

Semiconductor Spectroscopy Lab Software and Hardware Reference

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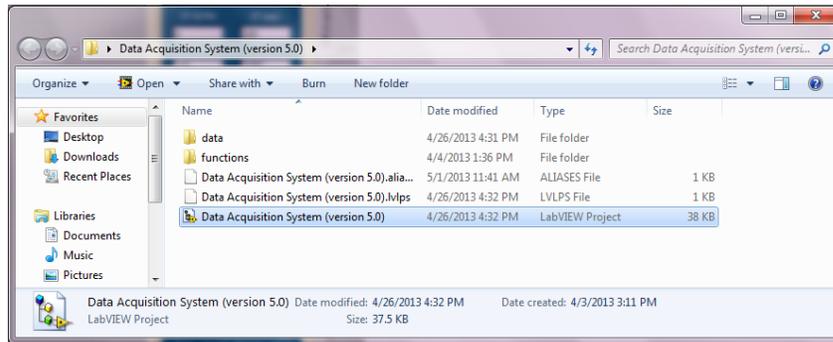
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Introduction

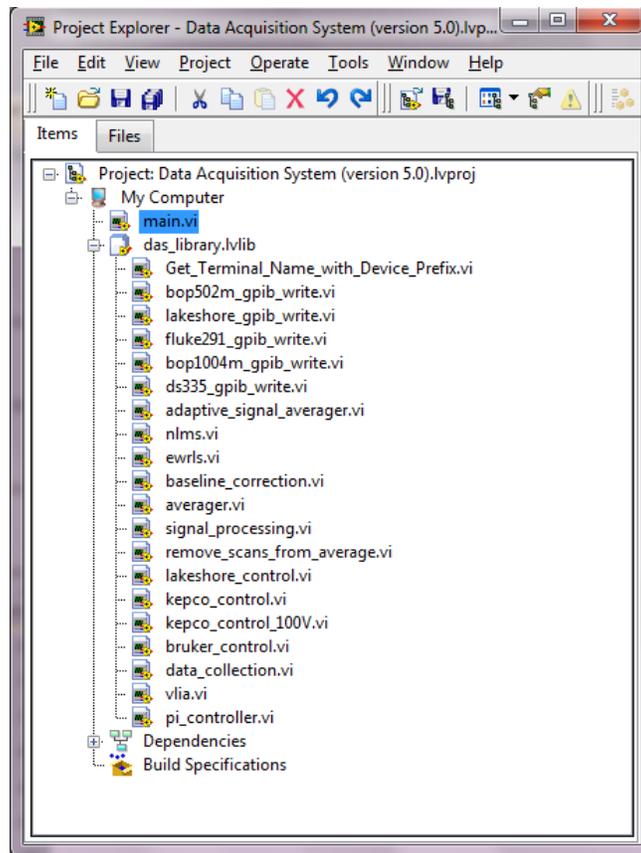
This document is intended to describe the software and some of the hardware that runs the lab spectrometers. It also provides recommended spectrometer settings for obtaining the optimal EPR and EDMR signals. The software has been created with and runs properly with Labview 2009. It should work for newer versions of Labview; however, it hasn't been tested or verified.

A. Locating and Opening the Software

1. Copy entire "Data Acquisition System (version 5.0)" folder to the user desktop. The location of this folder is "Corey Cochrane \PhD ESC\Labview\Final Version\Data Acquisition System (version 5.0)"
2. Open the folder and select "Data Acquisition System (version 5.0)" Labview project file.



3. Once Labview starts, the project explorer window will open. Double click on "main.vi" to open the GUI.



B. Software Configuration for EDMR

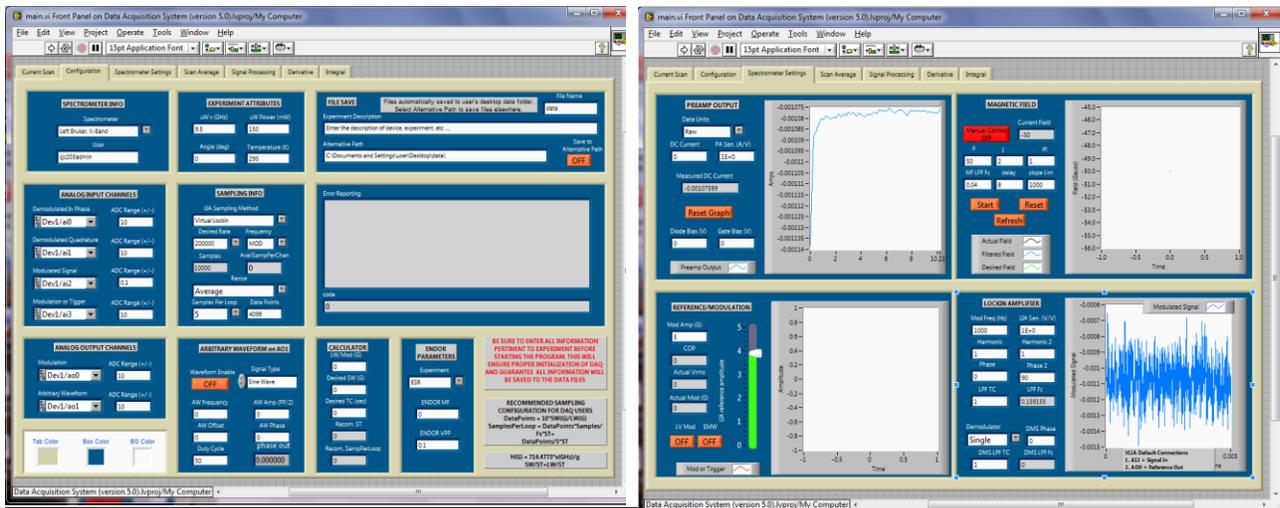
The software is configured from the second and third tabs in the LV GUI. They are labeled *Configuration* and *Spectrometer Settings*. So that the entered parameters do not have to be set each time the software is opened, one can make them default by (1) entering the desired default value of the variable, (2) right clicking on that variable, (3) selecting *data operations*, and finally (4) selecting *make current value default* for each parameter that is desired to be changed. For a first time spectrometer setup, ensure that the following two parameters are configured properly.

1. SPECTROMETER SELECTION: Under the *SPECTROMETER INFO* section of the *Configuration* tab, select the spectrometer in which the software is to be run. The user can select from the following EDMR spectrometers:

- Varian, X-Band:** Green Varian magnet / power supply, Micro Now microwave bridge, LS 475 Gaussmeter
- LakeShore, X-Band:** Lakeshore magnet / power supply, C. Arnow Microwave Bridge, LS 475 Gaussmeter,
- Hewlett Packard, K-Band:** HP Power Supply, white magnet, C. Arnow microwave bridge, LS 475 Gaussmeter
- Hewlett Packard, X-Band:** HP Power Supply, white magnet, Micro Now microwave bridge, LS 475 Gaussmeter
- Kepeco-50V-2A, VHF-Band:** Currently not in use. Can be used with the Kepeco 50V 2A power supply
- Kepeco-100V-4A, VHF-Band:** Kepeco-100V-4A power supply, custom built magnet, SG380 RF Sig Gen, LS 450 Gaussmeter

2. FILE SAVE: Also under the *SPECTROMETER INFO* section of the *Configuration* tab, type in the name of the user that is signed on to the computer so that the software knows where to save the generated Labview files. This location will be `C:\Documents and Settings\user\Desktop\data\`. If a user does not have a user account or if it is desired to save the .lvm files to a different location, simply turn on the *Save to Alternative Path* button and change the *Alternative Path* setting under the *FILE SAVE* block in the *Configuration* tab. Here, the user is also able to provide a description of the device and experimental details under *Experiment Description*.

Important Note: If a user enters a user name that is different than the user account in which the computer is logged on, LAVIEW will stop at the end of the scan because it is attempting to save the .lvm file to a location in which doesn't exist or current user has access to. Ensure that this is properly set up before run time.



3. **LOCKIN AMPLIFIER:** The first step that needs to be taken after selecting one of the EDMR spectrometers is to determine the lockin amplifier (LIA) that will be used. The options are *NI6259*, *SR830 GPIB*, or *Virtual Lockin*. Follow the steps below for specific details as how to setup each.

- a. **Virtual Lockin:** This is a software based lock-in amplifier has two independent demodulators and is only really intended for use with the EDMR spectrometers. The following controls need to be configured for proper functioning:

SAMPLING INFO in the *Configuration* Tab

1. **LIA Sampling Method:** set to virtual lockin.
2. **Sampling Rate:** Set to a rate that is at least 10x modulation frequency. Note that this parameter is limited to the maximum rate that is achievable in the DAQ being used. The maximum rate for the NI-6259 is about 1MS/s. Because the program actually reads in 2 channels (current and modulation) in this virtual LIA mode, the maximum rate that can be set is 500KS/s. However, because it is not that advantageous to move to high modulation frequencies because of the gain bandwidth tradeoff, a rate of 250KS/s should be more than sufficient when modulating at 1-20kHz.
3. **Frequency:** Set this parameter to *MOD* which signifies that the user is in modulation mode. The other mode is *DC*. In this mode, the user is able to sample the DC current Note that when in *DC* mode, ensure that the preamp does NOT have its high pass filter on.
4. **Resize:** This parameter configures the resampling that occurs after the signal is demodulated with the full sampling rate. Essentially, the DAQ reads data in by “chunks”, where the number of samples in each chunk is determined internally and is based on the configured sampling rate. When the signal is demodulated, the number of samples that are available are far more than what are actually needed to represent a DC signal. Therefore, the user can *average* or *decimate* (pick evenly spaced samples from chunk) these chunks. The averaging provides an additional noise reduction.
5. **Samples per Loop:** Determines the number of samples per loop (or “chunk” as describe above) that the software extracts from the fully sampled signal at each loop iteration. It is recommended that the user ensure that this value is set to the next available integer setting from the number calculated in *Recom. SampPerLoop* parameter in the calculator block on the same tab. Using this value ensures that the signal is “well represented”.
6. **Data Points:** The number of samples that represents the final data length. Setting this to its default value of 4096 is sufficient for just about all experiments. Ensure that this is not set too high (>10,000) because the computer may have a difficult time processing the data.

LOCKIN AMPLIFIER Block in the *Spectrometer Settings* Tab

1. **Mod Freq (Hz):** modulation frequency in Hz.
2. **Sensitivity (V/V):** set to 1 if only the preamp is used. If an additional amplifier is required, set this parameter to this inverse gain (or sensitivity) of the amplifier.
3. **Harmonic:** Harmonic of the first demodulator. Allows the user to demodulate any harmonic frequency of the fundamental set in the *Mod Freq (Hz)* parameter. For normal lock-in operation, set this to 1.
4. **Harmonic 2:** Harmonic of the second demodulator. Allows the user to demodulate any harmonic frequency of the fundamental set in the *Mod Freq (Hz)* parameter. For normal lock-in operation, set this to 1
5. **Phase:** Phase of the first demodulator in degrees. For initial phase calibration, set *phase* to 0 and set *phase 2* to 90. The phase offset is automatically calculated on the scan average tab based upon the in-phase and quadrature components.
6. **Phase 2:** Phase of the second demodulator in degrees. For initial phase calibration, set *phase* to 0 and set *phase 2* to 90. The phase offset is automatically calculated on the scan average tab based upon the in-phase and quadrature components.
7. **LPF TC:** Low pass filter time constant.

8. *Demodulator*: set to single. Do not worry about setting the remainder of the parameters in this block unless you are interested in playing around with “double demodulation schemes”

REFERENCE/MODULATION Block in the *Spectrometer Settings* Tab

1. *Mod Amp (G)*: This is not a control but rather a parameter the user can set for the software to save to the data file.
2. *Lockin Reference Amplitude*: This controls the voltage amplitude of the modulation signal that is generated in Labview. See below to determine how to select the channel in which it is generated.

ANALOG INPUT CHANNELS Block in the *Configuration* Tab

1. *Modulated Signal*: Sets the channel on the BNC2110 to sample the amplified modulated current from the preamp. The default source is AI2.
2. *ADC Range (+/-)*: Sets the limits of the DAQ for the Modulated Signal. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 . The real-time signal is displayed in the *LOCKIN AMPLIFIER* block of the *Spectrometer Settings* tab. Use this viewer to optimize this setting. When using modulation, the user should use the high pass filter setting on the lock-in amplifier to for maximizing the dynamic range of the DAQ. With the DC component removed, the user should set the this range as low as possible such that the signal doesn't “clip” when observed in the viewer.
3. *Modulation or Trigger*: Sets the channel on the BNC2110 to sample the amplified modulation signal (measured across a power resistor in the modulation box) so that modulation amplitude can be calibrated. The default source is AI3.
4. *ADC Range (+/-)*: Sets the limits of the DAQ for the amplified modulation signal.
5. The remaining parameters in this block are ignored when in the virtual lockin mode.

ANALOG OUTPUT CHANNELS Block in the *Configuration* Tab

1. *Modulation*: Sets the channel on the BNC2110 which will output the reference modulation signal. The default source is A00.
2. *ADC Range (+/-)*: Sets the limits of the DAQ for the Modulation Signal.

- b. *NI6259*: This setting is used if the NI6259 DAQ (or equivalent) is used to sample the output of an external hardware lockin such as the SR830. The following controls need to be configured for proper functioning:

SAMPLING INFO in the *Configuration* Tab

1. *LIA Sampling Method*: set to *NI6259*.
2. *Sampling Rate*: If it is desired to calibrate and view the FFT of the modulated signal, set the rate to a value that is approximately x10 the modulation frequency. However, if the user can do without this feature, it is only required that the DAQ sample the data at, 10 Hz to 1kHz.
3. *Frequency*: Set this parameter to *MOD* which signifies that the user is in modulation mode. The other mode is *DC*. In this mode, the user is able to sample the DC. Note that when in DC mode, ensure that the preamp does NOT have its high pass filter on and that this signal is not only being fed into the external lockin but also being sampled by the DAQ (default AI2).
4. *Resize*: This parameter configures the resampling that occurs after the signal is sampled by the DAQ. Essentially, the DAQ reads data in by “chunks”, where the number of samples in each chunk is determined internally and is based on the configured sampling rate. When the signal is sampled, the number of samples that are available are far more than what are actually needed to represent a DC signal. Therefore, the user can *average* or *decimate* (pick evenly spaced samples from chunk) these chunks. The averaging provides an additional noise reduction.
5. *Samples per Loop*: Determines the number of samples per loop (or “chunk” as describe above) that the software extracts from the fully sampled signal at each loop iteration. It is recommended that the user ensures that this value is set to the next available integer

setting from the number calculated in *Recom. SampPerLoop* parameter in the calculator block on the same tab. Using this value ensures that the signal is “well represented”.

6. **Data Points:** The number of samples that represents the final data length. Setting this to its default value of 4096 is sufficient for just about all experiments. Ensure that this is not set too high (>10,000) because the computer may have a difficult time processing the data.

