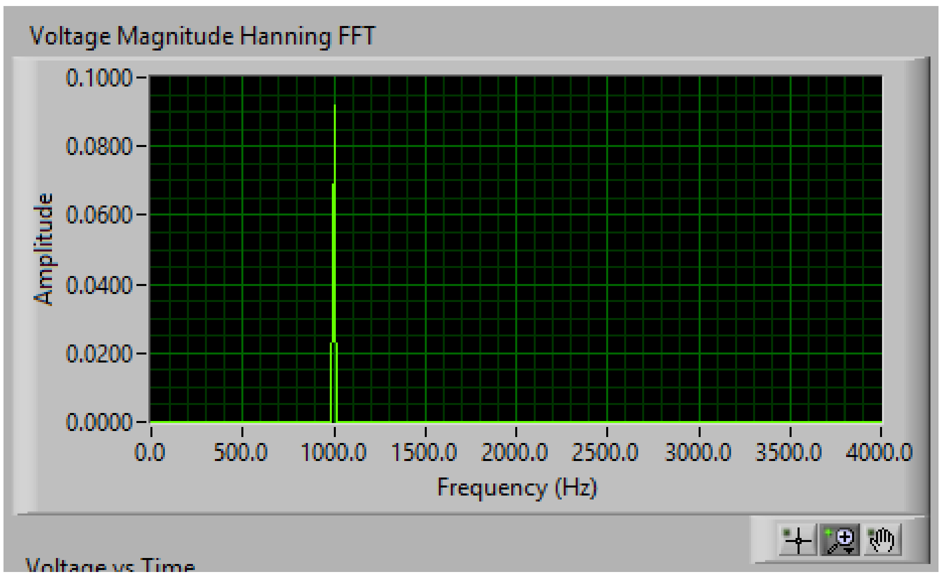


Entering the raw data path and the NLEIS processed data save location:

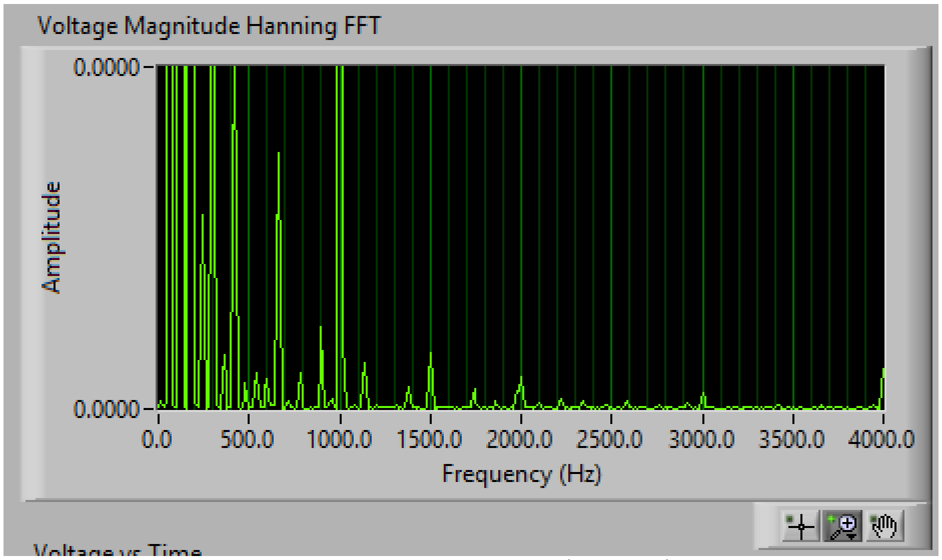


1. Voltage-current harmonic fitting recommendation.
This is the minimum best-practice recommendation to fit harmonic data well. The diode example accompanies this guide for visualization.
Step 1: Check the FFT transformation of current and voltage data in NLEISView_Subvi_v15.vi.



