



Terranova-MRI

Earth's field MRI & NMR teaching system

Hands on learning MRI & NMR principles

STUDENT GUIDE

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Caution and Safety Considerations

A DC current of up to 6 A is pulsed through the outermost polarising coil of this instrument. When using large currents for long time periods, the coil will become warmer. Caution must be taken not to allow the coil to become too hot. Under normal operating conditions the temperature of the coil should not exceed 40°C. If under extreme operating conditions, the surface temperature of the coil does exceed 40°C, halt use of the apparatus until the temperature of the coil returns to normal.

The probe also generates a small magnetic field when in operation. Although this field is small, it could potentially damage credit cards or watches, or other magnetically sensitive devices if they are left close to the probe. It is suggested that magnetically sensitive devices are kept at a distance of at least 3 m from the probe.

Preface

The purpose of this teaching manual is to provide a collection of self-contained student experiments that can be carried out using the Terranova-MRI apparatus. Each of these experiments will demonstrate and teach different principles of physics, NMR and MRI. They can be used as a unit or individually depending on the requirements of the course.

Each experiment contains an objective, background theory, a step-by-step guide to the experiment itself and a list of potential follow-up questions. Some of the follow-up questions will deliberately lead the student to consider topics that are covered in subsequent experiments as a means of providing continuity between the experiments. Throughout the experimental procedure questions will be posed to the student and some steps in the procedure will be left to the student to determine on their own. The intention is to make the student do some independent thinking and to require the student to extrapolate answers from the background information and the hints given.

The experiments are divided into three categories: Part I - Introductory Experiments, Part II – Earth's field NMR Experiments and Part III – Earth's field MRI Experiments. Part I is intended to take the student through the initial setup of the Terranova-MRI apparatus and the acquisition of a good quality NMR signal. These experiments focus in great detail on how to obtain a good quality NMR signal and explore the role of several important acquisition parameters and their effect on signal quality. Part II provides an overview of some of the fundamental topics of NMR. These include T_1 , T_2 and T_2^* relaxation time measurements, spin-echoes and multiple-echo sequences such as CPMG. Part III covers topics of interest in the field of MRI. These experiments cover basic 1D and 2D image acquisition using spin-echo, gradient echo and filtered back projection imaging methods, as well as more advanced topics such as image contrast.

An “appendix for the instructor” section is included with each experiment to provide the instructor with the answers to any advanced questions posed in the experiments and also to provide a more detailed description of the procedure steps that are left to the student to determine in the experiment. Thus the level of difficulty of each experiment can be tailored as desired. The appendix will also provide some example data. This data is included to provide the instructor with an idea of the type of results that should be obtained in each experiment.

Terranova-MRI EFNMR Student Guide

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Table of Contents

Preface	iii
Table of Contents	v
1. Basic NMR Signal Acquisition	1-1
1.1. Objective	1-1
1.2. Apparatus	1-1
1.3. Background Theory	1-3
1.3.1. Introduction to NMR	1-3
1.3.2. LCR Resonant Circuits	1-4
1.4. Procedure	1-6
1.4.1. Instrument Setup	1-6
1.4.2. The NMR Pulse Program	1-11
1.4.3. Initial Parameter Determination	1-12
1.4.4. Acquire an FID	1-14
1.4.5. Optimising the NMR parameters	1-15
1.5. Further Questions	1-16
1.6. Appendix for the Instructor	1-16
2. Shimming and Parameter Optimisation	2-1
2.1. Objective	2-1
2.2. Apparatus	2-1
2.3. Background Theory	2-1
2.3.1. Magnetic field homogeneity and shimming	2-1
2.3.2. Pre-polarisation	2-2
2.4. Procedure	2-2
2.4.1. Getting Started	2-2
2.4.2. Shimming	2-2
2.4.3. Auto-shimming	2-3
2.4.4. Parameter optimisation	2-5
2.4.5. Pre-polarising pulse duration	2-6
2.5. Further Questions	2-7
2.6. Appendix for the Instructor	2-7
3. Spin-Lattice (Longitudinal) Relaxation: T_1	3-1
3.1. Objective	3-1
3.2. Apparatus	3-1
3.3. Background Theory	3-1
3.3.1. Spin-Lattice Relaxation	3-1
3.4. Procedure	3-2
3.4.1. Getting Started	3-2
3.4.2. Measuring T_1 in the Polarising Field	3-2
3.4.3. Measuring T_1 in Earth's Magnetic Field	3-3
3.4.4. The Spin-Lattice Relaxation of Oil	3-5
3.5. Further Questions	3-5
3.6. Appendix for the Instructor	3-5
4. Spin-echoes and Spin-Spin (T_2) Relaxation	4-1
4.1. Objective	4-1
4.2. Apparatus	4-1
4.3. Background Theory	4-1
4.3.1. Spin-Spin and Effective Spin-Spin Relaxation	4-1
4.4. Procedure	4-2
4.4.1. Getting Started	4-2