LOCKIN AMPLIFIER Block in the *Spectrometer Settings* Tab

1. **Mod Freq (Hz):** This is not a control, but rather only an indicator which saves the entered value into the saved .lvm file.
2. **Sensitivity (V/V):** Sensitivity of the external lockin amplifier. Ensure to enter this for proper unit conversion.
3. **Harmonic:** Harmonic of the external lockin amplifier. This is not a control and is used to record the lockin harmonic used during the experiment.
4. **Harmonic 2:** This serves no purpose when the lockin mode is set to *NI6259*.
5. **Phase:** Phase of the external lockin amplifier. This is not a control and is used to record the lockin phase used during the experiment.
6. **Phase 2:** This serves no purpose when the lockin mode is set to *NI6259*.
7. **LPF TC:** Low pass filter time constant of the external lockin amplifier. This is not a control and is used to record the lockin time constant used during the experiment
8. **Demodulator:** This serves no purpose when the lockin mode is set to *NI6259*.

REFERENCE/MODULATION Block in the *Spectrometer Settings* Tab

1. **Mod Amp (G):** This is not a control but rather a parameter the user can set for the software to save to the data file.
2. **Lockin Reference Amplitude:** This controls the amplitude of the modulation signal that is generated in Labview ONLY IF the *LV Mod* button is selected. This allows Labview to provide the reference signal used for modulation. See below to determine how to select the channel in which it is generated.

ANALOG INPUT CHANNELS Block in the *Configuration* Tab

1. **Demodulated In Phase:** Sets the channel on the BNC2110 to sample the in-phase output signal of the external lockin. The default source is AI0.
2. **ADC Range (+/-):** Sets the limits of the DAQ for the sampling the in-phase component of the external lockin. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .
3. **Demodulated Quadrature:** Sets the channel on the BNC2110 to sample the quadrature output signal of the external lockin. The default source is AI1.
4. **ADC Range (+/-):** Sets the limits of the DAQ for the Modulated Signal. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .
5. **Modulated Signal:** Sets the channel on the BNC2110 to sample the amplified modulated signal from the preamp. The default source is AI2.
6. **ADC Range (+/-):** Sets the limits of the DAQ for the Modulated Signal. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .
7. **Modulation or Trigger:** Sets the channel on the BNC2110 to sample the amplified modulation signal (measured across a power resistor in the modulation box) so that modulation amplitude can be calibrated. The default source is AI3.
8. **ADC Range (+/-):** Sets the limits of the DAQ for the amplified modulation signal. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .

ANALOG OUTPUT CHANNELS Block in the *Configuration* Tab

1. **Modulation:** Sets the channel on the BNC2110 which will output the reference modulation signal ONLY if the *LV Mod* button is selected. The default source is AO0.

2. **ADC Range (+/-):** Sets the limits of the DAQ for the Modulation Signal.
- c. **SR830 GPIB:** Enables sampling of data through the GPIB interface of the SR830 lockin amplifier. Even though this is a convenient hardware feature, it is not recommended for use with the spectrometers as only one sample can be obtained per loop cycle which limits the sampling rate of the data. The fastest loop rate of the software is configured to 10 (or sometimes 5) iterations per second which is in some cases, may not be enough samples to fully represent a signal if the scan time is fast. When using this setting, use the steps described above (for configuring NI6259) to configure the software with the exception of the following:

SAMPLING INFO in the *Configuration* Tab

1. **LIA Sampling Method:** set to *SR830 GPIB*
2. **Resize:** Set to *None*.
3. **Samples per Loop:** Set to 1.

4. **PREAMP OUTPUT:** This set of controls and indicators are located under the *Spectrometer Settings* tab.

Data Units: This configures the software to save the data according to the units selected. For EDMR, ALWAYS set this value to a current. The pA setting is usually a good default setting.

DC Current: This parameter does not control anything. It is simply used to record the DC current measured by the DAQ as indicated in the *Measured DC Current* indicator. The units of this indicator and the display are Amps. It is recommended that the user always set this parameter as it allows for the calculation of ΔI over I .

PA Sens (V/A): Preamplifier sensitivity. Ensure that this is ALWAYS set for proper unit conversion.

Diode Bias: This parameter is simply used to record the diode bias to the saved file.

Gate Bias: This parameter is simply used to record the gate bias to the saved file.

5. **MAGNETIC FIELD CONTROL:** There are no steps needed to configure the magnetic field control. When the desired spectrometer is selected, the proper controls are read in internally so there is no interaction with the user. If it is desired to retune a magnet or configure the P and I controls for a new magnet, turn on the *Manual Control On* button. **Caution: If one is not familiar with control theory, please seek additional information as how to tune a PI controller before arbitrarily setting these parameters as it may cause serious damage to the magnet or power supply.** The one parameter that can be configured in this box is the *delay* variable which sets the time, in seconds, in between consecutive scans. Ensure that this parameter is configured for at least 8 seconds so that the field has enough time to settle.

6. **EXPERIMENT ATTRIBUTES:** These are not controls but rather parameters that can be set which are saved to the .lvm files. It is good practice to set these parameters for every experiment.

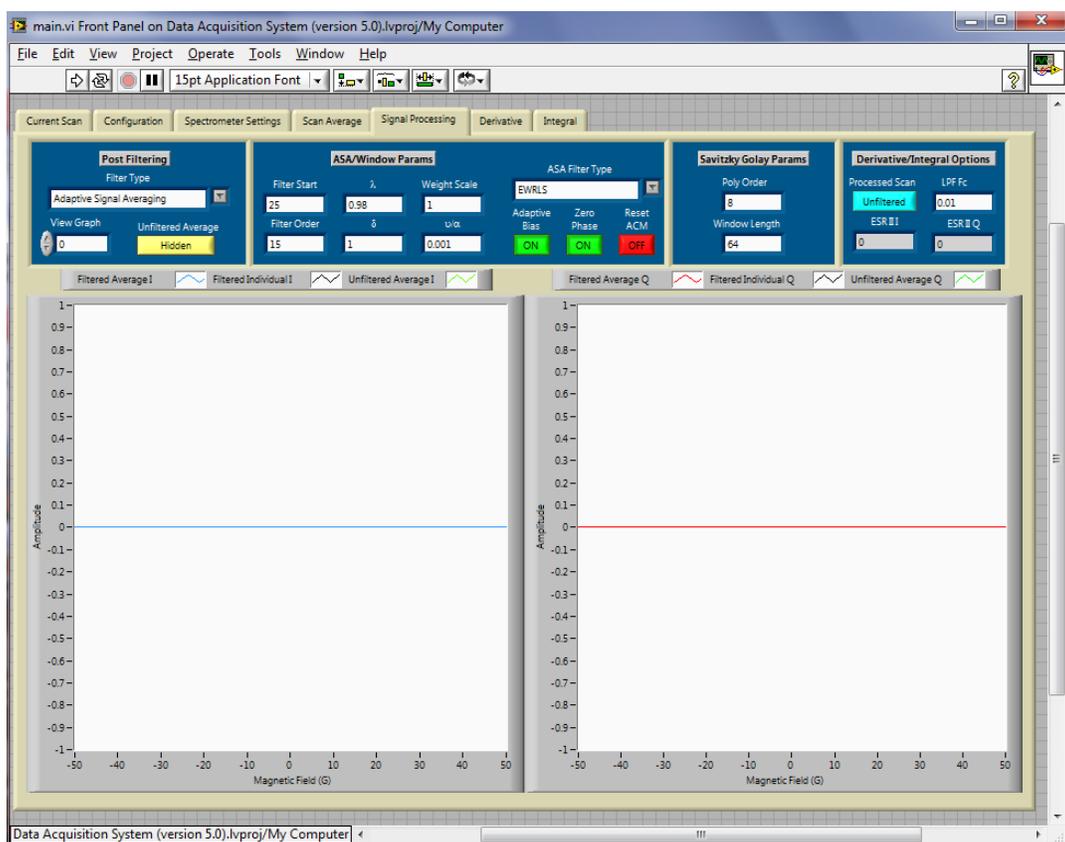
7. **CALCULATOR:** Allows the user to enter the desired line width (LW), sweep width (SW), and time constant (TC) to obtain a recommended sweep time (ST) and samples per loop (*Recom. SampPerLoop*). This simple calculation assumes that the signal should pass through resonance with 5 time constants. It is actually recommended that the user use AT LEAST 5 time constants to sweep through resonance. I would suggest multiplying both the recommended parameters by a value in between 1.5 to 2. This allows one to sweep through resonance with 7.5 to 10 time constants, respectively. This is desired because it allows one to more easily remove random noise.

8. **ENDOR PARAMETERS:** This allows the user to perform a low-field ENDOR measurement and can only be used with the low-field spectrometer. This is still a work in progress and is therefore not in a user friendly state. For more information on running the spectrometer in this mode, see the section entitled *Low-Field ED ENDOR* or contact Corey for assistance.

9. **ARBITRARY WAVEFORM:** This feature allows the user to utilize the additional signal generator of the NI DAQ. The user must select the *Waveform Enable* button for this function to work. The waveform type, frequency

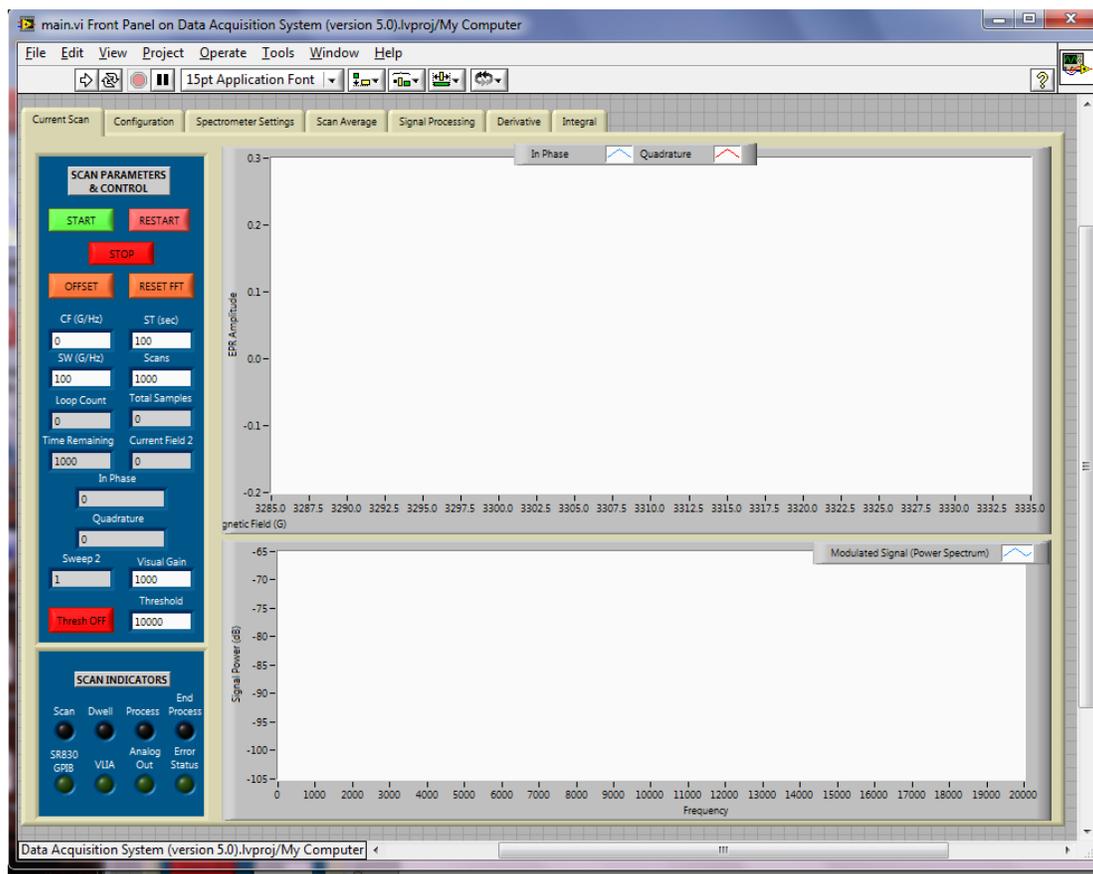
(bandwidth limited), offset, amplitude, and phase can be set within this block. This function can very useful for EDMR measurements which utilize a separate signal to bias the device under test. An example of this is spin dependent charge pumping.

10. SIGNAL PROCESSING: All the signal processing controls and results are located in the *Signal Processing* tab. This section allows the user to select from 3 different filters (or none). These filters are the *Windowed Averaging Filter*, *Savitzky Golay Filter*, and the *Adaptive Signal Averaging (ASA)* algorithm. If the ASA is to be used, the settings displayed in the *ASA/Window Params* block are recommended for first time users. To find out more about the ASA, the user is referred to the papers, C.J. Cochrane, **195**, pp. 17-22, (2008) and C.J. Cochrane, *Rev. Sci. Instrum.*, **83**, 105108, (2012). If it is desired to use *windowed averaging filter*, the window length is simply set by the *Filter Order* parameter in the *ASA/Window Params* block. If the *Savitzky Golay Filter* is to be used, configure the parameters located in the *Savitzky Golay Params* block. For information on this type of filter, see A. Savitzky, M. Golay, *Anal. Chem.*, 36, 8, p. 1627, (1964). The far right block allows the user to select either the *Filtered Average* or *Unfiltered Average* to be integrated or differentiated (displayed on the derivative and integral tabs). The *LPF Fc* allows the user to set the cutoff frequency for the filter used for removing high frequency noise from the differentiated signal. The *ESR III* and *ESR II Q* are integrated intensity indicators for the in-phase and quadrature averaged signals displayed on the *Integral* tab. Note that these two indicators are only meaningful if a SINGLE signal is being measured in which the ENTIRE width of the signal is present. The reason for this is because the processing algorithm entails an integration, baseline correction, offset removal, and then a summation of all samples present.



C. Running the EDMR Spectrometer

The snapshot below is a picture of the *Current Scan* Tab which is used to run an EDMR experiment. A description of how to utilize this GUI is documented below. **Note: Be sure to enter all information pertinent to experiment before starting the program. This will ensure proper initialization of data and guarantee all information will be saved to the data files.**



-  Starts the program for the EDMR spectrometers.
-  Stops the program for the EDMR spectrometers.

START: Starts the scan. Ensure that the current field is stabilized before starting the scan.

RESTART: Restarts the scan. The program will not commence until the START button is pressed again.

STOP: Stops the program. Serves same purpose as . Use this stop when *Arbitrary Waveform* is enabled

OFFSET: Removes the DC offset at time of button press. Recommend only using with virtual lockin.

RESET FFT: Resets the averaging of continuous FFT blocks.

CF (G/Hz): Center field (in Gauss). (Serves as the center frequency (in Hz) for low-field ENDOR)

ST (sec): Sweep time in seconds.

