FREQUENCY SWEEP NMR

FOR THE POLARIZATION OF ^{3}He

A thesis submitted in partial fulfillment of the requirement
for the degree of Bachelor of Science with Honors in
Physics from the College of William and Mary in Virginia

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Advisor: Dr. Todd Averett
Spring, 2004

ABSTRACT

Thomas Jefferson National Accelerator Facility is currently running experiments to determine the spin structure of the neutron. The Polarized ^{3}He laboratory at the College of William and Mary fills and characterizes target cells for these experiments. Jefferson Lab requires a new system of Frequency Sweep Nuclear Magnetic Resonance to measure the polarization of the target cells compelling the William and Mary lab to develop a system for this purpose. William and Mary's system, however, has a flaw. Large spikes in the data as the frequency is swept through resonance cause it to be unusable in their endeavors to pursue their research. The purpose of this thesis is to identify and eliminate the flaw, ultimately resulting in a usable set of data and a working frequency sweep NMR system.

1. INTRODUCTION

^{3}He atoms are used in experimental nuclear physics to allow the study of neutron spin structure. Because free neutrons are inherently unstable, they cannot be studied directly. The most probable state of the ^{3}He atom is in the S state with the spins of the two protons anti-aligned. Due to this fact, the spins of the protons cancel each other out, leaving only the spin of the neutron to contribute to the total spin of the nucleus. Thus, the ^{3}He nucleus can be used as a substitute for free neutrons.

The College of William and Mary produces glass target cells filled with ^{3}He for experiments at Thomas Jefferson National Accelerator Facility. During the experiments the neutrons of the ^{3}He nucleus are polarized by spin exchange as they interact with polarized Rb atoms located inside the target cells. A high-energy electron beam is passed through the target cell, scattering from the ^{3}He. To obtain the most accurate results, the highest possible polarization of the ^{3}He is crucial. Currently, the maximum polarization is around 40%-50%. If this can be improved, the Jefferson Lab experiments could be conducted with increased accuracy.

Currently a uniform holding field from Helmholtz coils is used to provide a spin axis for the ^{3}He nuclei. Polarization is measured using an NMR system that ramps the magnitude of the holding field over a range of 25-32 G. The spin of the neutrons will flip direction at about 29 G with an RF field at 91 kHz, providing a signal that can be measured by a set of coils located next to the target cell. *(Refer to Figure 1)*

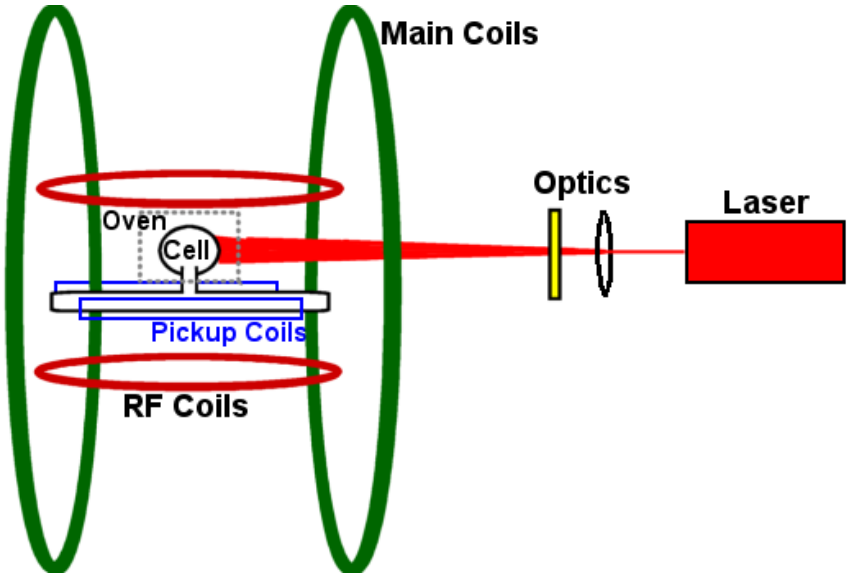

Figure 1: William and Mary NMR system [4]

Jefferson Lab devised another way of improving the accuracy of the experiments. Rather than using two large Helmholtz coils to control the holding field, they used a large iron box, wrapped with coils to produce a more uniform holding field. The field of the iron box can't be ramped quick enough for NMR due to Eddy currents, so a system to sweep the NMR frequency was developed at the College of William and Mary. Because of the new apparatus, the holding field had to be kept steady. During testing at William and Mary, there proved to be a problem. The NMR sweep curve contained large spikes. The purpose of this research is to find the problem in the NMR and eliminate it so that William and Mary can continue to produce, test and characterize new target cells for continuing research at Jefferson Lab.

2. BACKGROUND

2.1 FILLING THE CELL:

The target cells used in the polarization of ^{3}He are blown out of aluminosilicate glass at either Princeton or the University of Virginia. The aluminosilicate glass is used because of its ability to contain the ^{3}He due to its low porosity. It is then treated with nitric acid to smooth the inner surface, preventing it from retaining impurities. The glass is formed into two chambers connected with a tube with a radius of a ¼ inch. The upper chamber is spherical with a lip around the base where it connects to the tube. The bottom chamber is a cylinder of length 14 inches with a radius of ¾ inches, capped off by a half sphere of 0.1mm thick glass at either end. The thickness of the end caps is required to be thin in order to minimalize interactions between the glass and the electron beam during its use at Jefferson Lab. *(Refer to Figure 2)*.

The cell is evacuated and placed in an oven of 470 °C for seven days. The exposed glass that connects the target cell to the vacuum pump, called the evacuation tube, is flame baked, a process in which the glass is heated by a torch in order to free impurities from the inner surface. A ¼ g sample of Rb is then chased into the pumping chamber *(Refer to Figure 2)* where it sits in the lip by the connector tube. Once a sufficient vacuum is obtained, the cell is placed in a dewar of liquid ^{4}He in order to bring the temperature down to 4 K. Once cooled, the cell is filled with 8.5 atm (at room temperature) of ^{3}He and trace amounts of N_2 gas. The cell must remain at 4 K so that the internal pressure will not rupture the cell while it is being removed by torch from the filling apparatus.