4.4.2. Spin-Echoes	4-2
4.4.3. Magnetic Field Inhomogeneity and the FID	4-3
4.4.4. T_2 and T_2^* Relaxation	4-4
4.4.5. T_2 Measurement	4-5
4.5. Further Questions	4-6
4.6. Appendix for the Instructor	4-6
5. Multiple-echo experiments: CPMG	5-1
5.1. Objective	5-1
5.2. Apparatus	5-1
5.3. Background Theory	5-1
5.3.1. Carr-Purcell-Meiboom-Gill (CPMG) Pulse Sequence	5-1
5.3.2. Time Domain Filters	5-2
5.4. Procedure	5-4
5.4.1. Instrument Setup	5-4
5.4.2. Explore Spin-echo Parameters and Processing Options	5-4
5.4.3. Multiple Echo Acquisition with the CP Sequence	5-6
5.4.4. The Carr-Purcell-Meiboom-Gill (CPMG) Sequence	5-6
5.4.5. Single-shot T_2 measurements using CPMG	5-6
5.5. Further Questions	5-6
5.6. Appendix for the Instructor	5-6
6. Relaxation Time Contrast	6-1
6.1. Objective	6-1
6.2. Apparatus	6-1
6.3. Background Theory	6-1
6.3.1. Contrast in NMR Imaging	6-1
6.3.2. Paramagnetic Relaxation Time Contrast Agents	6-1
6.4. Procedure	6-2
6.4.1. Getting Started	6-2
6.4.2. ^1H NMR of H_2O doped with CuSO_4	6-2
6.4.3. Relaxation Time Measurement of CuSO_4 solutions	6-2
6.5. Further Questions	6-2
6.6. Appendix for the Instructor	6-3
7. Magnetic Resonance Imaging in 1D	7-1
7.1. Objective	7-1
7.2. Apparatus	7-1
7.3. Background Theory	7-1
7.3.1. Magnetic Field Gradients in MRI	7-1
7.3.2. MRI Fourier Transform Formalism: k -space	7-2
7.3.3. Gradient Echo Imaging	7-3
7.4. Procedure	7-4
7.4.1. Getting Started	7-4
7.4.2. 1D Imaging	7-4
7.4.3. Gradient echo imaging	7-6
7.4.4. Exploring Imaging Parameters	7-9
7.5. Further Questions	7-10
7.6. Appendix for the Instructor	7-10
8. 2D MRI: Gradient echo imaging	8-1
8.1. Objective	8-1
8.2. Apparatus	8-1
8.3. Background Theory	8-1
8.3.1. Phase Encoding	8-1
8.4. Procedure	8-3
8.4.1. Getting started	8-3
8.4.2. Improving Imaging Efficiency	8-3
8.4.3. 2D Imaging	8-5
8.5. Further Questions	8-7