SW (G/Hz): Sweep width (in Gauss). (Serves as the sweep frequency (in Hz) for low-field ENDOR)

Scans: Number of scans to average. (If the experiment needs to run more scans to average out the noise, the averaging process will continue; however, the user will NOT be able to view and or discard the additional scans in the display on *Scan Average* tab. Though, the files will continue to be saved with the updated average and individual scans.)

Visual Gain: Multiplicative constant applied to raw data displayed on the *Current Scan* tab. (This constant does not actually manipulate the amplitude of the data. It is simply used to scale the data for displaying purposes because Labview's autodisplay features does not work well with data that is of low level.)

Thresh ON/OFF: If ON, the program automatically restarts the current scan if measured amplitude is greater than the "Threshold" parameter.

Threshold: If "Thresh ON" is set, the program will restart the current scan if it detects that the amplitude of the raw voltage sampled by the DAQ multiplied by the visual gain (this is the scale of the graph located on the same page) is greater than this parameter. If "Thresh OFF" is set, this parameter is ignored.

Upper Graph: Displays the in-phase and out-of-phase demodulated components of the LIA. The units of this graph is the raw voltage sampled by the DAQ multiplied by the visual gain.

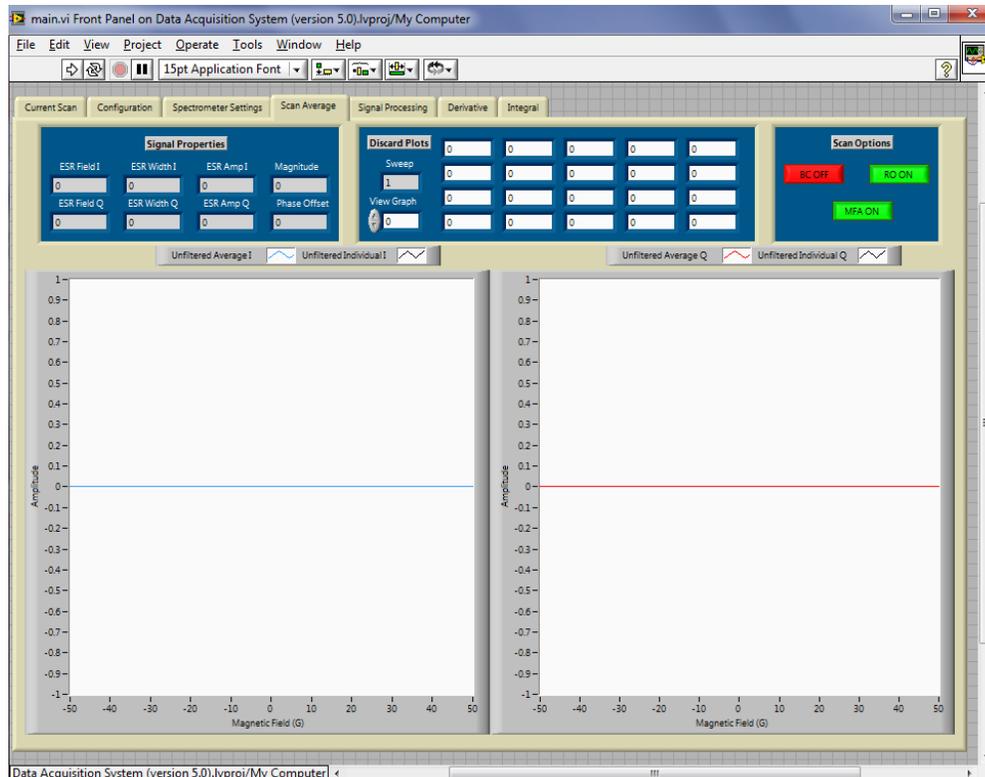
Lower Graph: Displays the FFT of the amplified modulated signal from the output of the preamp. This graph is very useful for determining optimum modulation frequencies, debugging spectrometer noise sources, and determining if passage effects are present.

Once the software is scanning, the user has the ability to monitor the scan average and individual scans for both the in-phase and quadrature components in the *Scan Average* tab as illustrated below. It also allows the user to monitor the center field, width, and amplitude of both averaged components. Additionally, using these measured parameters, the software calculates the magnitude and phase offset of the signal set. Note that all measurements on this tab are displayed in the units that were set in the *Spectrometer Settings* tab. The scale of the signals will be accurate as long as the preamp sensitivity and lockin sensitivity were properly set. On the far right, the user has the ability to configure three additional options.

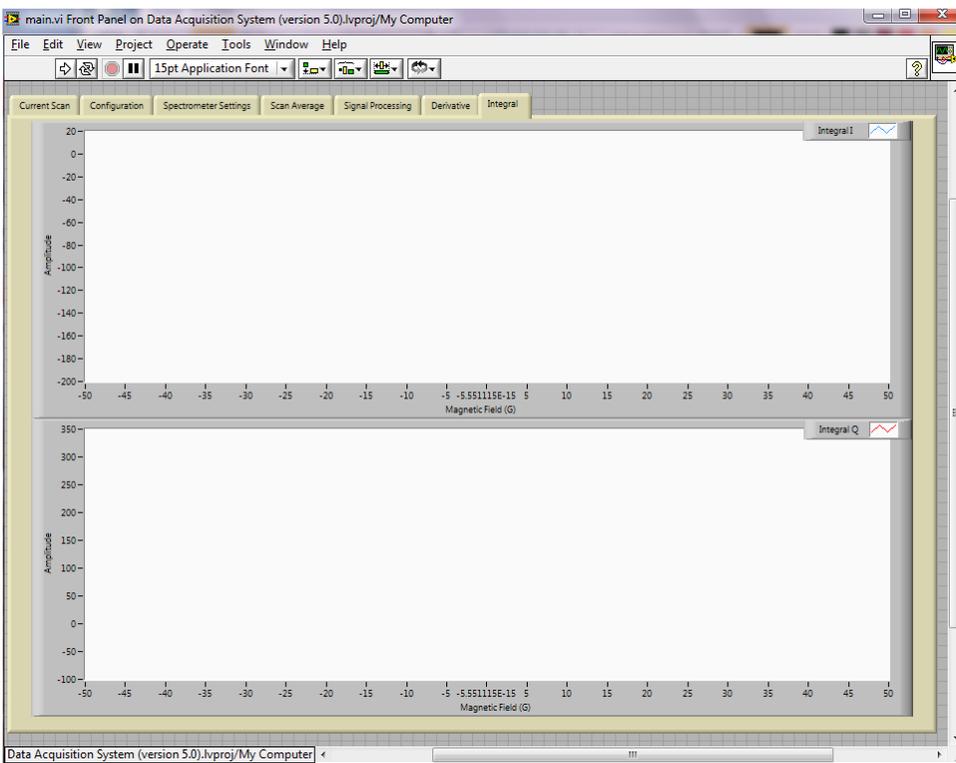
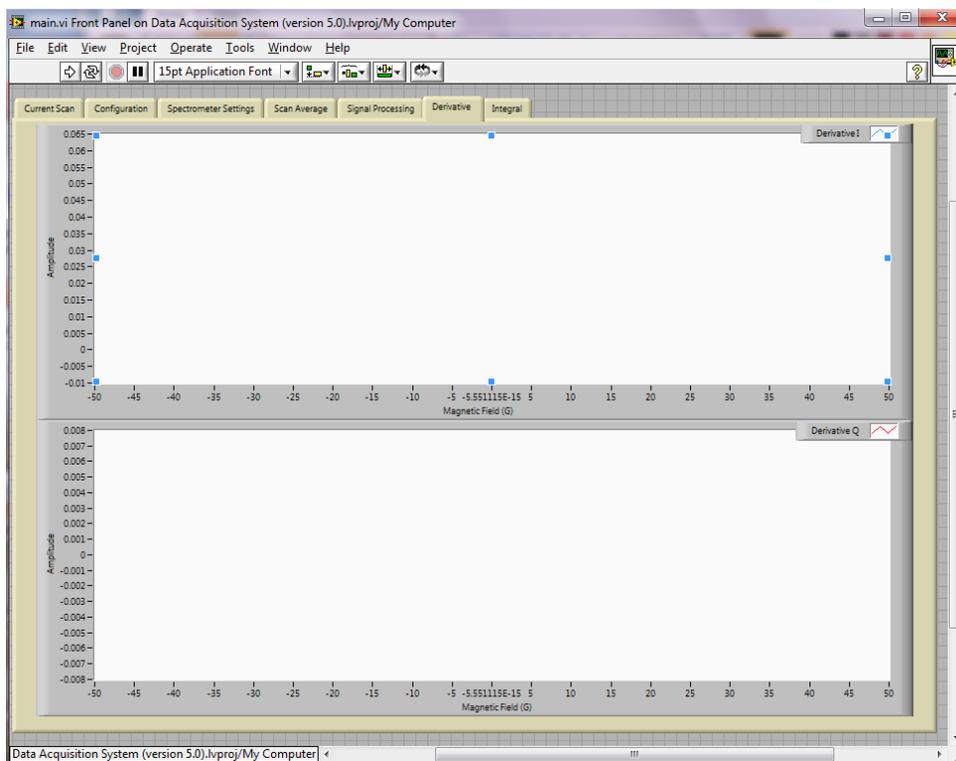
BC ON/OFF: This function performs a linear baseline correction to every individual scan before it is averaged.

RO ON/OFF: This function removes the offset of every individual scan before it is averaged.

MFA ON/OFF: This function performs a magnetic field adjustment to the data set. Always have this set to ON.



And finally, the last two tabs display the derivative and integral of the in-phase and quadrature components for either the filtered average or the unfiltered average (controlled by a button on the *Signal Processing* Tab)



D. Software Configuration for a Bruker

The software is configured from the second and third tabs in the LV GUI. They are labeled *Configuration* and *Spectrometer Settings*. So that the entered parameters do not have to be set each time the software is opened, one can make them default by (1) entering the desired default value of the variable, (2) right clicking on that variable, (3) selecting *data operations*, and finally (4) selecting *make current value default* for each parameter that is desired to be changed. For a first time spectrometer setup, ensure that the following two parameters are configured properly.

1. SPECTROMETER SELECTION: Under the *SPECTROMETER INFO* section of the *Configuration* tab, select the spectrometer in which the software is to be run. The user can select from the following EPR spectrometers:

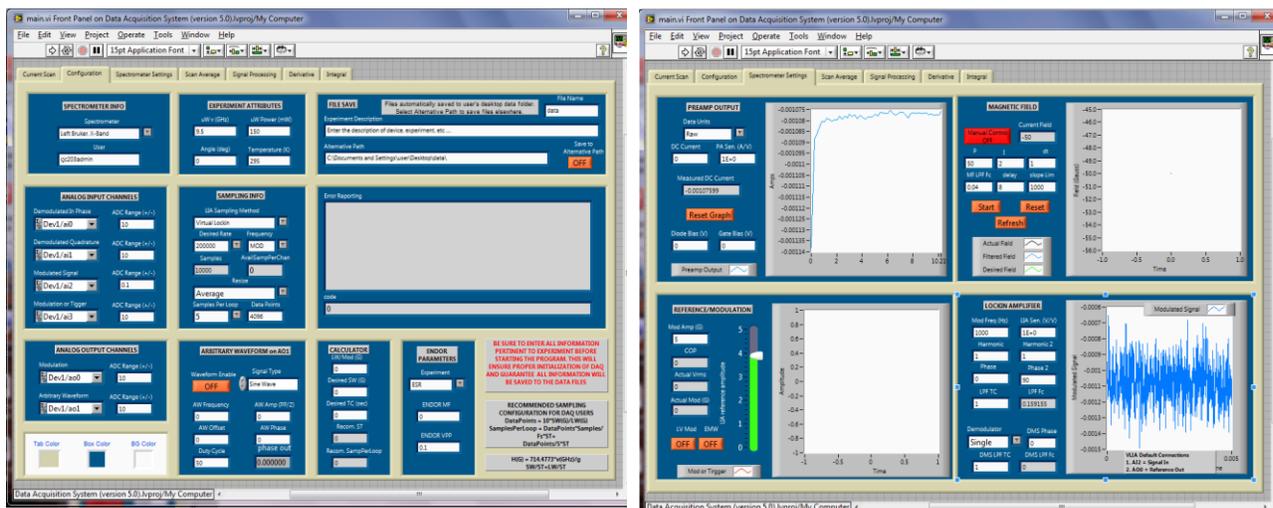
Left Bruker, X-Band: Bruker located on the left side of the lab.

Right Bruker, X-Band : Bruker located on the right side of the lab.

EMX Bruker, X-Band: Bruker located in the center of the lab. The software is not currently compatible with this spectrometer but should be easily adapted.

2. FILE SAVE: Also under the *SPECTROMETER INFO* section of the *Configuration* tab, type in the name of the user that is signed on to the computer so that the software knows where to save the generated Labview files. This location will be *C:\Documents and Settings\user\Desktop\data*. If a user does not have a user account or if it is desired to save the .lvm files to a different location, simply turn on the *Save to Alternative Path* button and change the *Alternative Path* setting under the *FILE SAVE* block in the *Configuration* tab. Here, the user is also able to provide a description of the device and experimental details under *Experiment Description*.

Important Note: If a user enters a user name that is different than the user account in which the computer is logged on, LAVIEW will stop at the end of the scan because it is attempting to save the .lvm file to a location in which doesn't exist or current user has access to. Ensure that this is properly set up before run time.



3. **LOCKIN AMPLIFIER:** Because the Bruker spectrometers are relatively old, the built in lockin amplifiers are outdated. Therefore, the user should utilize an external lockin amplifier which has the capability to generate its own reference signal. Therefore, the lockin settings should be configured for *NI6259* or *SR830 GPIB* where the *Virtual Lockin* option is not recommended because the interface to the Bruker mod coils requires a special type of cable. This makes driving the coils with a source other than Bruker's own somewhat difficult. Also, even though the *Virtual Lockin* has been run successfully at 100kHz (typical modulation for EPR measurements), it has not been rigorously verified to consistently work at these high modulation frequencies.

- a. **NI6259:** This setting is used if the NI6259 DAQ (or equivalent) is used to sample the output of an external hardware lockin such as the SR830. The following controls need to be configured for proper functioning:

SAMPLING INFO in the *Configuration* Tab

1. **LIA Sampling Method:** set to *NI6259*.
2. **Sampling Rate:** If it is desired to calibrate and view the FFT of the modulated signal, set the rate to a value that is approximately x10 the modulation frequency. However, if the user can do without this feature, it is only required that the DAQ sample the data at, 10 Hz to 1kHz.
3. **Frequency:** Set this parameter to *MOD* which signifies that the user is in modulation mode. The *DC* mode is not available with the Bruker spectrometer.
4. **Resize:** This parameter configures the resampling that occurs after the signal is sampled by the DAQ. Essentially, the DAQ reads data in by "chunks", where the number of samples in each chunk is determined internally and is based on the configured sampling rate. When the signal is sampled, the number of samples that are available are far more than what are actually needed to represent a DC signal. Therefore, the user can *average* or *decimate* (pick evenly spaced samples from chunk) these chunks. The averaging provides an additional noise reduction.
5. **Samples per Loop:** Determines the number of samples per loop (or "chunk" as describe above) that the software extracts from the fully sampled signal at each loop iteration. It is recommended that the user ensures that this value is set to the next available integer setting from the number calculated in *Recom. SampPerLoop* parameter in the calculator block on the same tab. Using this value ensures that the signal is "well represented".
6. **Data Points:** The number of samples that represents the final data length. Setting this to its default value of 4096 is sufficient for just about all experiments. Ensure that this is not set too high (>10,000) because the computer may have a difficult time processing the data.