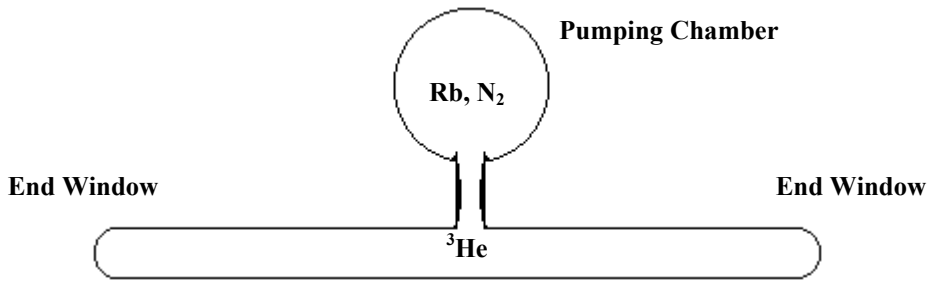

Figure 2: Jefferson Lab Target Cell

2.2 THE POLARIZING APPARATUS:

To polarize the cell, the process of optical pumping is used. This is a process that relies on the transfer of polarization from polarized rubidium vapor to the ^{3}He nucleus through spin exchange.

Once filled, the upper chamber of the target cell is installed in an oven in the polarized target system made out of Torlon and heated to 170 °C where the sample of Rb is vaporized. The Rb is contained in the upper chamber by the heat differential, caused when an oven heats the pumping chamber. *(Refer to Figure 2)* A circularly polarized laser with a wavelength of 795nm polarizes the Rb electrons in a process called optical pumping. The Rb would normally emit photons during the optical pumping process, which would depolarize the gas; however, the N_2 gas present allows the Rb electrons to de-excite without emitting a photon during the polarization process.

Once the Rb is completely polarized, the electrons collide with the ^{3}He nucleus. The polarized spins of the Rb electrons interact with the ^{3}He neutrons, transferring the spins. The magnetic moments of the Rb electrons are then realigned through the optical pumping in order to continue interacting with the ^{3}He.

3 POLARIZATION

3.1 OPTICAL PUMPING

The first step in the polarization process is the polarization of the vaporized Rb. The solid Rb is vaporized at 170°C by an oven constructed entirely of Torlon. The Torlon oven is fixed between two orthogonal sets of Helmholtz coils that create a holding field. The direction of this field dictates the direction of polarization. There, the Rb is exposed to 795 nm, circularly polarized laser light. Depending on the direction of the laser's polarization, it will only excite the valence electrons of a specific spin state because the spin states of the Rb electrons separate in a magnetic field. If the 795 nm laser is left circularly polarized, it will excite electrons from the $m=+1/2\ 5S_{½}$ state to the $m=-1/2\ 5P_{½}$ state, whereas the right circularly polarized laser will excite electrons from the $m=-1/2\ 5S_{½}$ state to the $m=+1/2\ 5P_{½}$ state. In this case, the electrons must be pumped to the $m=+1/2\ 5P_{½}$ state, thus the laser must be right polarized. Once the electrons are in the $m=+1/2\ 5P_{½}$ state, they will release energy and drop to either the $m=-1/2\ 5S_{½}$ or the $m=+1/2\ 5S_{½}$ state. If they drop to the $m=-1/2\ 5S_{½}$ state, then the laser will pump them back to the $m=+1/2\ 5P_{½}$ state until they decay to the $m=+1/2\ 5S_{½}$ state.[3] Thus, all the electrons will eventually be in the $m=+1/2\ 5S_{½}$ state and the Rb will be polarized.

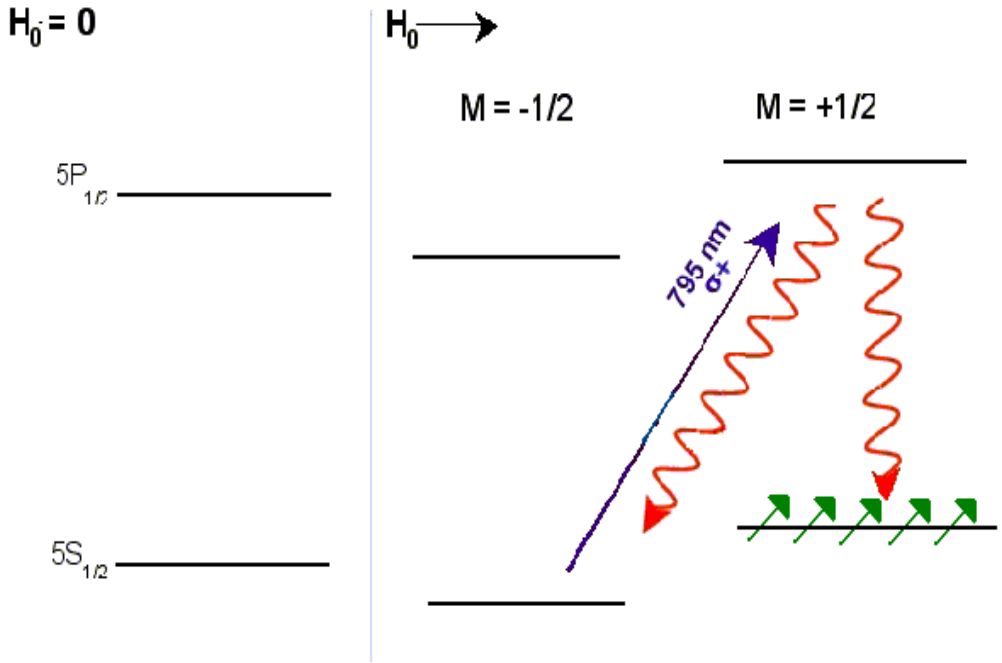

Figure 3. Optical Pumping of Rubidium. Valence electrons in the m=-1/2 $5S_{½}$ state are excited from the 795nm laser light into the m=+1/2 $5P_{½}$ state. They can then either decay back to the m=-1/2 $5S_{½}$ or m=+1/2 $5S_{½}$ state, however, if they drop to the m=-1/2 $5S_{½}$ level, they will be pumped back to the m=+1/2 $5P_{½}$. Eventually all the electrons end up in the m=+1/2 $5S_{½}$ state, polarizing the Rb. [4]

However, as shown in *figure 3*, a photon is emitted when the Rb electron decays from the m=1/2 $5P_{½}$ to the $5S_{½}$ state. This photon is randomly polarized and can be absorbed by another electron in a different spin state, causing the depolarization of the Rb. Due to the electron's tendency to dump photons during decay, high levels of polarization would not be possible.[2] To counter this effect nitrogen gas is introduced to the system. The N_2 absorbs the energy of the excited atom by it's rotational and vibrational motion.