8.6. Appendix for the Instructor	8-7
9. 2D MRI: Spin-echo imaging	9-1
9.1. Objective	9-1
9.2. Apparatus	9-1
9.3. Background Theory	9-1
9.3.1. Spin-echo imaging	9-1
9.3.2. Phase Cycling	9-2
9.4. Procedure	9-5
9.4.1. Getting started	9-5
9.4.2. Spin-echo imaging	9-5
9.4.3. Filtering and Phase Cycling	9-7
9.5. Further Questions	9-8
9.6. Appendix for the Instructor	9-8
10. 2D MRI: Filtered back projection imaging	10-1
10.1. Objective	10-1
10.2. Apparatus	10-1
10.3. Background Theory	10-1
10.3.1. Filtered back projection: the pulse sequence	10-1
10.3.2. Filtered back projection: image processing	10-2
10.4. Procedure	10-3
10.4.1. Getting Started	10-3
10.4.2. Filtered back projection (FBP) image acquisition	10-3
10.5. Further Questions	10-7
10.6. Appendix for the Instructor	10-7
11. Relaxation contrast imaging	11-1
11.1. Objective	11-1
11.2. Apparatus	11-1
11.3. Background Theory	11-1
11.3.1. Relaxation contrast in MRI	11-1
11.4. Procedure	11-1
11.4.1. Getting Started	11-1
11.4.2. T_1 weighted imaging	11-2
11.4.3. T_2 weighted imaging	11-2
11.5. Further Questions	11-3
11.6. Appendix for the Instructor	11-3



Terranova-MRI

Earth's field MRI & NMR teaching system

Introductory Experiments

1. Basic NMR Signal Acquisition

1.1. Objective

The object of this experiment is to setup the Earth's field NMR device and to acquire a hydrogen (^1H) nuclear magnetic resonance (NMR) signal, called a free induction decay (FID), from a tap water sample. The principles of NMR in the Earth's magnetic field and LCR resonant circuits are introduced and explored.

1.2. Apparatus

This experiment is performed using the Terranova-MRI EFNMR apparatus. The sample is a large 500 ml bottle of tap water. Other pieces of equipment that are required include: a Magnaprobe™ 3D compass, a 24 V / 6.5 A DC power supply and a PC running the *Prospa* software package.

For information on the power supply and PC requirements and instructions on the installation of the *Prospa* and *Terranova* software packages, please refer to the Terranova-MRI User's Manual.



Figure 1.1. The Earth's Field NMR Probe and axis conventions

The Terranova-MRI EFNMR apparatus (Figure 1.1 and Figure 1.2) consists of 2 coaxial solenoids and a third which is a collection of 4 coils generating x , y and z gradients (there are two x gradients, one for imaging and shimming, and one for pulsed gradient NMR). The probe is designed such that the three coils slot together in a bayonet configuration. This configuration allows for easy access to the three constituent coils. However, the probe is not designed to be dismantled and therefore should not be taken apart. The outermost, or polarising (B_p), coil is used to provide an initial polarising field via the application of a large current - establishing a nuclear magnetisation in a sample placed inside the apparatus. The middle, or gradient coils are used to provide linear magnetic field gradients across the sample. The inner most, or B_1 , coil is used to excite and detect the precessing magnetisation.

Table 1.1. Typical coil parameters

Coil Parameter	Polarising Coil	Gradient Coils (pgse,x,y,z)	B_1 Coil
Avg. coil diameter (mm)	170	105	84
Calculated magnetic field (mT/A)	3.13	N/A	30
Field gradient (mT/m/A)	N/A	2.38,0.31,0.28,0.28	N/A
Avg. resistance (Ω) incl. cable	2.8	3.1/2.0/1.5/1.5	325
Tuning cap for 2300 Hz (nF)	N/A	N/A	9.7