LOCKIN AMPLIFIER Block in the *Spectrometer Settings* Tab

1. **Mod Freq (Hz):** This is not a control, but rather only an indicator which saves the entered value into the saved .lvm file.
2. **Sensitivity (V/V):** Sensitivity of the external lockin amplifier. Ensure to enter this for proper unit conversion.
3. **Harmonic:** Harmonic of the external lockin amplifier. This is not a control and is used to record the lockin harmonic used during the experiment.
4. **Harmonic 2:** This serves no purpose when the lockin mode is set to *NI6259*.
5. **Phase:** Phase of the external lockin amplifier. This is not a control and is used to record the lockin phase used during the experiment.
6. **Phase 2:** This serves no purpose when the lockin mode is set to *NI6259*.
7. **LPF TC:** Low pass filter time constant of the external lockin amplifier. This is not a control and is used to record the lockin time constant used during the experiment
8. **Demodulator:** This serves no purpose when the lockin mode is set to *NI6259*.

REFERENCE/MODULATION Block in the *Spectrometer Settings* Tab

1. **Mod Amp (G):** This is not a control but rather a parameter the user can set for the software to save to the data file.

2. **Lockin Reference Amplitude:** This controls the amplitude of the modulation signal that is generated in Labview ONLY IF the *LV Mod* button is selected. This allows Labview to provide the reference signal used for modulation if the external lockin does not provide it. See below to determine how to select the channel in which it is generated.

ANALOG INPUT CHANNELS Block in the *Configuration* Tab

1. **Demodulated In Phase:** Sets the channel on the BNC2110 to sample the in-phase output signal of the external lockin. The default source is AI0.
2. **ADC Range (+/-):** Sets the limits of the DAQ for the sampling the in-phase component of the external lockin. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .
3. **Demodulated Quadrature:** Sets the channel on the BNC2110 to sample the quadrature output signal of the external lockin. The default source is AI1.
4. **ADC Range (+/-):** Sets the limits of the DAQ for the Modulated Signal. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .
5. **Modulated Signal:** Sets the channel on the BNC2110 to sample the modulated signal from the microwave bridge. The default source is AI2.
6. **ADC Range (+/-):** Sets the limits of the DAQ for the Modulated Signal. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .
7. **Modulation or Trigger:** Sets the channel on the BNC2110 to sample the trigger source provided by the Bruker. The default source is AI3.
8. **ADC Range (+/-):** Sets the limits of the DAQ for the trigger source. If the NI6259 is used, the user has the option to select the voltage ranges of ± 10 , ± 5 , ± 2 , ± 1 , ± 0.5 , ± 0.2 , ± 0.1 .

ANALOG OUTPUT CHANNELS Block in the *Configuration* Tab

1. **Modulation:** Sets the channel on the BNC2110 which will output the reference modulation signal ONLY if the *LV Mod* button is selected. The default source is AO0.
2. **ADC Range (+/-):** Sets the limits of the DAQ for the Modulation Signal.

- b. **SR830 GPIB:** Enables sampling of data through the GPIB interface of the SR830 lockin amplifier. Even though this is a convenient hardware feature, it is not recommended for use with the spectrometers as only one sample can be obtained per loop cycle which limits the sampling rate of the data. The fastest loop rate of the software is configured to 10 (or sometimes 5) iterations per second which in some cases, may not be enough samples to fully represent a signal if the scan time is fast. When using this setting, use the steps described above (for configuring *NI6259*) to configure the software with the exception of the following:

SAMPLING INFO in the *Configuration* Tab

1. **LIA Sampling Method:** set to *SR830 GPIB*
2. **Resize:** Set to *None*.
3. **Samples per Loop:** Set to 1.

4. **PREAMP OUTPUT:** This set of controls and indicators are located under the *Spectrometer Settings* tab.

Data Units: This configures the software to save the data according to the units selected. For EPR, ALWAYS set this value to a *RAW*.

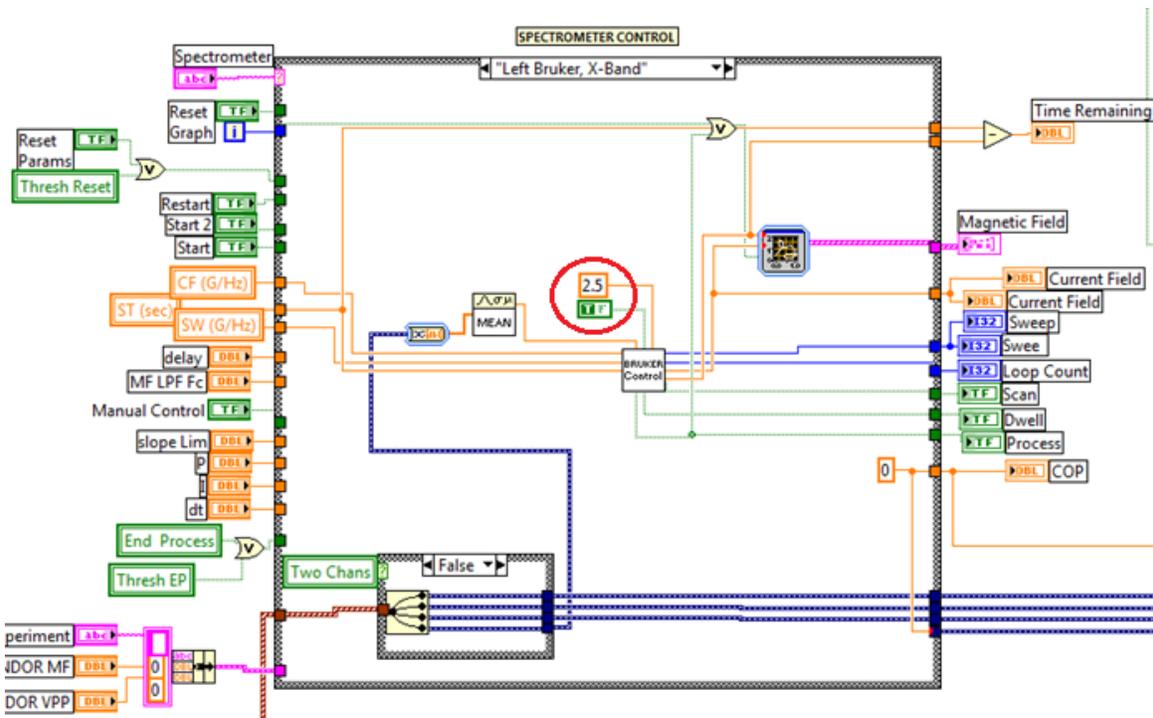
DC Current: Not used in EPR. Can set equal to 0.

PA Sens (V/A): Not used in EPR. Can set equal to 0.

Diode Bias: Not used in EPR. Can set equal to 0

Gate Bias: Not used in EPR. Can set equal to 0

5. MAGNETIC FIELD CONTROL: There are no steps needed to configure the magnetic field control because the Bruker hardware performs this task. As a result, the time in between scans is fixed in these spectrometers and cannot be modified which is not the case for the purely digitally controlled EDMR spectrometers. The software knows the approximate field in which the Bruker is scanning because it samples a trigger. The left Bruker is a “logic high” trigger while the right Bruker is a “logic low” trigger. For example, the left Bruker is scanning when the trigger is “high” (5 volts) and is not scanning when it is “low” (0 volts). Therefore, the code compares the measured trigger signal to a specified threshold voltage of 2.5 volts to determine whether the spectrometer is scanning. See the red circled code below for the implementation of this logic. If it is desired to configure the software to run on the Bruker EMX, simply select this spectrometer (I already made an option for it on the drop down list) and change the two parameters to whichever the EMX is equipped to run. Ensure that the code is saved before exiting.



6. EXPERIMENT ATTRIBUTES: These are not controls but rather parameters that can be set which are saved to the .lvm files. It is good practice to set these parameters for every experiment.

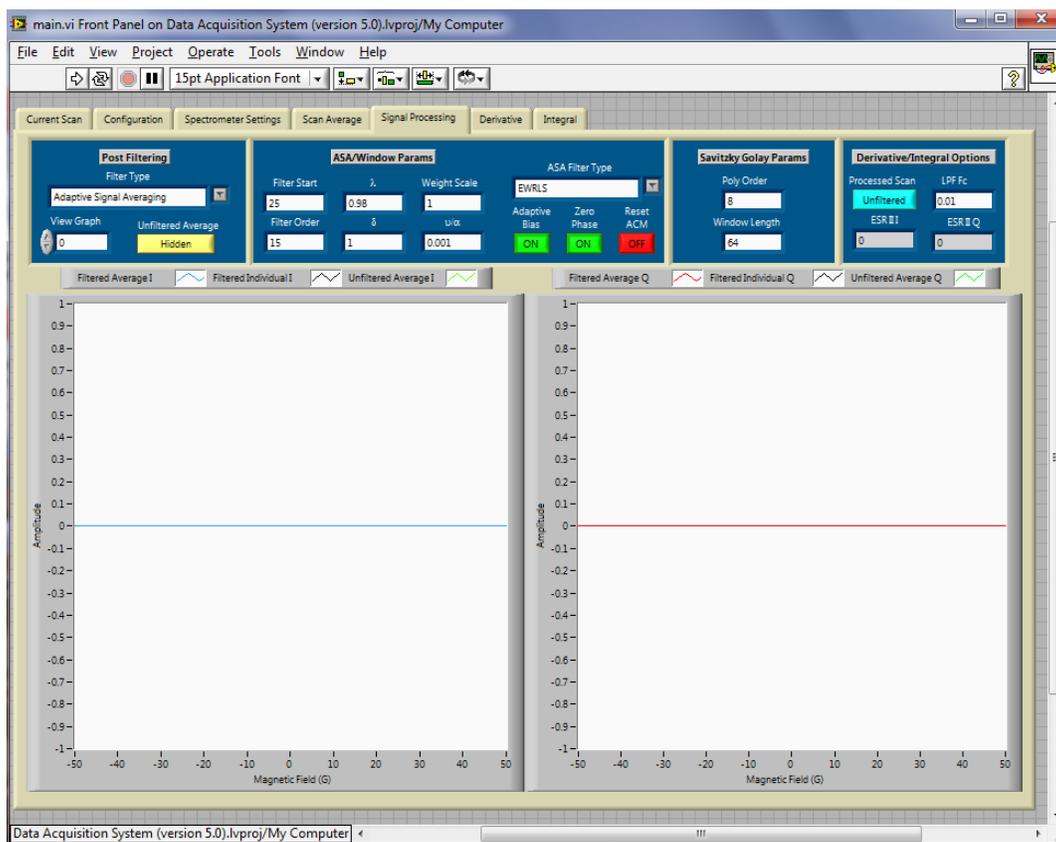
7. CALCULATOR: Allows the user to enter the desired line width (LW), sweep width (SW), and time constant (TC) to obtain a recommended sweep time (ST) and samples per loop (*Recom. SampPerLoop*). This simple calculation assumes that the signal should pass through resonance with 5 time constants. It is actually recommended that the user use AT LEAST 5 time constants to sweep through resonance. I would suggest multiplying both the recommended parameters by a value in between 1.5 to 2. This allows one to sweep through resonance with 7.5 to 10 time constants, respectively. This is desired because it allows one to more easily remove random noise.

8. ENDOR PARAMETERS: This allows the user to perform a low-field ENDOR EDMR measurement and can only be used with the low-field spectrometer. With minimal effort, this program can be used in the Bruker machines to run ENDOR EPR.

9. ARBITRARY WAVEFORM: This feature allows the user to utilize the additional signal generator of the NI DAQ. The user must select the *Waveform Enable* button for this function to work. The waveform type, frequency (bandwidth limited), offset, amplitude, and phase can be set within this block. Though this function may be very

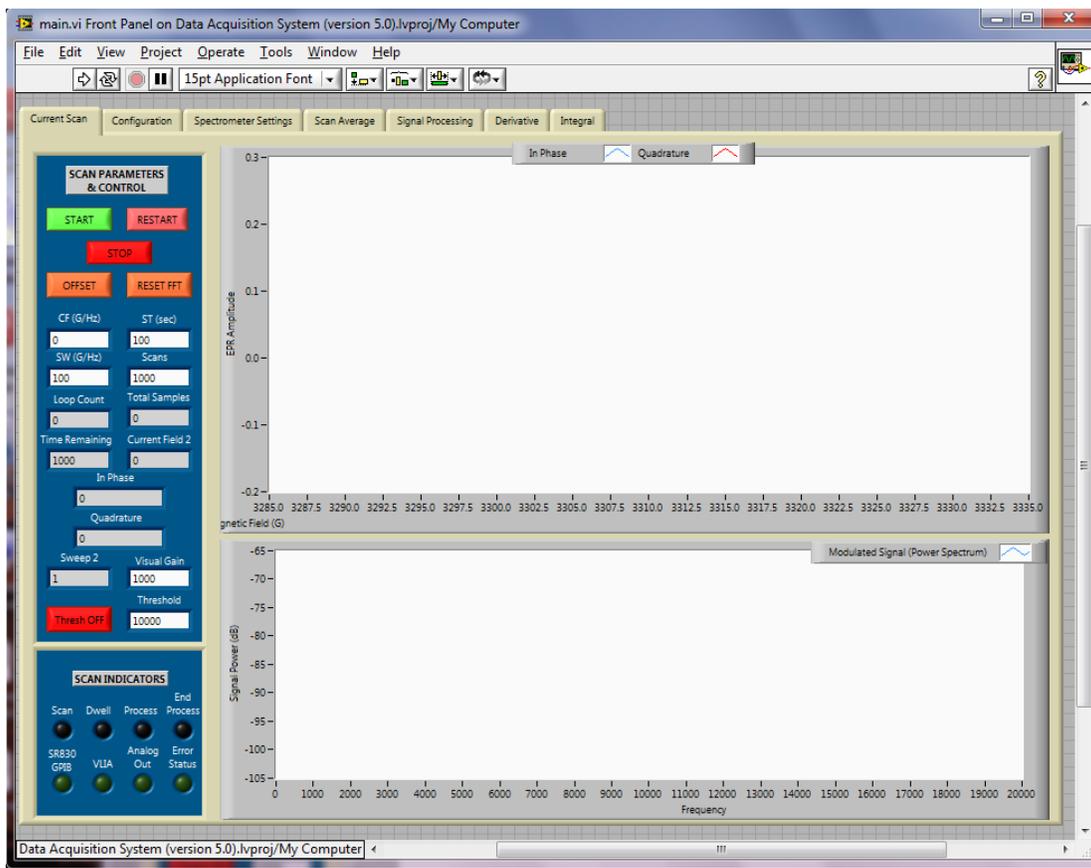
useful for EDMR measurements, it may not be that useful for conventional EPR measurements as additional waveforms are not usually needed.