3.2 Spin Exchange

When the Rb is in its final m=1/2 $5S_{½}$ state, it can transfer its electron spin to the nucleus of the ^{3}He nucleus through a hyperfine-like interaction. However, only about 3%

of the polarized Rb atoms lose their polarization through spin-exchange processes with ^{3}He, making it an extremely inefficient process. The ^{3}He's polarization can be described as a function of time through the equation

$$P_{^3He}(t) = < P_{Rb} > \frac{\gamma_{SE}}{\gamma_{SE} + \Gamma}\{1 - e^{[-(\gamma_{SE}+\Gamma)t]}\}$$

where γ_{SE} is the spin exchange rate, $\gamma_{SE} = k_{SE}[Rb]$, Γ is the polarization destruction rate and $< P_{Rb} >$ is the average polarization of the Rb.[3]

The amount of polarization that the ^{3}He will achieve is primarily dictated by the ^{3}He destruction rate, Γ. This factor has four primary contributors: $\Gamma_{Dipole}, \Gamma_{Wall}, \Gamma_{\nabla B}, \Gamma_{Beam}$. These factors are related through the equation

$$\frac{1}{\Gamma} = \frac{1}{\Gamma_{Dipole}} + \frac{1}{\Gamma_{Wall}} + \frac{1}{\Gamma_{\nabla B}} + \frac{1}{\Gamma_{Beam}} \qquad \textit{eq. 3}$$

where Γ_{Dipole} is the depolarization rate from ^{3}He-^{3}He collisions, Γ_{Wall} is the depolarization rate from interactions with the cell walls, $\Gamma_{\nabla B}$ is the depolarization rate from the magnetic fields and Γ_{Beam} is the depolarization rate from Jefferson Lab's electron beam. Due to the high density of ^{3}He in the target cells, Γ_{Dipole} is the dominant factor in the total depolarization rate for a good cell. The rate can be defined by

$$\Gamma_{Dipole} = \frac{[^3He]}{744} hrs^{-1}$$

where [^{3}He] is in amagats (1amagat is equal to 2.689×10^{19} atoms/m^3).[3]

Another process that would cause depolarization is the Γ_{Wall}, or the ^{3}He interactions with the cell walls. There are many different reasons that the cell would cause depolarization, one of which being the out gassing of paramagnetic gases such as O_2

and NO that are released when the cell is heated. Another would be the paramagnetic material like Rb_2O, which would form on the surface of the cell walls. [3] A third possibility would be an increase in sticking time of the ^{3}He to the cell walls due to microscopic cracks and scrapes on the interior surface of the glass. This variable can differ dramatically between cells depending on the quality of the glass blowing and the procedure for filling it.

The magnetic field gradients also contribute to the depolarization of the cell. The rate of decay from the magnetic fields, $\Gamma_{\nabla B}$, is proportional to the field gradients perpendicular to the holding field, $|\nabla B_x|^2$ and $|\nabla B_y|^2$ as shown by the equation:

$$\Gamma_{\nabla B} = D_{^3He} \frac{|\nabla B_x|^2 + |\nabla B_y|^2}{B_z^2}$$

where $D_{^3He}$ is the self-diffusion constant of ^{3}He (in this case $D_{^3He}$=0.28cm^{2}s^{-1}) and B_z is the Helmholtz field. [3]

4. TRADITIONAL CHARACTERIZATION OF THE CELL

4.1 SPIN-UP

Once the ^{3}He is polarized, the polarization must be tested. The traditional method of testing was the AFP (adiabatic fast passage) method of NMR (nuclear magnetic resonance). This method involved holding the frequency of a RF field at 91 kHz perpendicular to the Helmholtz magnetic field *(Refer to Figure 4)*.

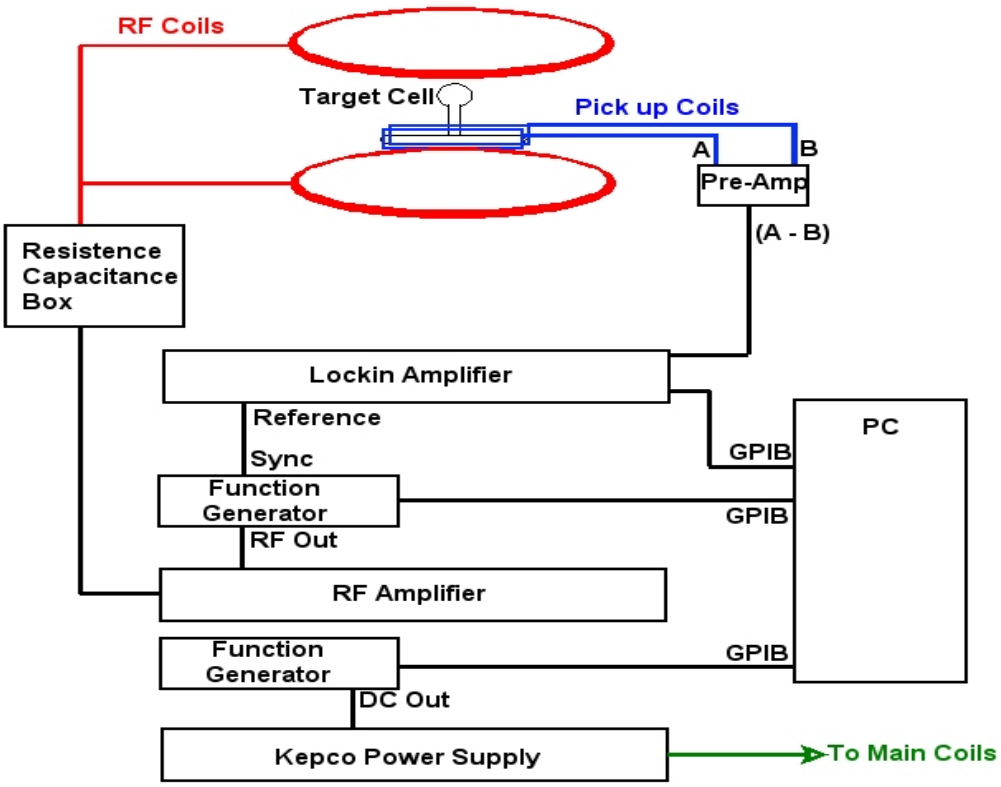

Figure 4: AFP NMR system [4]

The RF frequency corresponds to the Larmor frequency of the ^{3}He given by the equation $\omega = \gamma\beta$, where γ is the gyromagnetic ratio of the nucleus and B is the magnitude of the magnetic field. The Helmholtz field was then swept from 25 G to 32 G, past the resonance for ^{3}He, and back down to 25 G in a total of 11 seconds, thus fulfilling the adiabatic conditions for AFP NMR. When the Helmholtz field approaches 28.3 G, the resonance for ^{3}He, the magnetic moment will flip 180° and change the polarization direction. When the amplitude of the holding field is ramped back down, the spins will flip back to their original direction. The change in the magnetic moments direction produces a change in the magnetic flux in the pickup coils aligned on either side of the

lower chamber of the target cell. This change induces a current in the pickup coils. This signal is then amplified by the Lock-in Amplifier and saved. The amplitude of the signal is proportional to the polarization of the cell.[2]