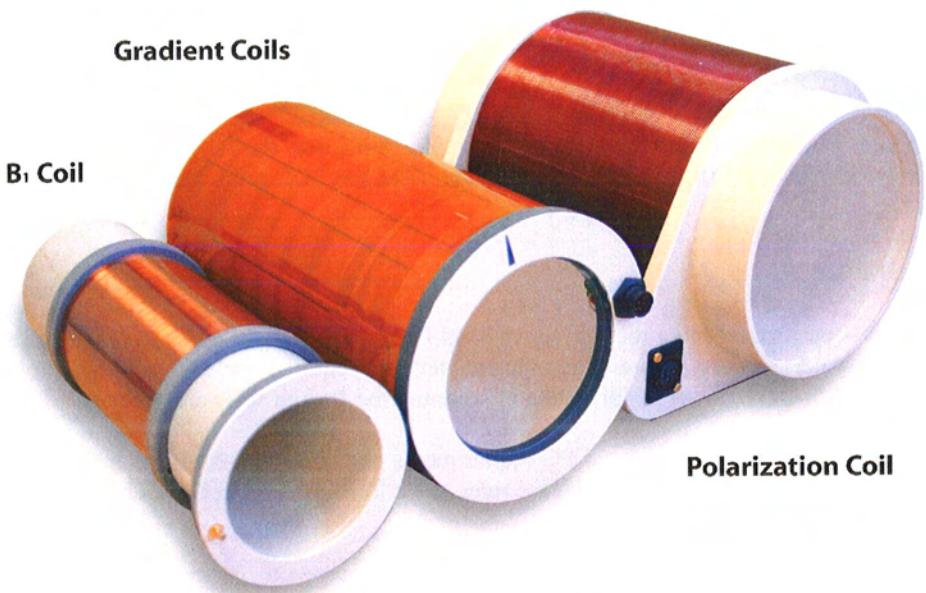


Figure 1.2. A view of the three EFNMR/MRI coils

The EFNMR experiment is controlled by a digital signal processor (DSP), which is itself controlled by a personal computer (PC) running the data processing package *Prospa*. In a typical experiment *Prospa* sends a precompiled DSP pulse program and all necessary parameters to the DSP unit via the PC's USB port. It then starts this program running.

This program controls the complete NMR experiment, sending pulses to the B_p , gradient and B_1 coils and then acquiring the NMR data at the appropriate time. While the pulse program is running on the DSP, *Prospa* waits for a signal from the DSP to indicate that data is available for uploading. Once this data has been collected, it is displayed, any required analysis is performed, and then the experiment is ended, or if desired, repeated, perhaps with new parameters being sent to the DSP.

The entire EFNMR system is pictured in Figure 1.3; the various connections between components are shown and labelled. The DSP is contained within the central box of electronics, called the *spectrometer*. The spectrometer is connected to the PC via a USB interface. Additional cables connect the spectrometer to its power source and to the B_1 , gradient and polarisation coils.

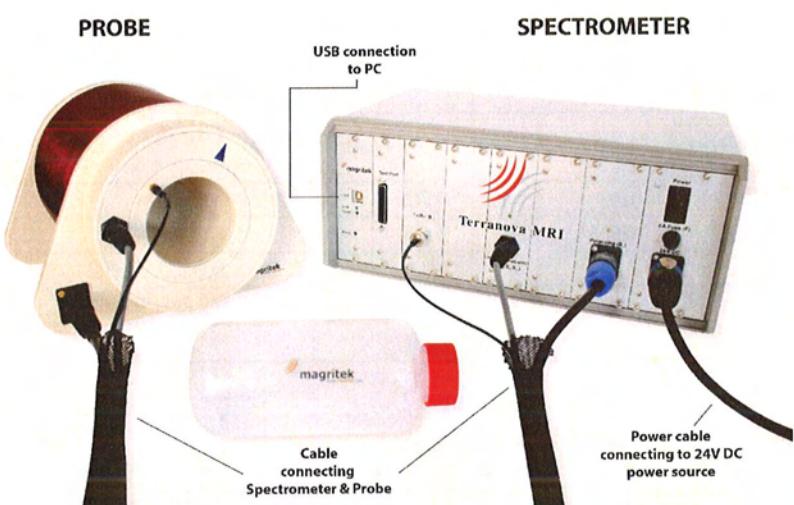


Figure 1.3. The EFNMR system: Spectrometer and Probe

1.3. Background Theory

1.3.1. Introduction to NMR

The nuclear magnetic resonance (NMR) phenomenon is dependent on a nuclear property called spin. Nuclei with a spin quantum number, I , greater than or equal to $\frac{1}{2}$ possess a magnetic moment, which tends to align either parallel or anti-parallel to an externally applied magnetic field, B_0 . From quantum mechanics it can be derived that the number of discrete orientations of a nuclear magnetic moment in the presence of a uniform external magnetic field is given by $2I+1$, each corresponding to a spin quantum number $m = -I \dots I$.