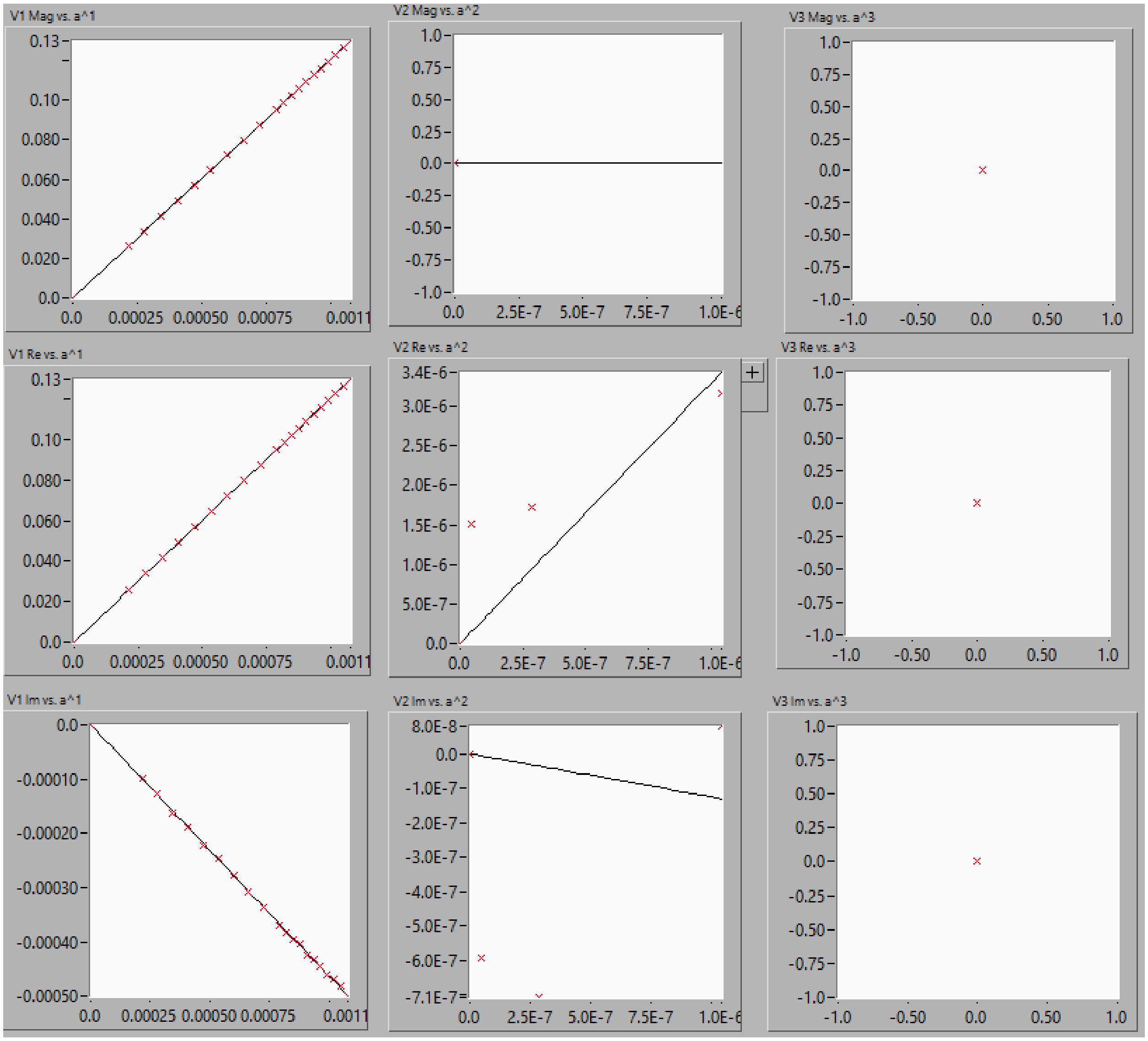


You should also Zoom into the voltage response (for galvanostatic experiment) and the current response to see if there is any response at 2nd harmonic and 3rd harmonic (at 2 times and 3 times the perturbation frequency).