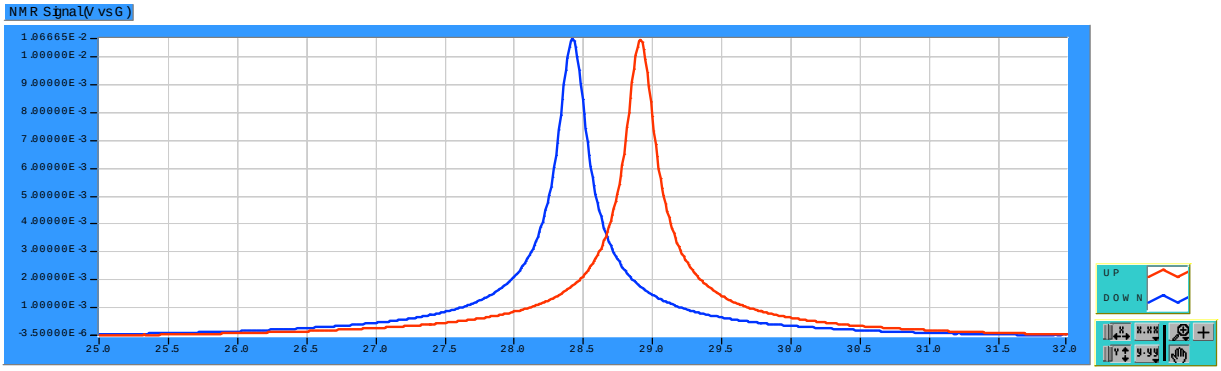

Figure 5. Polarization curve from Spin-up taken from the program "NMR Helium XY FG-V WM.vi." [1]

These measurements are taken every four hours while the cell is polarizing until the polarization levels off. This is known as a Spin-up. A good cell will take quite a while to level off indicating a large amount of polarization.

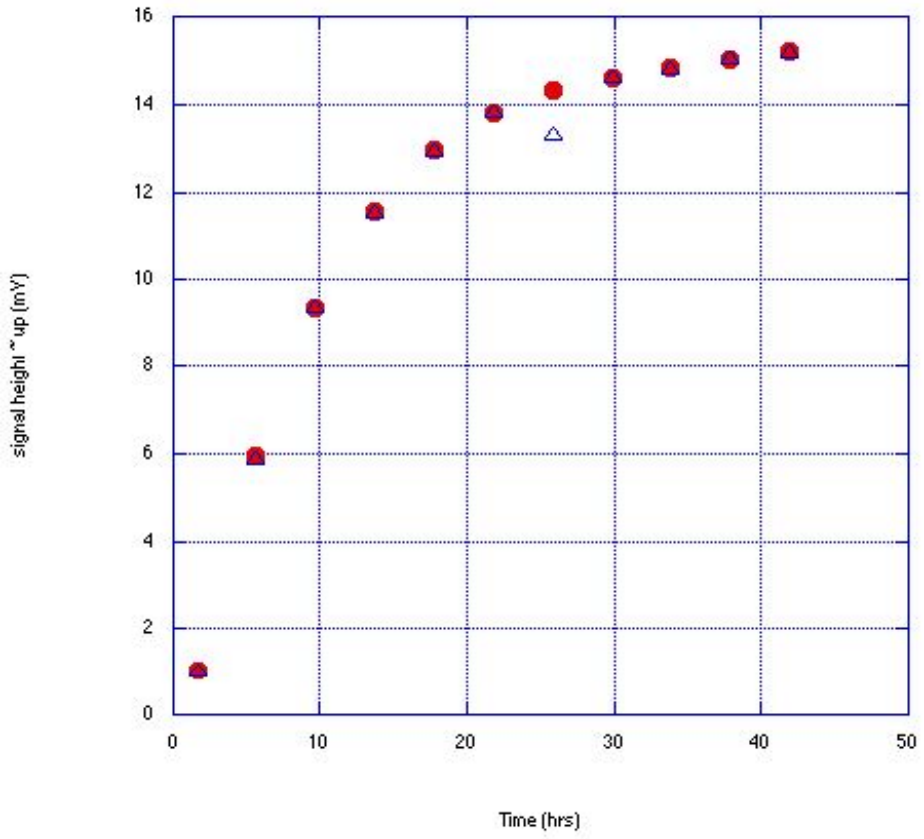

Figure 6. Spin-up Curve showing polarization vs. time. [2]

4.2 SPIN-DOWN

Once the cell is fully polarized, it is necessary to know the time that it takes to lose its polarization. To do this, the oven and the lasers are turned off to stop the optical pumping from contributing to the cell's polarization. At this point, because the Rb is no longer able to realign its magnetic moments to polarize the ^{3}He, the cell begins to lose polarization. The NMR system is left in place to take measurements every four hours as the cell depolarizes roughly following the equation:

$$P_{^3He}(t) = P_{init} e^{-\Gamma t}$$

where P_{init} is the cell's polarization when the laser was turned off. The curve will not follow this formula exactly because the AFP NMR increases the Γ factor, expediting the

depolarization process. This is known as the AFP loss. Once again this data is plotted into a curve indicating the time that the cell takes to lose its polarization. A good cell will take around 50 hours to depolarize completely.[2]