There are many nuclei, such as ^{23}Na (spin $3/2$) and ^{17}O (spin $5/2$), with spin quantum numbers greater than $\frac{1}{2}$. These nuclei are very interesting in the field of NMR because they possess a non-zero quadrupole moment and consequently exhibit exotic phenomena such as multiple-quantum coherences. However, for the scope of this experiment we will only consider the simplest case of $I = \frac{1}{2}$. In this case there are only two possible orientations of the nuclear magnetic moment: spin-up ($m = \frac{1}{2}$) and spin-down ($m = -\frac{1}{2}$).

The spin-up and spin-down states possess an energy, $E_m = -\gamma\hbar m B_0$, where γ is the gyromagnetic ratio of the nuclei of interest, \hbar is Planck's constant divided by 2π , and B_0 is the external static magnetic field. Therefore spin-up is the preferred lower energy state when γ is positive.

The relative populations of the spin-up and spin-down states are governed by Boltzmann statistics. In the absence of thermal motions all of the spins would populate the lower energy spin-up state. However, under more temperate conditions the population difference is very small, as shown by equation 1-1.

$$\frac{P_{\uparrow}}{P_{\downarrow}} = \exp\left(\frac{\gamma\hbar B_0}{kT}\right) \quad [1-1]$$

Although very small, this population difference gives rise to a bulk magnetisation parallel to the external magnetic field. The bulk magnetisation for an ensemble of N spins can be computed using Boltzmann statistics. In general, for all values of I , the magnetisation can be written as

$$M = N\gamma\hbar \frac{\sum_{m=-I}^I m \exp\left(\frac{\gamma\hbar B_0}{kT}\right)}{\sum_{m=-I}^I \exp\left(\frac{\gamma\hbar B_0}{kT}\right)} \quad [1-2]$$

Using the high temperature approximation $kT \gg |\gamma\hbar m B_0|$, a linear expansion for the Boltzmann exponential term can be substituted into equation 1-2 to yield

$$M = \frac{N\gamma^2\hbar^2 B_0}{kT} \frac{\sum_{m=-I}^I m^2}{2I+1} = \frac{N\gamma^2\hbar^2 I(I+1)B_0}{3kT} \quad [1-3]$$

The net magnetisation vector, as defined in equation 1-3, is directed along the longitudinal axis of the laboratory frame, defined by the direction of the static magnetic field vector, \mathbf{B}_0 . The plane perpendicular to this axis is called the transverse plane. At equilibrium, each spin in the ensemble precesses about the static magnetic field vector, \mathbf{B}_0 , in a manner that is often likened to a spinning top. The consequence of this precession is that the local magnetic moment of each individual spin has a component in both the longitudinal and transverse directions. However, the phase of precession of each spin is random and so in the ensemble average the transverse components cancel leaving a net magnetisation vector directed along the longitudinal axis.

In a basic NMR experiment, the bulk magnetisation vector is manipulated through the application of an alternating electromagnetic field pulse. In the quantum mechanical picture, this alternating

1-4 Introductory Experiments

electromagnetic field pulse induces transitions between the energy levels corresponding to $m = \frac{1}{2}$ (spin-up) and $m = -\frac{1}{2}$ (spin-down) and introduces a phase coherence between the wavefunctions of the spins. The energy difference between these levels is given by equation 1-4.

$$\Delta E = \gamma \hbar B_0 \quad [1-4]$$

Therefore the frequency of the pulse is given by equation 1-5, which is called the Larmor equation and the frequency the Larmor frequency.

$$\omega = \gamma B_0 \quad [1-5]$$

This transition frequency is the same as the frequency with which the spins precess about the static magnetic field vector as derived from classical mechanics.

In conventional NMR applications, where the static field B_0 is large, the Larmor frequency is in the radio-frequency range and it is therefore referred to as an RF pulse. In Earth's field NMR applications, this pulse falls in the ultra-low frequency (ULF) band (300 – 30 kHz) and so is properly called an ULF pulse. However, in order to conform to the terminology most frequently encountered in the fields of NMR and MRI, we will henceforth refer to our pulses as RF pulses.