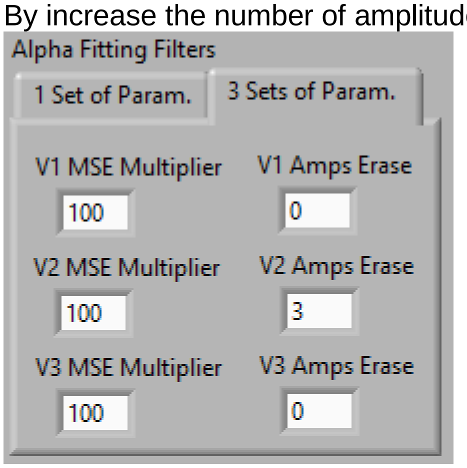


Based on this FFT, I would interpret that 2nd and 3rd harmonic is not present at this frequency.

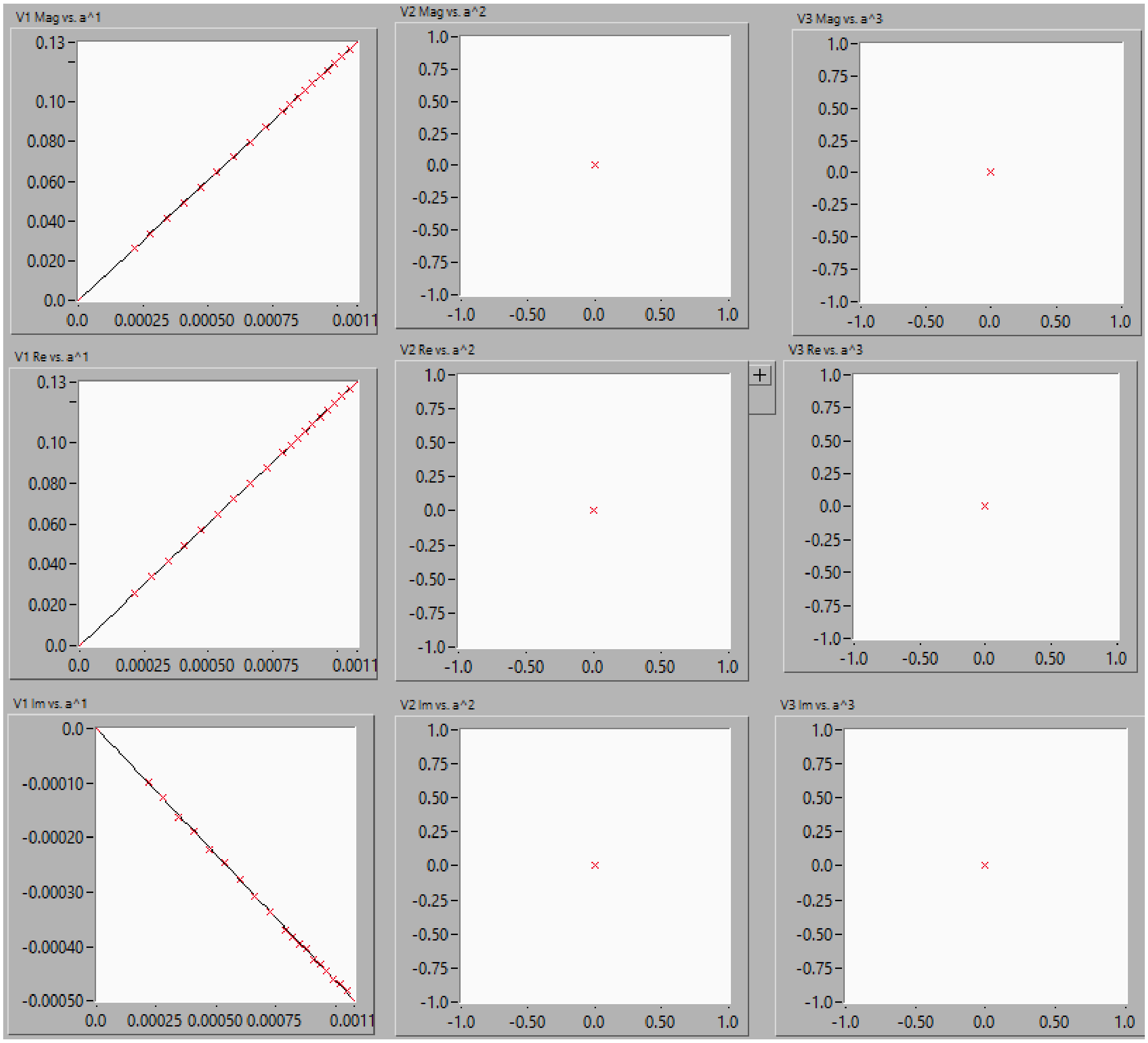
Step 2: If the fitting picks up noises and fit it to 2nd and 3rd harmonic or there appear to be outlier at high amplitude value, you can change the number of amplitude included to remove the outlier/misfitting.
At 1000 Hz some noises are fitted to 2nd harmonic:



By increase the number of amplitudes erased:

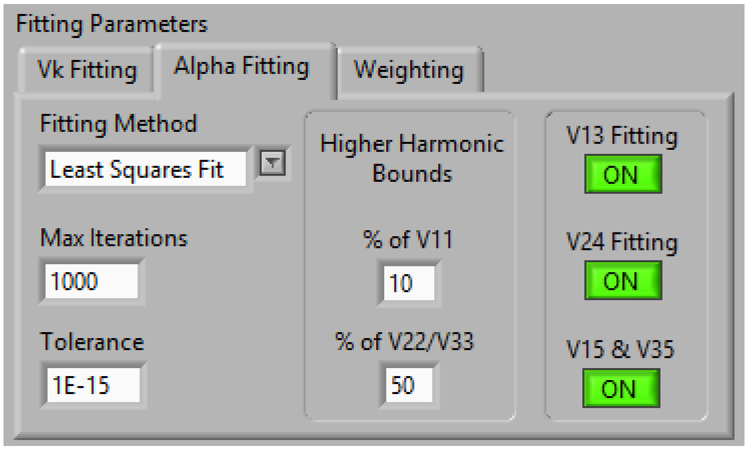


We will see that on the graph that the program is not fitting 2nd harmonic anymore:

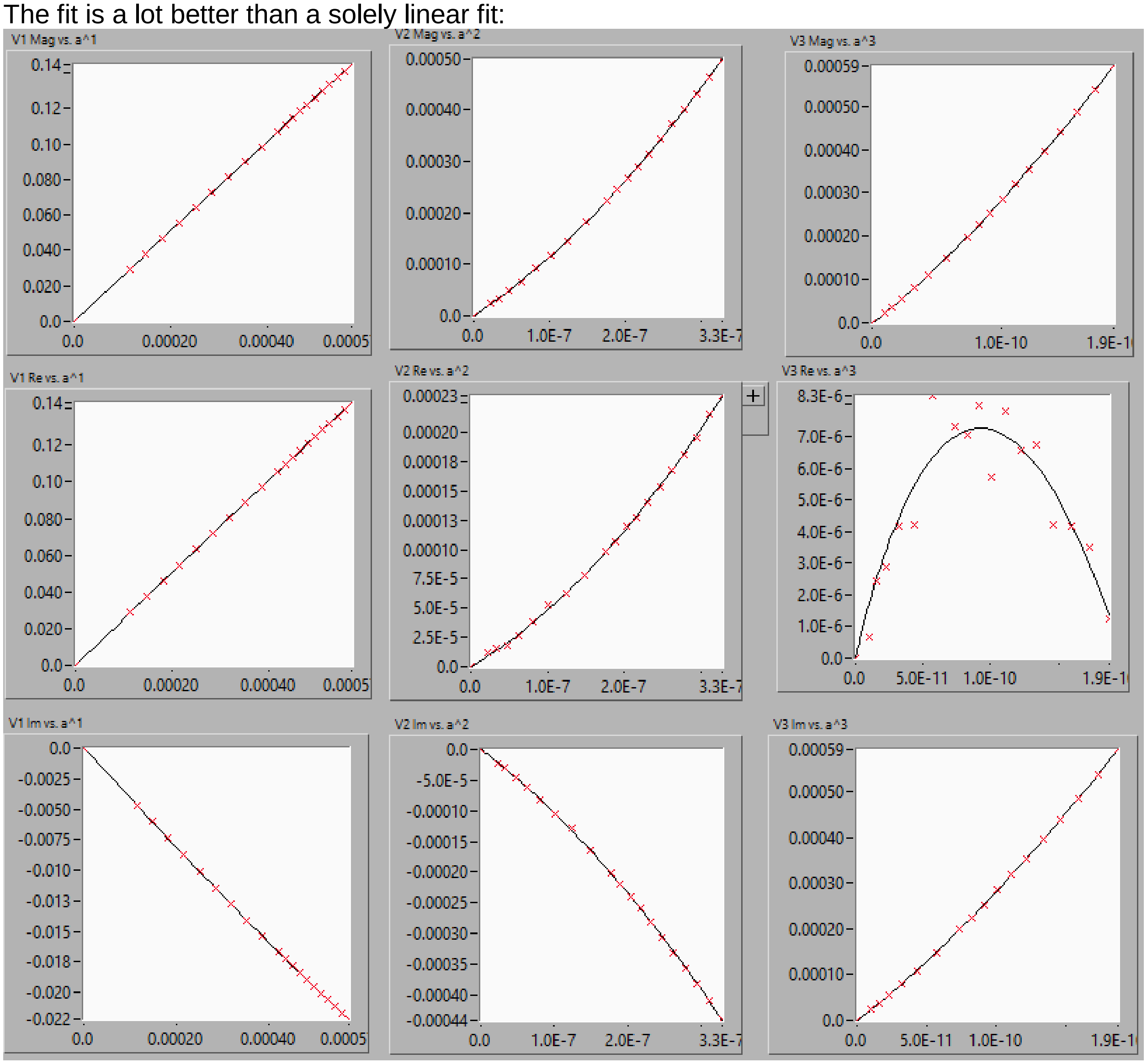


Step 3: If at certain frequency, the linear fit on the