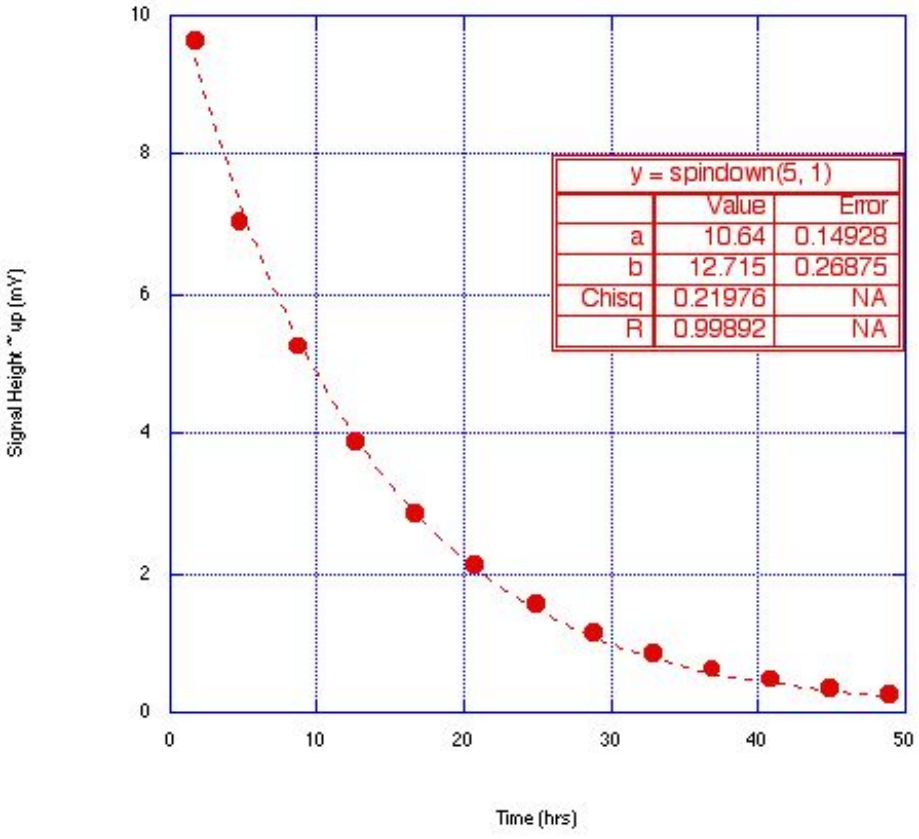

Figure 7. Spin-down curve showing signal height vs. time. [2]

5. FREQUENCY SWEEP NMR

5.1 THE NEW NMR SYSTEM

Jefferson Lab is developing a new apparatus where in place of the Helmholtz coils there will be an iron box wrapped in coils that will create a more uniform holding field. The drawback to this system is that it creates large Edy currents in the metal when the holding field is ramped, slowing the sweep down enough to prevent AFP from flipping

the spins, thus a new testing system was required. The holding field had to be kept constant at 28.3 G and a method was devised to ramp the RF frequency. The initial range was to be 80.3 kHz to 102 kHz, passing through the resonance frequency at 91 kHz. The NMR system was modified to accept the ramping frequency by running an Agilent 33120A function generator to trigger the lock-in amplifier. A waveform was downloaded into the function generator to allow it to ramp in a frequency modulation mode over the desired range.

5.2 THE PROBLEM

The system, however, had limitations. The frequency sweep flipped the spins of the ^{3}He and produced a polarization curve out of the lock-in amplifier but there were spikes in the signal *(Refer to Figure 8)*. These spikes in the curve were close to the amplitude of the polarization, which caused it to be unusable in characterizing the target cells.

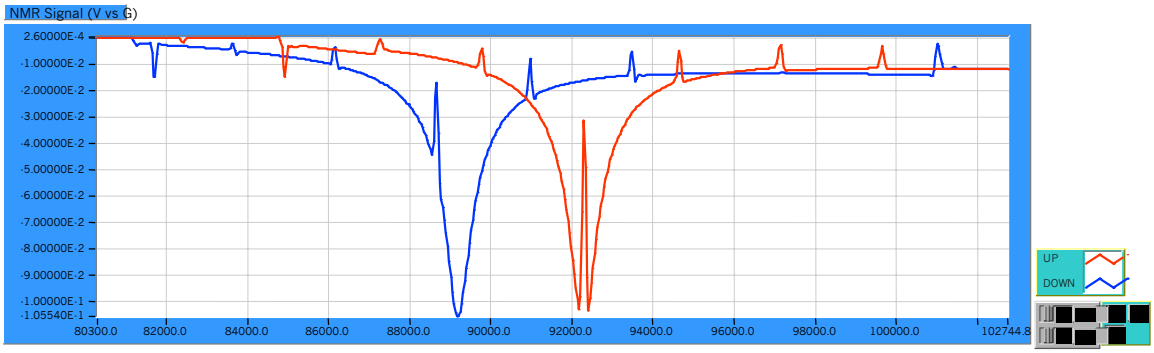

Figure 8: Spikes in Frequency Sweep NMR [1]

5.3 ATTEMPTED SOLUTIONS

5.3.1 DOWNLOAD WAVEFORM

The original code that controlled the frequency sweep NMR system relied on a waveform that had to be downloaded so that the lock-in amplifier had a reference to

trigger on while the Agilent 33120A ramped the frequency. The downloaded waveform was checked for discontinuities and found to be clean. A digital recording oscilloscope, borrowed from Agilent, was used in an attempt to record the 11 sec. signal from the Agilent 33120A function generator. Unfortunately, the digital oscilloscope could only record 20 milliseconds at a time. Seeing as the spikes in the signal were, on average, over a second apart, this proved to be of little use.

5.3.2 HP3324A Function Generator

The glitch in the polarization curve could have also been caused by a non-uniform frequency ramp in the new digital function generators. Although the Agilent 33120A was designed to have a uniform frequency sweep with minimal fluctuations, a theory was proposed that the digital function generators would jump from one preset frequency to another on such a small interval that a digital oscilloscope could overlook them. These jumps could possibly be detected and misinterpreted by the lock-in amplifier and be seen as a discontinuity. Although this could not be seen directly because the previous attempt at recording the entire signal had failed, the function generator was replaced. A HP 3324A function generator was obtained, calibrated and installed to replace the digital function generator. The older analog signal of the HP3324A in conjunction with a smaller frequency range, around 85kHz to 97kHz, was hoped to eliminate the problem due to it's ability to ramp the frequency smoothly, albeit less accurately.

A program was written to allow the HP3324A to ramp the frequency up, hold it, and then ramp down. It was inserted into the pre-existing NMR code. It was designed to not only allow the characterization of new ^{3}He cells but also allow the calibration of cells filled with water used for NMR calibration. The water cells require the frequency to hold

for a short time after ramping up. The old method of downloading the waveform was also discarded because the HP3324A had suitable frequency sweeping functions programmed into it, eliminating another source of possible spikes.

The frequency sweep NMR was tested on a cell after the code had been converted to incorporate the HP3324A and once again, the spikes were prominent in the signal. The sweep was narrowed from 85 to 97 kHz to 87 to 95 kHz in an attempt to decrease the frequency of the spikes appearance and a signal was acquired that only contained 3 spikes without compromising the precision of the measurement.

6. CONCLUSION

The William and Mary ^{3}He lab has successfully produced a set of code that will perform Frequency Sweep AFP NMR to characterize the target cells filled on site, though the spikes remain in the polarization curve. It was determined that since the spikes appear in other laboratories around the country, such as the Dr. Wolfgang Korsch's laboratory located at the University of Kentucky, and all the systems which produce the spikes use the same lock-in amplifier, the spikes must be originating from the PerkinElmer 7265 lock-in amplifier. This device must be replaced in order to use the code Modified NMR code to characterize new cells for the coming Jefferson Lab experiments.