10. SIGNAL PROCESSING: All the signal processing controls and results are located in the *Signal Processing* tab. This section allows the user to select from 3 different filters (or none). These filters are the *Windowed Averaging Filter*, *Savitzky Golay Filter*, and the *Adaptive Signal Averaging (ASA)* algorithm. If the ASA is to be used, the settings displayed in the *ASA/Window Params* block are recommended for first time users. To find out more about the ASA, the user is referred to the papers, C.J. Cochrane, **195**, pp. 17-22, (2008) and C.J. Cochrane, *Rev. Sci. Instrum.*, **83**, 105108, (2012). If it is desired to use *windowed averaging filter*, the window length is simply set by the *Filter Order* parameter in the *ASA/Window Params* block. If the *Savitzky Golay Filter* is to be used, configure the parameters located in the *Savitzky Golay Params* block. For information on this type of filter, see A. Savitzky, M. Golay, *Anal. Chem.*, **36**, p. 1627, (1964). The far right block allows the user to select either the *Filtered Average* or *Unfiltered Average* to be integrated or differentiated (displayed on the derivative and integral tabs). The *LPF Fc* allows the user to set the cutoff frequency for the filter used for removing high frequency noise from the differentiated signal. The *ESR II I* and *ESR II Q* are integrated intensity indicators for the in-phase and quadrature averaged signals displayed on the *Integral* tab. Note that these two indicators are only meaningful if a SINGLE signal is being measured in which the ENTIRE width of the signal is present. The reason for this is because the processing algorithm entails an integration, baseline correction, offset removal, and then a summation of all samples present.



E. Running the Bruker Spectrometer

The snapshot below is a picture of the *Current Scan* Tab which is used to run an EPR experiment. A description of how to utilize this GUI is documented below. **Note: Be sure to enter all information pertinent to experiment before starting the program. This will ensure proper initialization of data and guarantee all information will be saved to the data files.**



-  Starts the program for the EPR spectrometers.
-  Stops the program for the EPR spectrometers.

START: Unused when in use with a Bruker spectrometer. Press the start button on the Bruker control panel to start the experiment.

RESTART: Unused when in use with a Bruker spectrometer. Press the reset button on the Bruker control panel to restart the scan.

STOP: Stops the program. Serves same purpose as .

OFFSET: Removes the DC offset at time of button press. Recommend only using with virtual lockin.

RESET FFT: Resets the averaging of continuous FFT blocks.

CF (G/Hz): Center field (in Gauss). (Serves as the center frequency (in Hz) for ENDOR)

ST (sec): Sweep time in seconds.

SW (G/Hz): Sweep width (in Gauss). (Serves as the sweep frequency (in Hz) for ENDOR)

Scans: Number of scans to average. (If the experiment needs to run more scans to average out the noise, the averaging process will continue; however, the user will NOT be able to view and or discard the additional scans in the display on *Scan Average* tab. Though, the files will continue to be saved with the updated average and individual scans.)

Visual Gain: Multiplicative constant applied to raw data displayed on the *Current Scan* tab. (This constant does not actually manipulate the amplitude of the data. It is simply used to scale the data for displaying purposes because Labview's autodisplay features does not work well with data that is of low level.)

Thresh ON/OFF: Unused when in use with a Bruker spectrometer.

Threshold: Unused when in use with a Bruker spectrometer.

Upper Graph: Displays the in-phase and out-of-phase demodulated components of the LIA. The y axis scale of this graph are the raw voltage sampled by the DAQ multiplied by the visual gain.

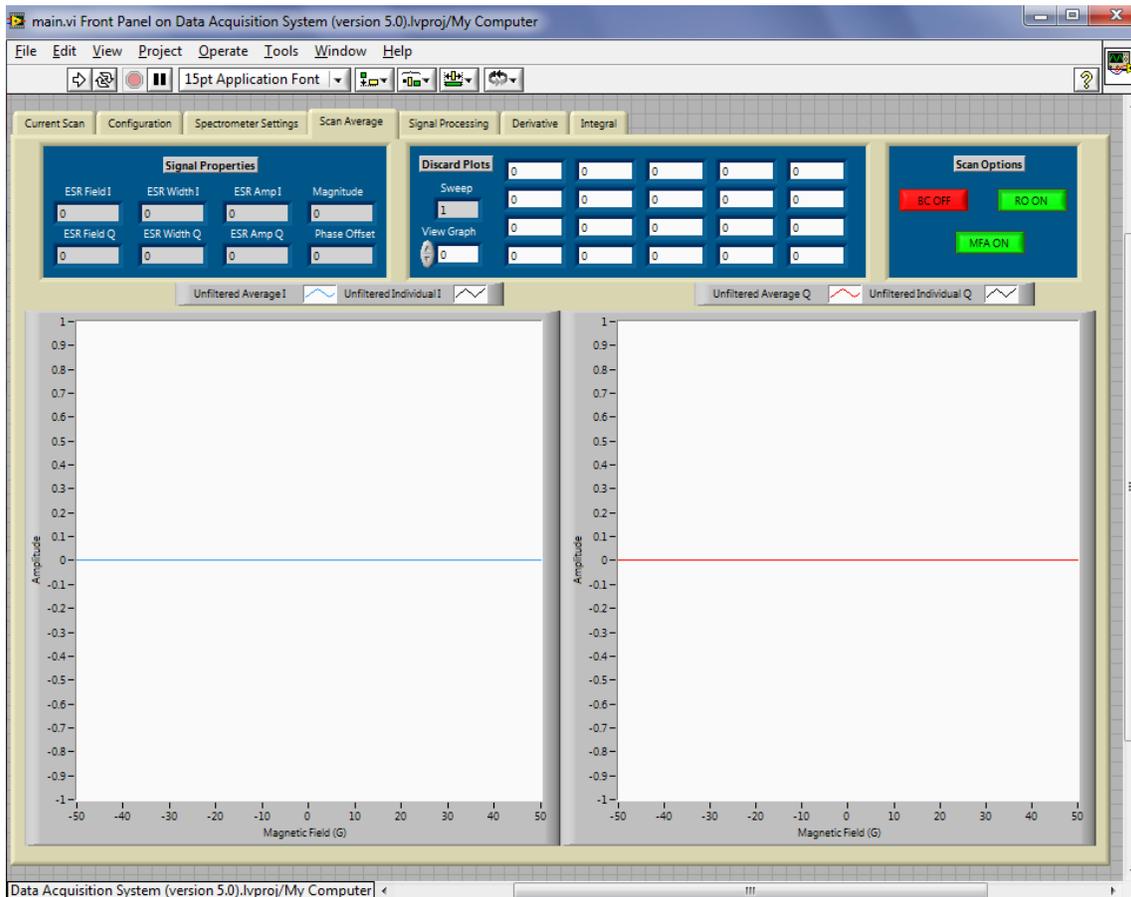
Lower Graph: Displays the FFT of the amplified modulated EPR signal from the output of the microwave bridge. This graph is very useful for determining optimum modulation frequencies, debugging spectrometer noise sources, and determining if passage effects are present.

Once the software is scanning, the user has the ability to monitor the scan average and individual scans for both the in-phase and quadrature components in the *Scan Average* tab as illustrated below. It also allows the user to monitor the center field, width, and amplitude of both averaged components. Additionally, using these measured parameters, the software calculates the magnitude and phase offset of the signal set. Note that all measurements on this tab are displayed in the units that were set in the *Spectrometer Settings* tab. The scale of the signals will be accurate as long as the preamp sensitivity and lockin sensitivity were properly set. For the Bruker spectrometers, the units should be set to *Raw*. On the far right, the user has the ability to configure three additional options.

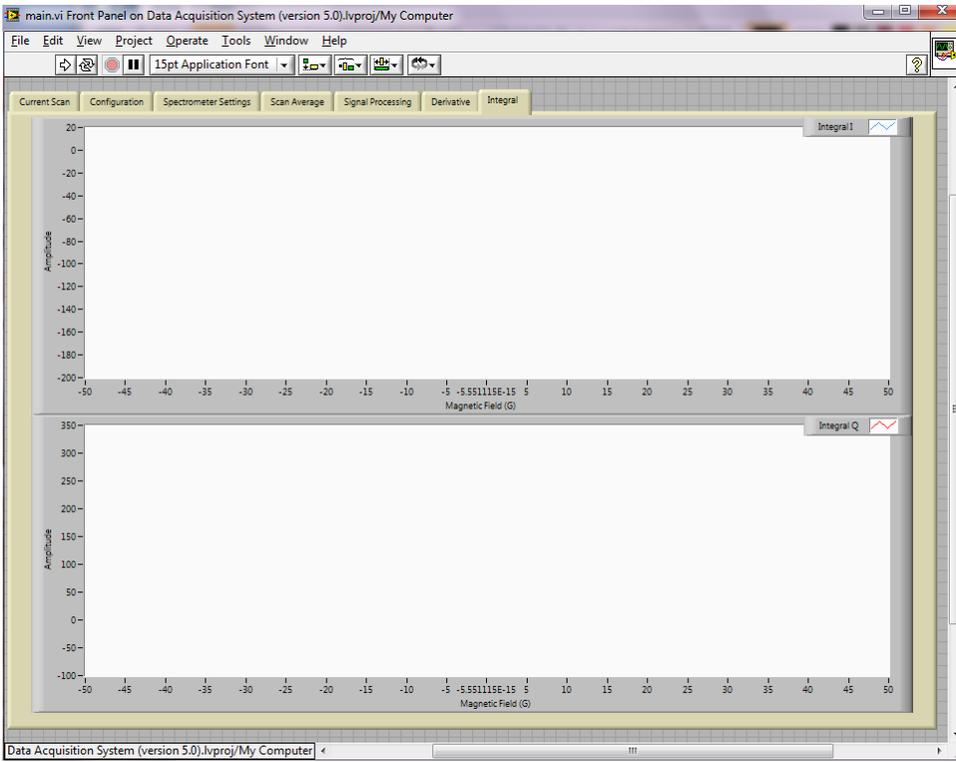
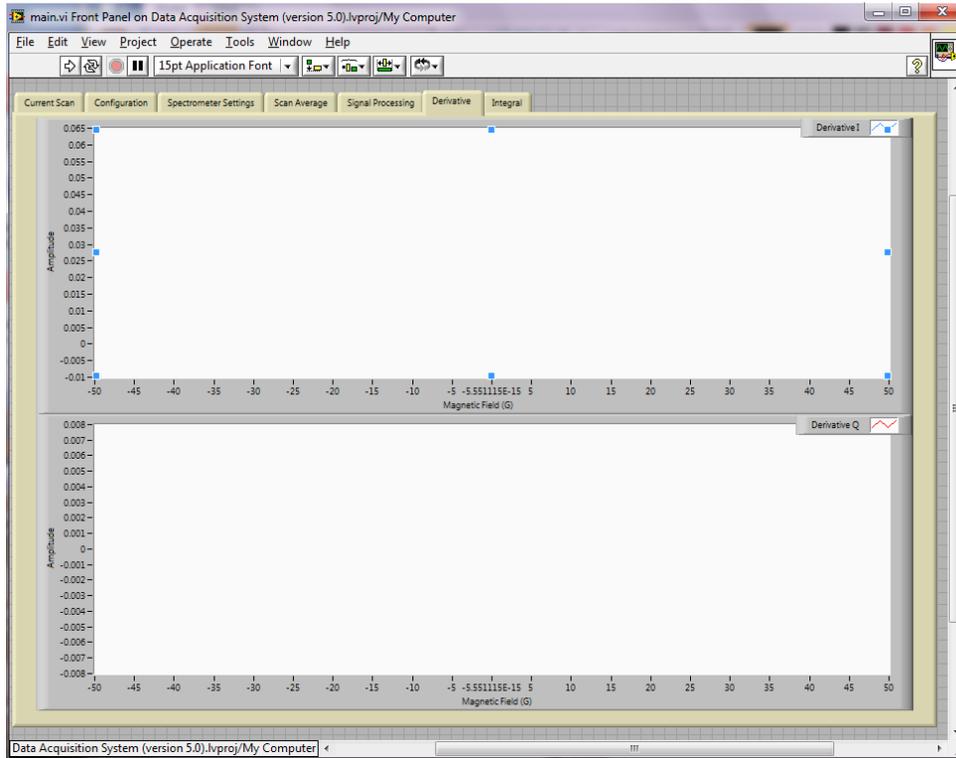
BC ON/OFF: This function performs a linear baseline correction to every individual scan before it is averaged.

RO ON/OFF: This function removes the offset of every individual scan before it is averaged.

MFA ON/OFF: This function performs a magnetic field adjustment to the data set. Because the magnetic field is not actually being measured, this function serves no purpose when in use with a Bruker spectrometer.



And finally, the last two tabs display the derivative and integral of the in-phase and quadrature components for either the filtered average or the unfiltered average (controlled by a button on the *Signal Processing* Tab)



F. Labview Output Files

The software is written to save a *.lvm file after each scan is complete. The heading for each columns represent:

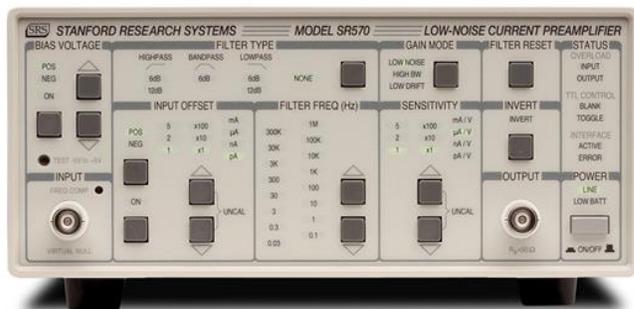
- Field: magnetic field (G)
- avg_i: unfiltered in-phase average
- avg_q: unfiltered quadrature average
- avg_if: filtered in-phase average
- avg_qf: filtered quadrature average
- ind_i: unfiltered in-phase individual
- ind_q: unfiltered quadrature individual
- ind_if: filtered in-phase individual
- ind_qf: filtered quadrature individual
- der_i: derivative of in-phase average
- int_i: integral of in-phase average
- der_q: derivative of quadrature average
- int_q: integral of quadrature average

	A	B	C	D	E	F	G	H	I	J	K	L	M
1	Date:	2/8/2013 10:52											
2	Sample:	Austria Microsystems SiO2											
3													
4	DAS Version Num		5										
5	Scans		45										
6	Sweep Time (s)		60										
7	Center Field (G)		47										
8	Sweep Width (G)		172										
9	Mod Freq (Hz)		1600										
10	Mod Ampl (G)		2										
11	uW Freq (GHz)		0.308										
12	uW Power (mW)		0.26										
13	Gate Bias (V)		1.6										
14	Diode Bias (V)		0.32										
15	DC Current (A)		4.34E-07										
16	PA Sens (A/V)		2.00E-08										
17	Phase (deg)		-59.3										
18	LIA Sens (V)		1.00E+00										
19	Time Const (s)		0.15										
20	Angle (deg)		0										
21	Temperature (K)		295										
22	Harmonic		1										
23	Units		pA										
24													
25	Field	avg_i	avg_q	avg_if	avg_qf	ind_i	ind_q	ind_if	ind_qf	der_i	int_i	der_q	int_q
26	-3.90E+01	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	1.63E-05	0.00E+00	-4.80E-06	0.00E+00
27	-3.90E+01	7.92E-02	1.57E-01	0.00E+00	0.00E+00	5.56E-01	-1.45E+00	0.00E+00	0.00E+00	5.61E-06	-7.18E-01	-6.56E-05	1.81E-01
28	-3.89E+01	1.18E-01	1.47E-01	0.00E+00	0.00E+00	6.82E-01	-1.02E+00	0.00E+00	0.00E+00	-2.07E-05	-1.40E+00	-1.30E-04	3.52E-01
29	-3.89E+01	1.63E-01	1.59E-01	0.00E+00	0.00E+00	6.93E-01	-6.87E-01	0.00E+00	0.00E+00	-6.45E-05	-2.03E+00	-2.05E-04	5.35E-01

G. Suggestions for Preamp Settings in EDMR

A few rules of thumb should be followed when performing an EDMR measurement with this form of preamplifier (e.g. SR570).