graph seems insufficient for V1, you can turn on V13 fitting. Accordingly, for V2, you can turn on V24 fitting, for V3, you can turn on V15 & V35 fitting.

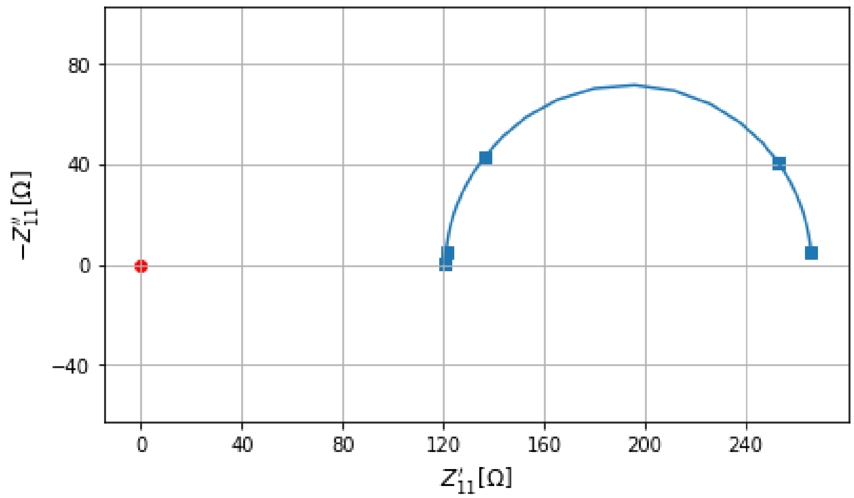
For example, at 1 Hz, the V13, V24, and V15&V35 fitting options are selected:

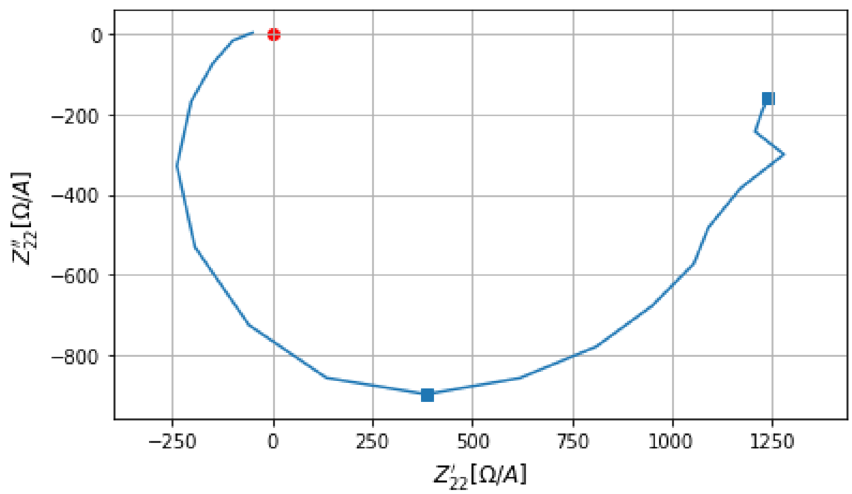


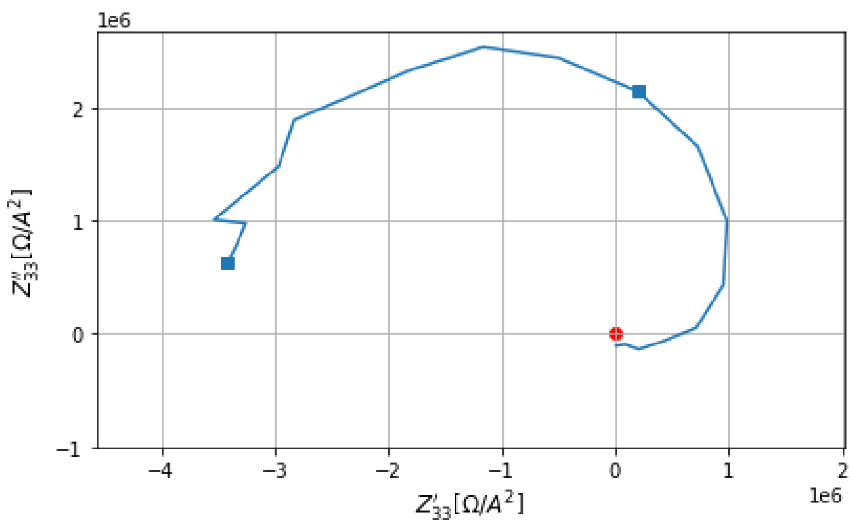
The fit is a lot better than a solely linear fit:



The Diode NLEIS data is in text file “NLEIS.txt” and the copied to the excel file “NLEIS.xlsx” and visualize with some plots:


80
40
0
−40
$-Z_{11}''[\Omega]$
0
40
80
120
160
200
240
$Z_{11}'[\Omega]$


0
−200
−400
−600
−800
$Z_{22}''[\Omega/A]$
−250
0
250
500
750
1000
1250
$Z_{22}'[\Omega/A]$


1e6
2
1
0
−1
$Z_{33}''[\Omega/A^2]$
−4
−3
−2
−1
0
1
2
1e6
$Z_{33}'[\Omega/A^2]$