REFERENCES:

(1) Buckley, Erin, Polarized ^{3}He. College of William and Mary, REU Summer Report, 2003

(2) Fuoti, Kirsten, Investigation and Characterization of Sol-Gel Coated Cells to Improve ^{3}He Polarization. Senior Thesis, College of William and Mary, April 2003.

(3) Kramer, Kevin M., A Search for Higher Twist Effects in the Neutron Spin Structure Function $g_2^n(x,Q^2)$. Dissertation, College of William and Mary, April 2003.

(4) Milkie, Daniel, Polarization and Polarimetry of ^{3}He. Senior Thesis, College of William and Mary, April 2002.

(5) Powles, J. G., "The Adiabatic Fast Passage Experiment in Magnetic Resonance." Proc. Phys. Soc., 71, 496.

(6) Walker, Thad G., Happer, William, Spin-exchange optical pumping of noble-gas nuclei. Reviews of Modern Physics, Vol. 69, No.2, April 1997.

APPENDIX A. SOFTWARE DOCUMENTATION

(Code algorithms modified from Buckley [1])

A.1 Freq. Sweep.vi

Written for William and Mary NMR system by:
Erin Buckley, The College of William and Mary, Williamsburg, VA, embuc2@wm.edu
Kirsten Fuoti, The College of William and Mary, Williamsburg, VA, kafuot@wm.edu
Advisor: Todd Averett, The College of William and Mary, Williamsburg, VA, averett@physics.wm.edu

7/19/03

PURPOSE:
This VI is intended to perform an NMR sweep by ramping the frequency of the RF Coils through resonance. It uses Function Generator 13 to trigger the lock-in amplifier.

This VI communicates with the RF Function Generator (Right Agilent 33120A, GPIB address 11), Function Generator 13 (GPIB address 13), and the lock-in amplifier (Perkin-Elmer 7265, GPIB address 12).

To ramp the frequency, this program uses the frequency modulation mode of the RF Function Generator. After starting the sweep, it sets up Function Generator 13 in burst modulation mode. FG 13 emits a single square wave burst, which triggers the lock-in to record the RF data. The sweep continues for the length of one period (the inverse of the modulating frequency), and then the RF Function Generator is reset.

SUB VI'S REQUIRED:
"GPIB readwrite.vi"
"GPIB Error Report.vi"

FRONT PANEL:
Controls for carrier frequency, modulating frequency, carrier voltage, GPIB address, frequency deviation, number of cycles per burst, and output impedance are located on the front panel.

Default Control Values:

Carrier Voltage	1.85 Vrms
Carrier Frequency	80300 Hz
Modulating Frequency	.0857 Hz
GPIB Address	11
Output Impedance	50 ohms
Frequency Deviation	22500 Hz
Number of Cycles per burst	1

NORMAL OPERATION:

This VI normally runs within the "Modified NMR" VI. Its values are set by controls in the Modified NMR front panel.

DIAGRAM:

0		Resets Function Generator 11
1		Sets units to Vrms
2		Disables FM so parameters can be set properly.
3		Sets output impedance of the function generator to either 50 or infinity.
	True	Infinity
	False	50
4		Set shape of modulating waveform to USER.
5		Choose NMR WAVE as user waveform.
6		Set the carrier waveform to sine.
7		Set carrier voltage.
8		Set modulating frequency.
9		Set peak to peak deviation.
10		Set carrier frequency.
11		Turn on FM.
12		Set FG 13 to Burst Mode
13		Set units of FG 13 to Vpp.
14		Set voltage of FG 13 to 1.85.
15		Set shape of waveform to sine.
16		Set frequency to 91 kHz.
17		Set number of cycles in burst mode to 1 so that the lock-in is triggered only once.
18		Wait length of one period for the lock-in to read the data.
19		Reset FG 13.
20		Reset RF FG.

A.2 NMR Freq Agilent.vi

Written for William and Mary NMR system by:
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Erin Buckley, The College of William and Mary, Williamsburg, VA, embuc2@wm.edu
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Advisor: Todd Averett, The College of William and Mary, Williamsburg, VA, averett@wm.edu
7/16/03

PURPOSE:
This VI is designed to take NMR measurements by sweeping the RF frequency and maintaining a constant B-field in the main coils. It records the induced voltage in the pickup coils and graphs the results.

This VI communicates with the RF Function Generator (Right Agilent 33120A, GPIB address 11), Holding Field Function Generator (Left Agilent 33120A, GPIB address 10), the lock-in amplifier (PerkinElmer 7265, GPIB address 12), and an additional function generator (GPIB address 13), used only to trigger the lock-in amplifier.

Once the system is initialized, this VI uses the "Freq. Sweep" sub VI to sweep the field produced by the RF coils. This sub VI makes use of the frequency modulation function of the RF function generator. The RF function generator is sync'd to the lock-in. This SYNC signals the lock-in to start taking data at 100Hz. This data is stored in the lock-ins curve buffer and is retrieved after the sweep. The data is plotted and saved to the file "C:\NMR Data\3He\".

SUB VI'S REQUIRED:
"Freq. Sweep.vi", "NMR Save Single.vi"
"GPIB readwrite.vi"
"GPIB Error Report.vi"

FRONT PANEL:
The title "NMR Measurement" is placed in the top center.

The upper left indicators show the start and stop time of the measurement. The plots below these indicators graph the lock-in amplifier values of the pick-up coil voltage as a function of the frequency of the RF coils. The frequency values are calculated by the program rather than directly measured. Controls for the graphs are located in the teal panels on the right.

The stage of the program is shown in the upper right corner. Below this indicator is a "GO" button and a green light to show when the program is running. Underneath the "GO" button are three indicators, one which shows the date of the measurement, one which tells the current sweep number, and a third which relays the status of the X and Y channel download from the lock-in curve buffer.

Two vertical panels labeled “NMR Sweep Inputs” and “Frequency Sweep Inputs” allow the user to set the lock-in sensitivity, reference phase, number of sweeps, minutes between sweeps, sit time, carrier frequency and voltage, frequency deviation, and modulating frequency. Below these panels are indicators that show Auto Phase, Auto Offset, and Auto Sensitivity. The Measured Phase, Sensitivity, number of curve buffer points, and Sweep Period are shown on the bottom row of the front panel.

NORMAL OPERATION:

Before running this program, the RF function generator must be set to the proper setting using “Load Waveform (Freq. Sweep).vi”. This VI will prepare the function generator for the sweep by setting values and loading the waveform.