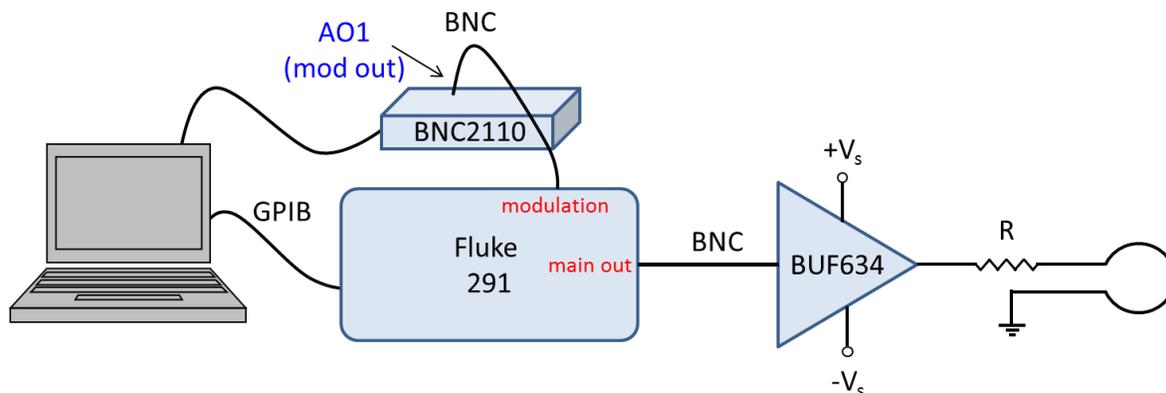
1. The first stage gain setting should be set as high as possible such that it doesn't overload the preamp. If a particular gain setting is desired which overloads the preamp, use the input offset to cancel this current. (Note that adding current to cancel the DC input current will NOT completely remove the flicker noise associate the large input currents. The technique simply allows for the utilization of larger gain settings without overloading the preamplifier front end circuitry.)
2. Use a second stage high pass filter to block the DC component (and much of the flicker noise) that gets passed from the first stage. This will allow for the modulated signal to be further amplified without overloading the following gain stage. Also, this removes the DC offset and allows for the best dynamic range setting of the ADC. The cutoff frequency of the filter should be set to $\approx 10\times$ lower than the modulation frequency to prevent attenuation to the signal.
3. Chose a modulation frequency such that it is greater than the flicker noise but lower than bandwidth of the preamplifier (see chart below for the SR570 preamp specifications). I recommend using a 200-1.8kHz modulation frequency when using the SR570 (corresponds to a sensitivity of 100nA/V).
4. Use low pass (antialiasing) filters to prevent aliasing of the ADC. The cutoff frequency should be set higher than the modulation frequency but lower than the Nyquist rate (Sampling Rate in Hz / 2). If really high gains on the preamplifier are used, a low pass filter may not be necessary if sampling fast enough because the bandwidth limitation of the preamp will provide an 8dB/octave attenuation rolloff.
5. Ensure that the sample rate on the ADC is more than 10x that of the modulation frequency. (This provides 10 samples of data per modulation period, a value 5 times more than obtained when using the Nyquist rate.)
6. Ensure that the signal entering the ADC does not exceed the quantization limits for the ADC. This can be determined by the red indicator lights on the Stanford Instruments lock-in amplifiers or visually on the Labview GUI when utilizing the virtual lock-in amplifier. This will cause distortion in the EPR spectrum.



Sensitivity	LN BW (3 dB)	Noise/ $\sqrt{\text{Hz}^2}$
1 mA/V	1 Mhz	150 pA
100 uA/V	500 kHz	60 pA
10 uA/V	200 kHz	2 pA
1 uA/V	20kHz	600 fA
100 nA/V	2 kHz	100 fA
10 nA/V	200 Hz	60 fA
1 nA/V	15 Hz	10 fA
100 pA/V	10 Hz	5 fA
10 pA/V	10 Hz	5 fA
1 pA/V	10 Hz	5 fA

H. Low-Field Electrically Detected ENDOR

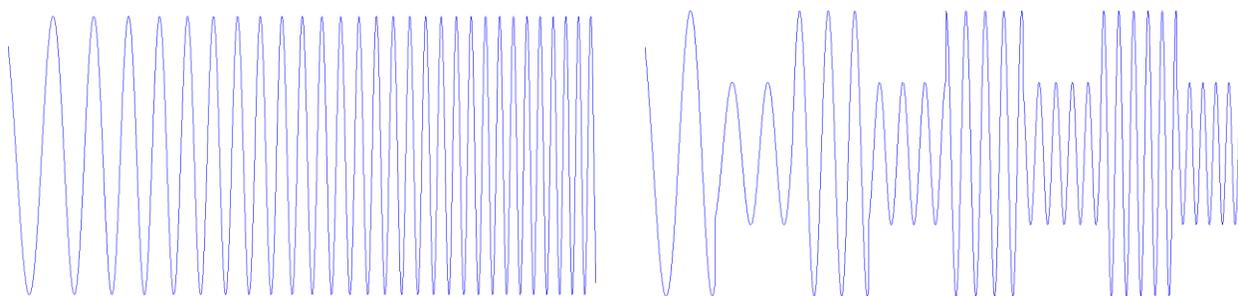
The low-field EDMR spectrometer can easily be setup to perform an electrically detected ENDOR experiment. The following diagram illustrates the required hardware setup.



As illustrated, the additional hardware components that are required are the Fluke 291 waveform generator and a circuit that consists of an amplifier (BUF634), power resistor, and coil (should be placed perpendicular to the B_0 and B_1 magnetic fields). Note that there is NO tuning or matching network needed because the internal signal reflections should not be a problem (if very short cables are used) due to the low frequency signals that needs to be generated to satisfy the resonance condition of the nuclei at low-magnetic fields. For example, if one satisfies the EPR resonance condition ($h\nu = g_e\mu_B B_0$) of a $g_e = 2$ signal with $\nu = 350 \text{ MHz}$, one would expect to observe resonance at $B_0 = 125 \text{ G}$. Assuming there are electron nuclear hyperfine interactions that are located near this field, the frequency at which the nuclear resonance is satisfied can be approximated using $\nu = g_N\mu_N B_0/h$. For a hydrogen atom ($g_N = 5.58$), the resonant frequency is approximately $\nu = 532 \text{ kHz}$ (for matrix ENDOR). Because Hydrogen has one of the higher nuclear g values, it will have a higher resonance frequency than most other nuclei. Therefore, if an ENDOR experiment is performed on the low field spectrometer at $B_0 = 125 \text{ G}$, the user should not configure a frequency sweep that is too much higher than this value. Some common nuclei that may be observed at these fields are ^{15}N ($g_N = 0.56, \nu = 53 \text{ kHz}$), ^{11}B ($g_N = 1.79, \nu = 171 \text{ kHz}$), or ^{31}P ($g_N = 2.26, \nu = 215 \text{ kHz}$).

Because the output of the Fluke 291 does not have the power to generate high currents, the BUF634 is used to buffer the input voltage and also amplify the output current (maximum of 250 mA). The power resistor is used so that the output coil (of minimal resistance) does not short the output of the amplifier to ground. The value of this resistor should be determined such that the maximum current through it does not exceed the BUF634's 250mA limit. For example, if the Fluke 291 generates a waveform that has a maximum voltage of +10V (ensure that the BUF634 is powered by a voltage greater than this but less than $\pm 18\text{V}$) and it is desired that the current through the loop be 225mA, then the value of the power resistor should be $R = V/I = 10\text{V}/225\text{mA} = 44 \Omega$. The power dissipation from this resistor is then calculated to be $P = I^2R = 0.225^2 \times 44 = 2.22 \text{ W}$. Therefore, a suitable and widely available resistor to use in this application would be a 50 Ω , 3W power resistor.

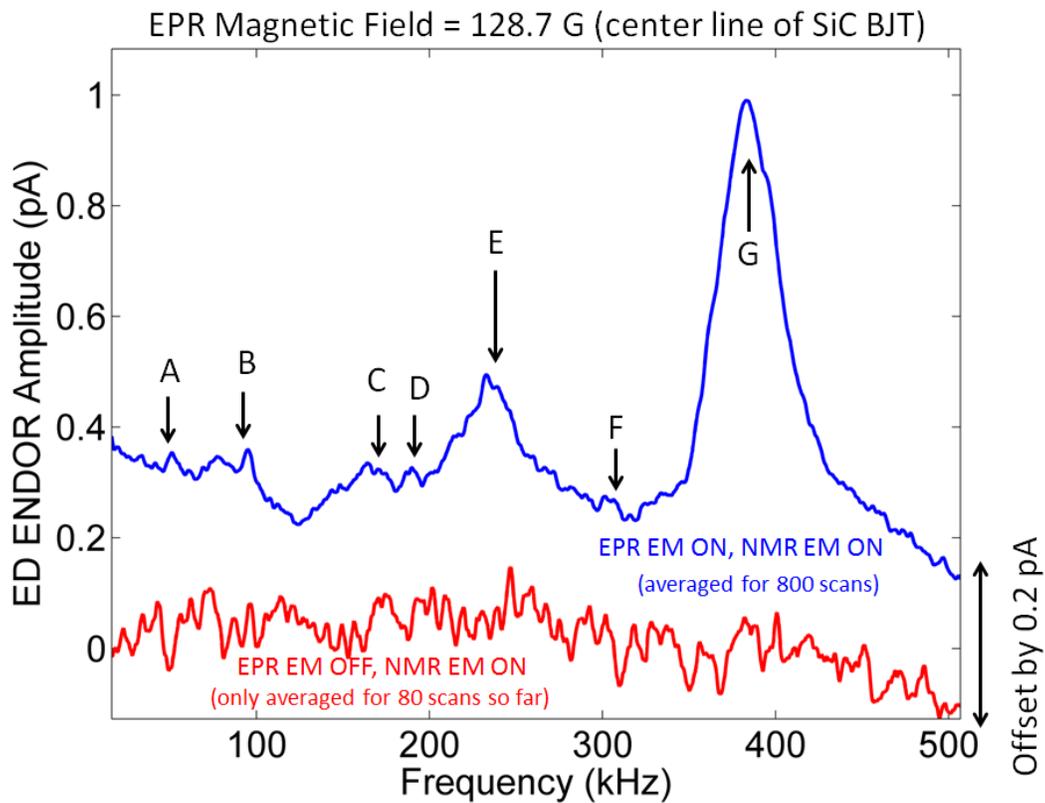
As indicated in the figure above, the software sends commands via the GPIB interface to the Fluke 291 to produce an amplitude modulated (square wave), frequency swept sine wave. The frequency sweep is used for searching for the nuclear resonance condition while the amplitude modulation enhances the sensitivity of the detection by allowing lock-in detection to be used (the lock-in can either be the virtual lockin or any external lockin). If the virtual lockin is used, the *Arbitrary Waveform Generator* section of the software is used to generate a square wave (or sine wave) on the AO1 pin from the BNC2110 to modulate the envelope of the frequency swept signal. See figure below for an example of a (left) frequency swept sine wave and (right) amplitude modulated, frequency swept sine wave. The envelope can then be demodulated via lockin detection. (Note that this technique will result in the detection of absorption spectra rather than derivatives as observed in conventional EDMR experiments when magnetic field modulation is utilized.)



The parameters that need to be configured to run the ENDOR experiment which differ from running the spectrometer in EDMR mode are:

- Experiment:** In the *ENDOR PARAMETERS* block under the *Configuration* tab
 - select *ENDOR*.
- ENDOR MF:** In the *ENDOR PARAMETERS* block under the *Configuration* tab
 - This configures the static field at which the ENDOR experiment is performed
 - **REMEMBER TO DISABLE THE MAGNETIC FIELD MODULATION!!!**
- ENDOR Vpp:** In the *ENDOR PARAMETERS* block under the *Configuration* tab
 - Configures the peak-to-peak amplitude of the frequency swept signal
- CF (G/Hz):** In the *SCAN PARAMETERS & CONTROL* block under the *Current Scan* tab
 - serves as the center frequency (in Hz) for ENDOR experiments
 - must be positive and cannot exceed maximum frequency of the Fluke 291 waveform generator
- SW (G/Hz):** In the *SCAN PARAMETERS & CONTROL* block under the *Current Scan* tab
 - serves as the sweep frequency (in Hz) for ENDOR experiments
 - must be positive and cannot exceed maximum frequency of the Fluke 291 waveform generator
- Waveform Enable:** In the *ARBITRARY WAVEFORM on AO1* block under the *Configuration* tab
 - Ensure that this is enabled
- Signal Type:** In the *ARBITRARY WAVEFORM on AO1* block under the *Configuration* tab
 - Set to square wave
- AW Frequency:** In the *ARBITRARY WAVEFORM on AO1* block under the *Configuration* tab
 - Ensure that this is set to the exact frequency in which you are demodulating at. (In other words, set this frequency equal to the *Mod Freq (Hz)* parameter in *LOCKIN AMPLIFIER* block under the *Spectrometer Settings* tab.) As in a conventional EDMR experiment, the choice of the modulation frequency should take into account the gain and bandwidth specifications of the preamp. A 1kHz modulation frequency allows for a higher sensitivity setting to be used on the preamp (ie: 100nA/V).
- AW Amp (PP/2):** In the *ARBITRARY WAVEFORM on AO1* block under the *Configuration* tab
 - Set the amplitude of modulation square wave
 - Because the Fluke291 mixes (modulates) this signal with its internally generated, frequency swept signal (with amplitude configured by *ENDOR Vpp*), ensure that the product of these two amplitudes DO NOT exceed the maximum input limit on the BUF634 amplifier.
- AW Phase:** In the *ARBITRARY WAVEFORM on AO1* block under the *Configuration* tab
 - Set to 0. Phase optimization is still performed when demodulating in the *LOCKIN AMPLIFIER* block.
- AW Offset:** In the *ARBITRARY WAVEFORM on AO1* block under the *Configuration* tab
 - Set to 0. (This could be used to control the modulation depth; however, it is instead performed in the Fluke 291.)
- Duty Cycle:** In the *ARBITRARY WAVEFORM on AO1* block under the *Configuration* tab
 - Set to 50%

The low-field ENDOR setup has not yet been shown to work perfectly; however, the results that have been obtained are promising. Below is a spectra that was obtained on the base collector junction of a 4H SiC BJT.