Default Control Values:

Lock-in Sensitivity	200mV
Reference Phase	-36
Number of Sweeps	1
Minutes between Sweeps	240
Sit Time	0
Carrier Frequency	80300 Hz
Carrier Voltage	1.85 Vrms
Frequency Deviation	22500 Hz
Modulating Freq.	.0857 Hz

A polarized target cell should be in place. The Kepco Power Supply should be connected to the Main Coils and controlled by the Holding Field Function Generator. The RF Amplifier should be connected to the RF Coils through the Resistance/Capacitance Box and controlled by the RF Function Generator. The lock-in amplifier should be connected to the preamp, triggered by Function Generator 13, and referenced to the RF Function Generator. The preamp should be connected to the pickup coils through the breakout box.

Preamp settings:
Gain = 1x10
Gain Mode = low noise
Filters = 10k, 100k
Roll off = 6dB/oct
AC Coupling
Mode = A-B

The lock-in sensitivity is typically set at 200mV. We have found that by setting the reference phase to –36 yields the best results. The carrier frequency should be set to 80300 Hz with a frequency deviation of 22500 Hz. The carrier voltage works best at 1.85 Vrms and the modulating frequency should be set to 0.0857 Hz. The number of sweeps and time in between them should also be selected.

After starting the program, click the "GO" button. Dialog boxes will pop up for Auto-Phase, Auto-Offset, and Auto-Sensitivity. Select "yes" for Auto-Offset, but not for Auto-Phase or Auto-Sensitivity.

DIAGRAM:

FRAME	DESCRIPTION
Outside	The large box is a while loop. The green light is reset to off. The "GO" button controls the T/F control that starts the rest of the program
False	
True	Initializations:
0	**Start Stage** indicated. Graphs are cleared, certain variables are zeroed.
1	Setup RF Function Generator and Function Generator 13.
0	**Initialization Stage**, RF Function Generator reset.
1	Function Generator 13 reset.
2	
0	Initialize lock-in. Set display to X volt, Mag, Phase, Y voltage.
1	Pause for command activation
2	Set reference mode, input mode.
3	Pause for command activation.
3	
0	Lock-in is setup. User is asked if Auto-Phase is wanted.
1	User is asked if Auto-Offset is wanted.
2	User is asked if Auto-Sensitivity is wanted.
4	Get time of day to set as Start Time
5	Begins For Loop for number of sweeps set by user.
0	Records date and time of sweep.
1	Sends commands to lock-in.
0	True Auto-Phase command sent, pauses for activation.
1	True Auto-Offset command sent, pauses for activation.
2	True Auto-Sensitivity command sent, pauses for activation, prompts user to see if sensitivity is set.
2	Read in Phase from lock-in.
0	Phase is requested from lock-in.
1	Phase is read from lock-in.
2	Phase is displayed.
3	
0	Calculate the number of Curve Buffer Points. Setup lock-in curve buffer to store X, Y, and Sensitivity at 100Hz when given an external trigger (from FG SYNC).
1	Wait for command activation.

2 **Sweeping Stage**. Run "Freq. Sweep.vi"
3 Wait for command activation.
4 **READING X** Stage. Dump X Channel to the computer.
5 X Values are recovered from the Lock-in.
While Loop Repeats until done (status bit 1 asserted)
An indication of progress is done in the lower left.
0 A value is read from the lock-in and converted to a number.
1
While Loop
Keeps checking the status register to see if another piece of data is ready (status 7) or is done (status 1).
6 **READING Y** Stage. Dump Y Channel to the computer.
7 Y Values are recovered from the Lock-in.
While Loop Repeats until done (status bit 1 asserted)
An indication of progress is done in the lower left.
0 A value is read from the lock-in and converted to a number.
1
While Loop
Keeps checking the status register to see if another piece of data is ready (status 7) or is done (status 1).
8 Frequency Values are calculated based on waveform parameters.
9 X Channel Data is graphed with Frequency Values and saved to "C:\NMR Data\3He\"
10 Y Channel Data is graphed with Frequency Values and saved to "C:\NMR Data\3He\"

4 Latest NMR Measurement file is deleted
5 Latest NMR Measurement file is saved with latest X Channel filename.
6 Checks to see if another sweep is needed. If so, download indicators are cleared and system waits according to front panel "Minutes between Sweeps" control.

6 Stop Time is indicated.
7 **COMPLETED Stage**. Green light turned off.
8 Program Stop.

A.3 tim fn gen.vi

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2/2/04

PURPOSE:
This VI is intended to perform an NMR sweep by ramping the frequency of the RF Coils through resonance, holding at the maximum frequency, then ramping back down. The lock-in amplifier is triggered through the code in "NMR freq HP tim.vi"

This VI communicates with the RF Function Generator (HP 3324A, GPIB address 11), Function Generator 13 (GPIB address 13), and the lock-in amplifier (Perkin-Elmer 7265, GPIB address 12).

To ramp the frequency, this program uses the multiple frequency sweep mode of the RF Function Generator. After starting the sweep, "NMR freq HP tim.vi" will instruct the lock-in amplifier to begin recording the RF data. The RF will sweep up once, hold at the maximum frequency to allow for water calibration, then sweep down and remain at that frequency.

SUB VI'S REQUIRED:
"GPIB readwrite.vi"
"GPIB Error Report.vi"

FRONT PANEL:
Controls for start/stop frequency, holding frequency, amplitude, sweep up time, hold time, water/helium toggle and GPIB address are located on the front panel.

Default Control Values:

Start/Stop frequency	85 kHz
Holding frequency	97 kHz
Amplitude	1.85 Vrms
Sweep up time	5.50 sec
Hold time	0.01 sec
Sweep type	Water
GPIB address	13

NORMAL OPERATION:
This VI normally runs within the "NMR freq HP tim" VI. Its values are set by controls in the "NMR freq HP tim.vi" front panel.

DIAGRAM:

0
- 0 Turn off RF output.
- 1 Pause for command activation.

1
- 0 Set amplitude
- 1 Pause for command activation.

2
- 0 Set waveform as a sine wave.
- 1 Pause for command activation.

3
- 0 Set start/stop frequency.
- 1 Pause for command activation.

4 Set parameters for ramp up: start sweep up frequency, stop sweep up frequency, marker frequency.

5 Set parameters for ramp down: start sweep down frequency, stop sweep down frequency, marker frequency.

6
- True Set holding conditions for water calibration setting.
- False Blank for Helium setting.

7 Turn on output. Start Sweep.

A.4 NMR Freq HP tim.vi

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7/16/03

PURPOSE:
This VI is designed to take NMR measurements by sweeping the RF frequency and maintaining a constant B-field in the main coils. It records the induced voltage in the pickup coils and graphs the results.