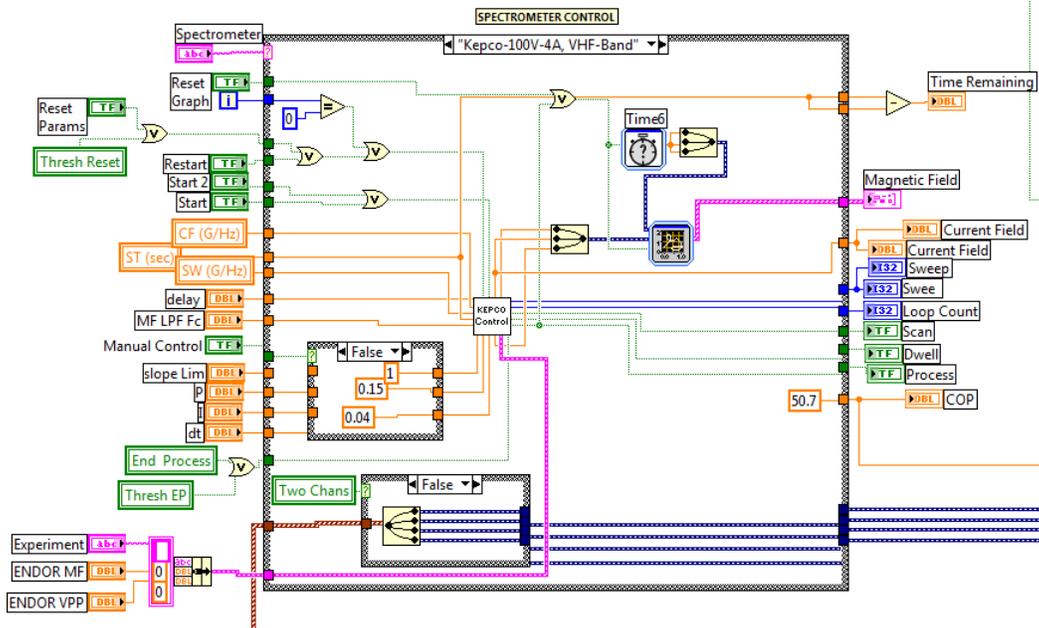


$$h\nu = g_N \mu_N B_0 \quad \rightarrow \quad g_N = \frac{h\nu}{\mu_N B_0} = 1.02 \cdot 10^{-2} \cdot \nu \text{ (kHz)}$$

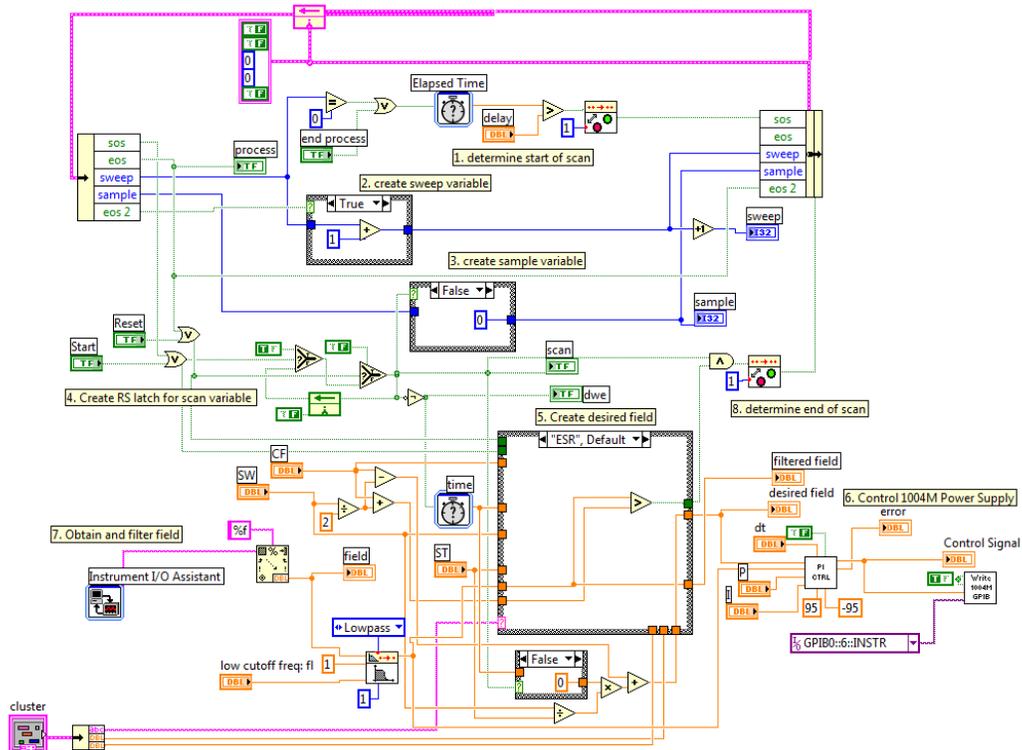
Large Peaks	{	E	$\nu = 233\text{kHz}, g_N = 2.37$	$(^{31}\text{P}: 2.26)$	Small Peaks ???	{	A	$\nu = 51\text{kHz}, g_N = 0.52$	$(^{15}\text{N}: 0.56)$
		G	$\nu = 384\text{kHz}, g_N = 3.92$	$(^1\text{n}: -3.82)$			B	$\nu = 95\text{kHz}, g_N = 0.97$	$(^{75}\text{As}: 0.96)$
							C	$\nu = 170\text{kHz}, g_N = 1.73$	$(^{11}\text{B}: 1.79)$
							D	$\nu = 189\text{kHz}, g_N = 1.93$	$(^{115}\text{Sn}: -1.84)$
							F	$\nu = 305\text{kHz}, g_N = 3.11$	$(^{203}\text{Tl}: 3.2)$

Even though there appears to be many absorption peaks, it is likely that some of them are artifacts from the hardware. Like mentioned earlier, this is a work in progress so alterations of the amplifier circuit may be needed or modifications to the software may be required.

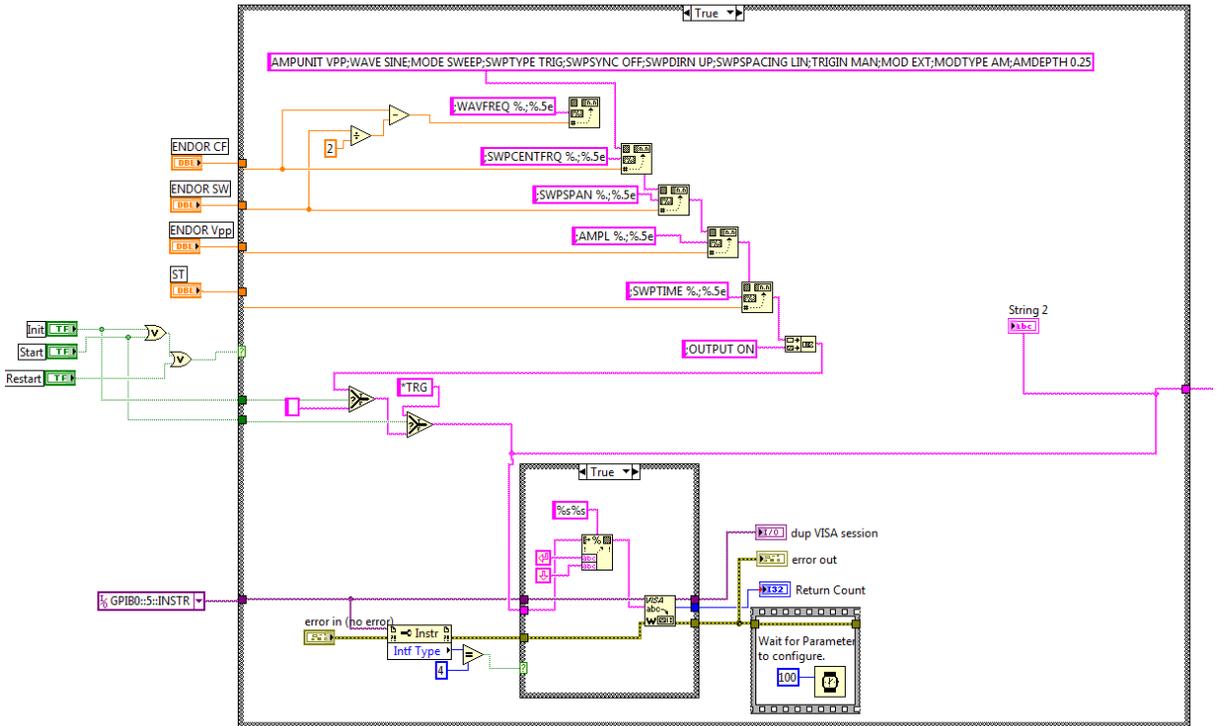
If it is desired to modify the software that “talks” to the Fluke 291, one needs to follow the steps below to access this code. First, open the block diagram of the main GUI and locate the snippet of code below.



Under the spectrometer selector, ensure (as in the picture) that "Kepco-100V-4A, VHF Band" is selected. Then, double click on "Kepco Control". This should bring up the block diagram illustrated below.



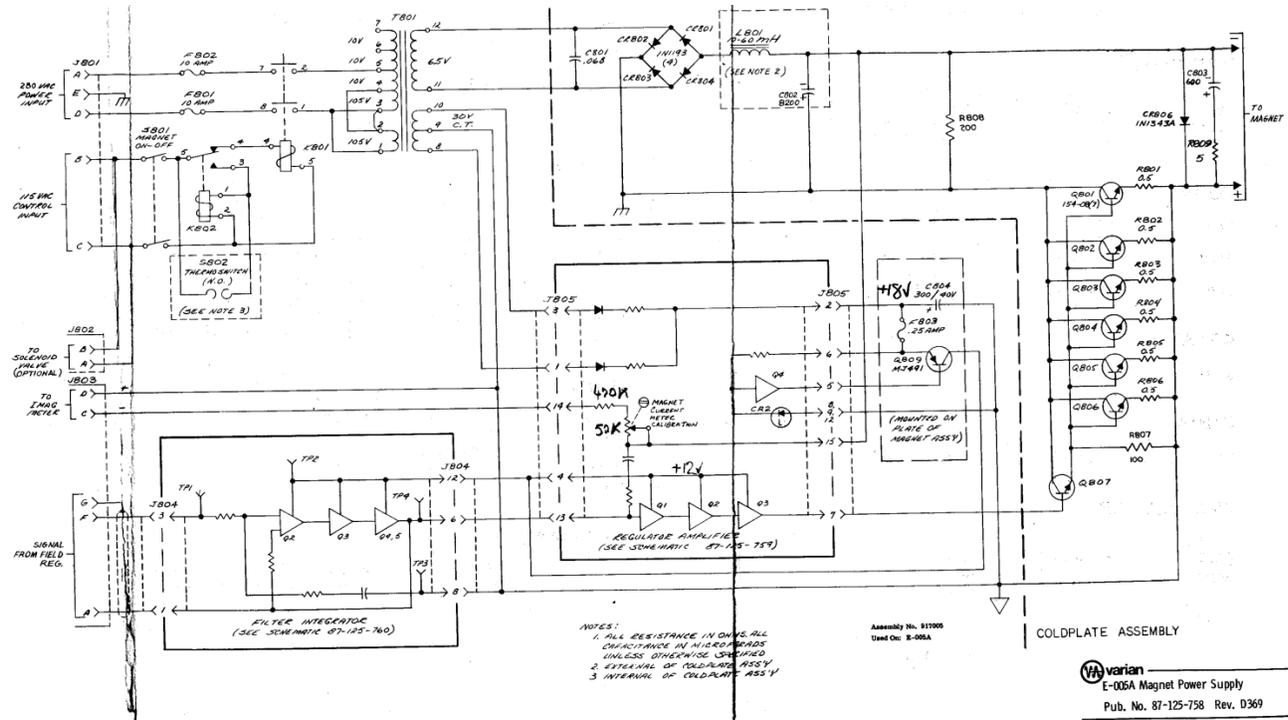
Now, find the section entitled "5. Create desired field". Unlike in the picture, make sure that this selector says ENDOR. Once selected, open the block diagram in the middle of the selector box which should take you to the code which generates the commands that are sent to the Fluke 291. This vi is illustrated below.



Here, one will be able to modify the parameters that are sent to the Fluke 291 waveform generator. As illustrated in the vi, the code shown will only execute on initialization, start, or a reset of the software. When the case structure executes, all parameters are concatenated into a single string and then sent to the Fluke 291 over the GPIB interface. Two parameters that may be desired to be change are the modulation depth (*AMDEPTH*) and or modulation type (*MODTYPE*). Be sure to look into the command list in the Fluke 291 user manual if other options are to be configured. Note that if the Fluke 291 does not recognize or does not “like” a received command, it will sound an audio beep.

I. Magnet Control with the Varian Power Supply/Magnet

One of the main problems with the design of the Varian power supply that powers the magnet is that it requires a floating control input to drive the magnetic field. In other words, the control signal driving the magnet should not be connected to ground. If controller's signal is referenced to ground, the power supply will ramp up to its maximum current range, will most likely blow the fuse in the back of the supply, and possibly damage the coils of the magnet. Note that this will also happen if the power supply is turned on and nothing is connected to the control input. Therefore, be aware when operating this supply. There are detailed instructions below to so that this situation can be avoided. The only documentation that we have on this power supply is illustrated below.



The Varian power supply is controlled through the Lakeshore 475 DSP Gaussmeter. The software sends commands to the Gaussmeter for user defined magnetic field sweeps. The Gaussmeter receives these commands and then controls the sweep with an internal digital PI controller which uses a hall probe to measure the field and accuracy of the desired sweep. The output of the controller is a bipolar analog voltage which is referenced to ground. To overcome this, I built a bipolar optoisolating circuit that takes the DC (ground referenced) control voltage from the Gaussmeter and sends the signal optically to a set of photodiodes whose circuit reference is not grounded. This effectively allows the grounded control signal from the Gaussmeter to control a power supply whose control input cannot be grounded. I found a great application note from an Agilent Technologies Application Note, *Overview of High Performance Analog Optocouplers- Application Note 135*.

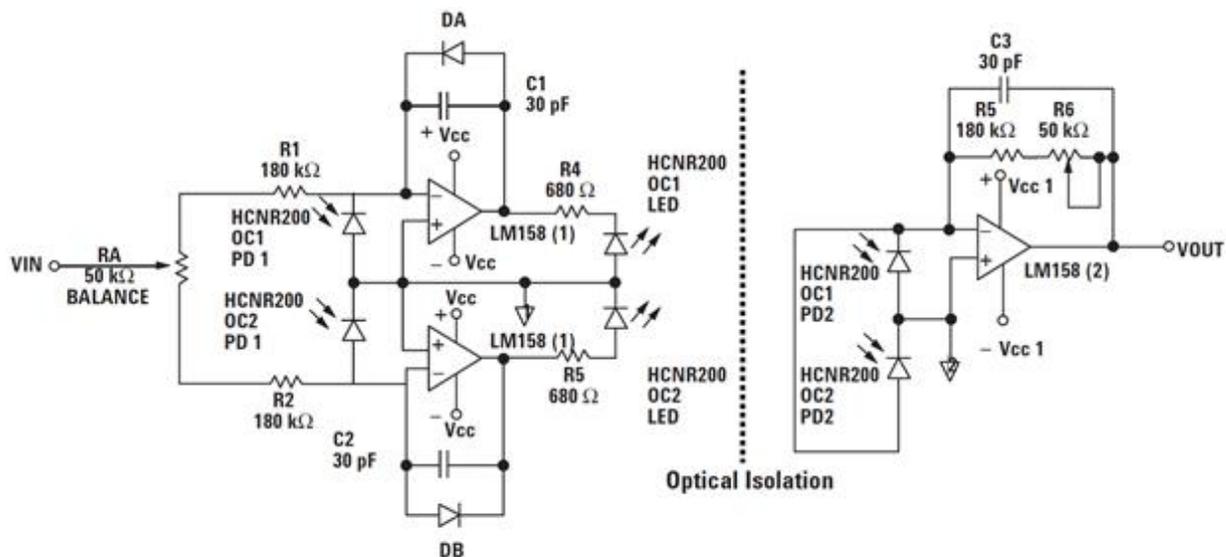


Figure 3. Bipolar Input Voltage Analog Isolation Amplifier using the HCR201

I incorporated two switches on the project box in which this circuit is enclosed so that each separate circuit can be turned on individually. Each circuit is powered with a $\pm 12\text{V}$ DC supply. (There are actually 4 different $+12\text{V}$ dc supplies that are connected to the circuit.) The only other connections into the project box are V_{in} and V_{out} . The Gaussmeter's control voltage should be connected to V_{in} of the box and the V_{out} should be routed to the magnet's power supply. Note that if this circuit breaks, this magnetic CANNOT be used! This is precisely why it is being fully documented in this reference.

As a result of using this modified controller, the Varian spectrometer must be turned on using the following steps:

1. Turn on PolyScience recirculator
2. Turn on LakeShore 475 DSP Gaussmeter
3. Turn on the optoisolating circuit which isolates the control input of Varian power supply from earth ground (circuit described below).
4. Open up the latest Data Acquisition System program in LV and run it. The path is as follows, ($\backslash\dots\backslash\text{user_name}\backslash\text{Desktop}\backslash\text{Data Acquisition System}$)
5. Once the "REMOTE" blue LED lights up on the Gaussmeter, turn on the Varian power supply.
6. Make sure the field does NOT go above 5kG, if it does, turn off the Varian power supply and ask for help! If it settles at the desired field, then everything was powered on properly. Note that it is now safe to stop and start the LV program as needed.
7. All other components can be turned on.

The powering down of the spectrometer can be accomplished by the following steps:

1. Set the field to a low value, say 100G...
2. Turn off the Varian power supply.
3. The remaining components do not need to be turned off in any specified order.

J. Magnet Control with the HP Power Supply/Magnet

Because there are two spectrometers in the lab (K- and X-band) whose magnets are powered by the same HP power supply, a few comments should be made as to how to program these supplies. The text below was taken from the HP power supply user manual and describes how to remotely program the power supply using an analog voltage as the controlling signal.

Operating and Service Manual: Regulated DC Power Supply, HP Models 6259B, 6260B, 6261B, 6268B, 6269B, HP Part No 5950-1766

3-42: Constant Voltage Output, Voltage Input (Variable Gain). In the remote programming arrangement shown in Figure 3-6, the series combination of external voltage source E_S and reference resistor R_R replaces the supply's internal voltage programming current source. As a result, the voltage this external current source develops across gain control R_p becomes the output voltage of the supply, and the gain relationship between E_S and the output voltage equals the resistance ratio R_p/R_R .

3-43: When using this programming technique, select a value for R_R that is less than 10k ohms and that would conduct at least 5 milliamps if connected across the programming voltage source with its voltage at the maximum value of the input voltage to be used. Once the value of R_R is selected, multiply R_R by the maximum voltage gain desired to find R_p . (If desired, the power supply's front panel voltage controls can be used in place of external gain control from the circuit and strapping together terminals A1 and A2.)

3-44: The output voltage of the supply can be adjusted to exactly zero with a zero programming voltage input either by installing and adjusting R111 as discussed in paragraph 5-82 or, if the instrument is equipped with Option 020 or 022, by adjusting potentiometer R112 as discussed in paragraph 5-83.

From these excerpts, determine the values of R_p and R_R as follows:

1. Find R_R using, $R_R = V_{c_max}/5mA \ni R_R < 10k\Omega$, where V_{c_max} is the maximum output control voltage from the LakeShore DSP Gaussmeter. This value can be programmed in the Gaussmeter and should be set to 10V. Therefore, $R_R = 2k\Omega$
2. Find the maximum desired gain, $G = R_p/R_R = V_{p_max}/V_{c_max}$, where V_{p_max} is the maximum output voltage on the HP supply ($< 35V$). If it is desired to use the maximum output voltage of the power supply $V_{p_max} = 35$, then the $R_p = R_R \cdot V_{p_max}/V_{c_max} = 2k\Omega \cdot 35V/10V = 7k\Omega$

Because $2k\Omega$ and $7k\Omega$ are not that common, $R_R = 2.2k\Omega$ and $R_p = 7.5k\Omega$

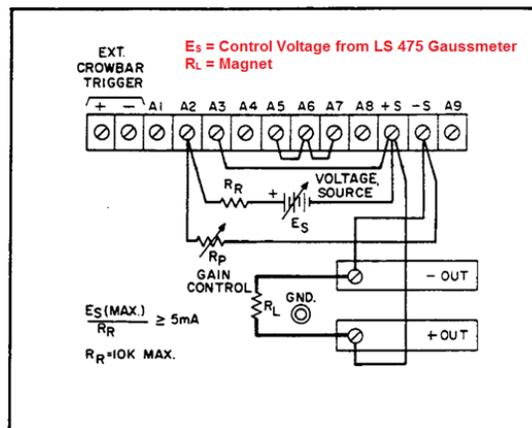
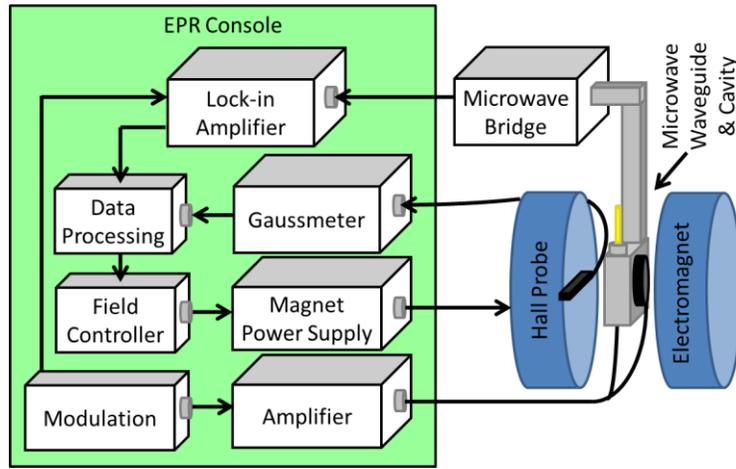
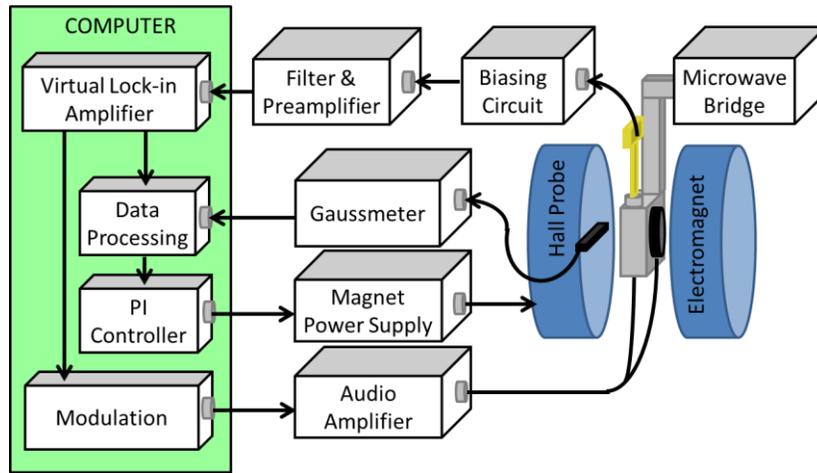


Figure 3-6. Voltage Programming of Output Voltage (Variable Gain)

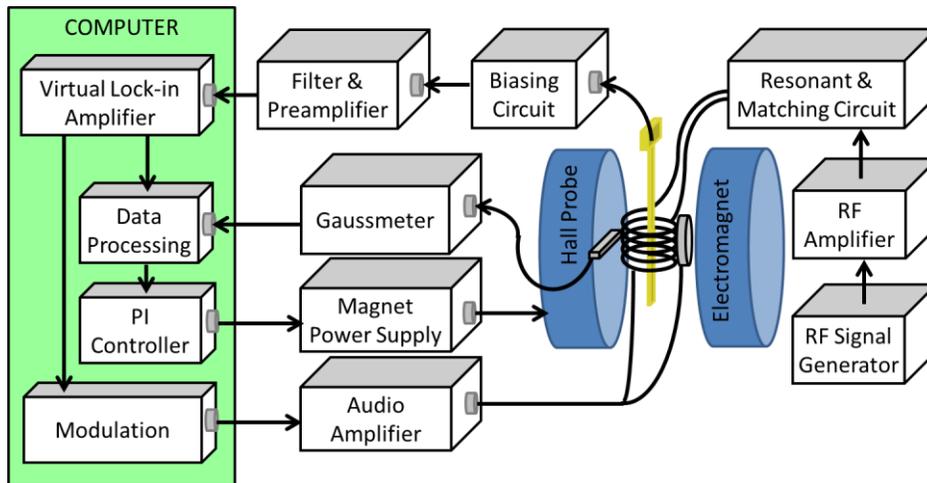
K. Differences in EDMR and EPR Spectrometers



Bruker Spectrometer

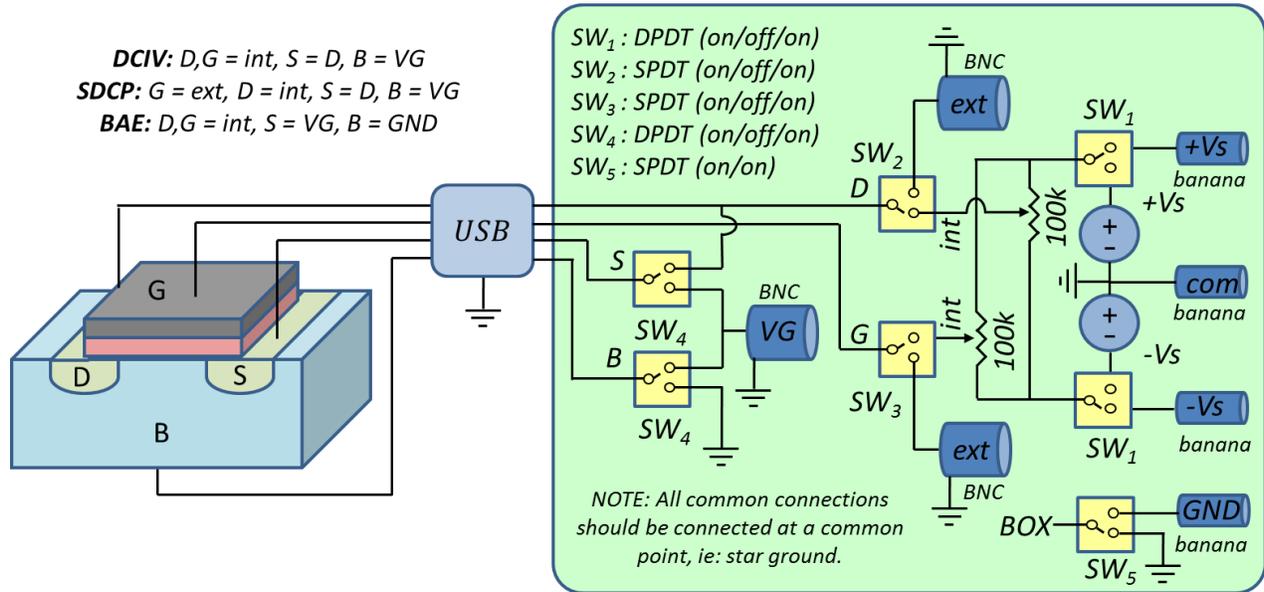


X-Band Spectrometer



RF-Band Spectrometer

L. Generic Biasing Box



M. Miscellaneous Software Reference

NI BNC 2110 Connections (into PCI 6259): Note BNC 2110 ONLY supports differential connections

A10 – In Phase ESR/EDMR Signal From Lockin Amplifier

A11 – Quadrature ESR/EDMR Signal From Lockin Amplifier

A12 – ESR Signal from Microwave Cavity or EDMR Signal from Preamp

A13 – Trigger from Bruker Consoles (ESR) or Modulation Reference (EDMR)

AO0 – Modulation Reference when using the Virtual Lockin Amplifier (VLIA)

AO1 - Arbitrary waveform of Users preference (ie: charge pumping signal)

GPIB Addresses:

Hardware	GPIB Address
Lakeshore 475	12
Lakeshore 450	12
SRS DS335	22
SRS830	8

DAQs in the Lab

Specification	PCI6259	PCI6023E	PCI6063E
resolution (bits)	16	12	16
maximum F_s (KS/s)	1000	200	200
Maximum input range (V)	± 10	± 10	± 10
Minimum input range (mV)	± 100	± 50	± 50