This VI communicates with the RF Function Generator (HP 3324A, GPIB address 13), Holding Field Function Generator (Left Agilent 33120A, GPIB address 10), the lock-in amplifier (PerkinElmer 7265, GPIB address 12), and an additional function generator (GPIB address 11), used only to trigger the lock-in amplifier.

Once the system is initialized, this VI uses the "tim fn gen.vi" sub VI to sweep the field produced by the RF coils. This sub VI makes use of the frequency sweeping function of the RF function generator. The RF function generator is sync'd to the lock-in. This SYNC signals the lock-in to start taking data at 100Hz. This data is stored in the lock-ins curve buffer and is retrieved after the sweep. The data is plotted and saved to the file "C:\NMR Data\3He\".

SUB VI'S REQUIRED:
"Hp init.vi", "tim fn gen.vi", "NMR Save Single.vi"
"GPIB readwrite.vi"
"GPIB Error Report.vi"

FRONT PANEL:
The title "NMR Measurement" is placed in the top center.

The upper left indicators show the start and stop time of the measurement. The plots below these indicators graph the lock-in amplifier values of the pick-up coil voltage as a function of the frequency of the RF coils. The frequency values are calculated by the program rather than directly measured. Controls for the graphs are located in the teal panels on the right.

The stage of the program is shown in the upper right corner. Below this indicator is a "GO" button and a green light to show when the program is running. Underneath the "GO" button are three indicators, one which shows the date of the measurement, one which tells the current sweep number, and a third which relays the status of the X and Y channel download from the lock-in curve buffer.

Two vertical panels labeled “NMR Sweep Inputs” and “Frequency Sweep Inputs” allow the user to set the lock-in sensitivity, reference phase, number of sweeps, minutes between sweeps, sit time, carrier frequency and voltage, frequency deviation, and modulating frequency. Below these panels are indicators that show Auto Phase, Auto Offset, and Auto Sensitivity. The Measured Phase, Sensitivity, number of curve buffer points, and Sweep Period are shown on the bottom row of the front panel.

NORMAL OPERATION:

Before running this program, the RF function generator must be set to the proper setting using “Load Waveform (Freq. Sweep).vi”. This VI will prepare the function generator for the sweep by setting values and loading the waveform.

Default Control Values:

Lock-in Sensitivity	200mV
Reference Phase	-36
Number of Sweeps	1
Minutes between Sweeps	240
Sit Time	0
Carrier Frequency	80300 Hz
Carrier Voltage	1.85 Vrms
Frequency Deviation	22500 Hz
Modulating Freq.	.0857 Hz

A polarized target cell should be in place. The Kepco Power Supply should be connected to the Main Coils and controlled by the Holding Field Function Generator. The RF Amplifier should be connected to the RF Coils through the Resistance/Capacitance Box and controlled by the RF Function Generator. The lock-in amplifier should be connected to the preamp, triggered by Function Generator 13, and referenced to the RF Function Generator. The preamp should be connected to the pickup coils through the breakout box.

Preamp settings:
Gain = 1x10
Gain Mode = low noise
Filters = 10k, 100k
Roll off = 6dB/oct
AC Coupling
Mode = A-B

The lock-in sensitivity is typically set at 200mV. We have found that by setting the reference phase to –36 yields the best results. The carrier frequency should be set to 80300 Hz with a frequency deviation of 22500 Hz. The carrier voltage works best at 1.85 Vrms and the modulating frequency should be set to 0.0857 Hz. The number of sweeps and time in between them should also be selected.

After starting the program, click the "GO" button. Dialog boxes will pop up for Auto-Phase, Auto-Offset, and Auto-Sensitivity. Select "yes" for Auto-Offset, but not for Auto-Phase or Auto-Sensitivity.

DIAGRAM:

FRAME			DESCRIPTION
Outside			The large box is a while loop. The green light is reset to off. The "GO" button controls the T/F control that starts the rest of the program
False			
True			Initializations:
0			**Start Stage** indicated. Graphs are cleared, certain variables are zeroed.
1			Setup RF Function Generator and Function Generator 13.
	0		**Initialization Stage**, RF Function Generator reset.
	1		Pause for command activation.
	2		Function Generator 13 reset. "tim fn gen.vi" is initialized and called.
	3		Pause for command activation.
2			
	0		Initialize lock-in. Set display to X volt, Mag, Phase, Y voltage.
	1		Pause for command activation.
	2		Set reference mode, input mode.
	3		Pause for command activation.
3			
	0		Lock-in is setup. User is asked if Auto-Phase is wanted.
	1		User is asked if Auto-Offset is wanted.
	2		User is asked if Auto-Sensitivity is wanted.
4			Get time of day to set as Start Time
5			Begins For Loop for number of sweeps set by user.
	0		Records date and time of sweep.
	1		Lock-in amp is triggered.
	2		Read in Phase from lock-in.
		0	Phase is requested from lock-in.
		1	Phase is read from lock-in.
		2	Phase is displayed.
		3	Lock-in sensitivity is set.
	3		Read in phase from lock-in
	4		
		0	Calculate the number of Curve Buffer Points. Setup lock-in curve buffer to store X, Y, and Sensitivity at 100Hz when given an external trigger (from FG SYNC).

1 Wait for command activation.
2 **Sweeping Stage**. Run "Freq. Sweep.vi"
3 Wait for command activation.
4 **READING X** Stage. Dump X Channel to the computer.
5 X Values are recovered from the Lock-in.
While Loop Repeats until done (status bit 1 asserted)
An indication of progress is done in the lower left.
0 A value is read from the lock-in and converted to a number.
1
While Loop
Keeps checking the status register to see if another piece of data is ready (status 7) or is done (status 1).
6 **READING Y** Stage. Dump Y Channel to the computer.
7 Y Values are recovered from the Lock-in.
While Loop Repeats until done (status bit 1 asserted)
An indication of progress is done in the lower left.
0 A value is read from the lock-in and converted to a number.
1
While Loop
Keeps checking the status register to see if another piece of data is ready (status 7) or is done (status 1).
8 Frequency Values are calculated based on waveform parameters.
9 X Channel Data is graphed with Frequency Values and saved to "C:\NMR Data\3He\"
10 Y Channel Data is graphed with Frequency Values and saved to "C:\NMR Data\3He\"

5 Latest NMR Measurement file is deleted
6 Latest NMR Measurement file is saved with latest X Channel filename.
7 Checks to see if another sweep is needed. If so, download indicators are cleared and system waits according to front panel "Minutes between Sweeps" control.

6 Stop Time is indicated.
7 **COMPLETED Stage**. Green light turned off.
8 Program Stop.