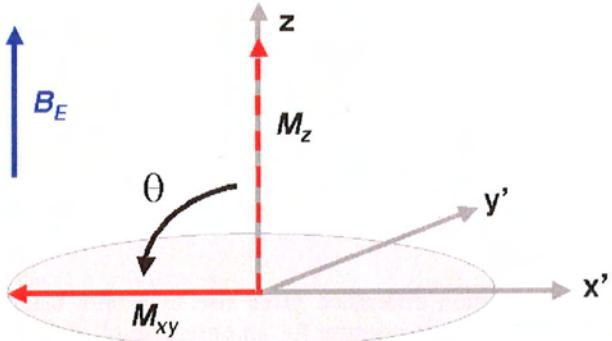


Figure 1.4. The RF pulse tips the magnetisation, M_z , into the transverse plane by a tip angle θ . The direction of the Earth's magnetic field, B_E , defines the z direction.

In the classical picture, the equilibrium magnetisation of a sample in an external static magnetic field lies along the longitudinal or z axis of the laboratory frame (see Figure 1.4). In the case of EFNMR this external static magnetic field is B_E , the Earth's magnetic field. The direction of B_E defines the direction of z . The RF pulse disturbs this bulk magnetisation vector, M_z , from equilibrium and rotates it into the transverse plane by an angle, θ , called the tip angle. This excited magnetisation vector, M_{xy} , precesses about the Earth's magnetic field vector, i.e. about the z direction, at the Larmor frequency. The B_1 coil detects this precessing magnetisation. The record of this precessing magnetisation is called the free induction decay (FID). Note that only the component of the precessing magnetisation that lies in the transverse plane is detected.

1.3.2. LCR Resonant Circuits

The B_1 coil, when connected to the spectrometer, forms a parallel LCR circuit. The B_1 coil is an inductor with an inductance, L , and an internal resistance, R . It is connected in parallel to a fixed capacitance, C , within the spectrometer. This resonant circuit is used to detect the EFNMR signal. The circuit will resonate at a frequency, ω_0 , given by:

$$\omega_0 = \frac{1}{\sqrt{LC}} \quad [1-6]$$

The equivalent circuit diagram for the LCR circuit, when the system is in “receive” mode, is shown in Figure 1.5. The input voltage, V_s , is induced by the precessing nuclear magnetism of the sample.

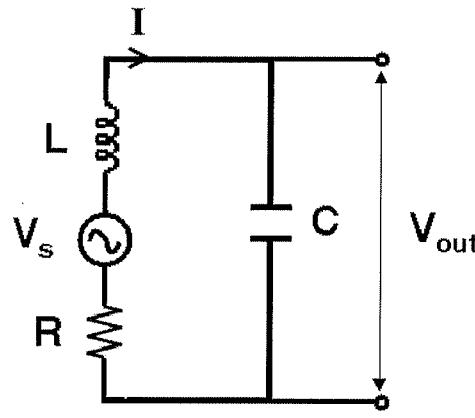


Figure 1.5. Circuit diagram for the B_1 coil in receive mode.

At resonance, when $\omega_0 = \frac{1}{\sqrt{LC}}$, the imaginary term in the denominator will go to zero and so the current will be:

$$I = \frac{V_s}{R}.$$

Therefore the output voltage at resonance is

$$|V_{out}| = |IZ_c| = \frac{|V_s|}{R\omega_0 C} = \frac{|V_s|\omega_0 L}{R} = |V_s|Q \quad [1-7]$$

where Q is the quality factor. Thus the resonant circuit amplifies the signal voltage by a factor of Q .

The definition of the quality factor (equation 1-8) can be understood by examining the resonance curve plot (Figure 1.6).

$$Q = \frac{f_0}{\Delta f} = \frac{\omega_0}{\Delta\omega} \quad [1-8]$$

The Q factor is a measure of the width of the resonance peak, the higher the Q factor the narrower the peak and hence the greater the amplification of the LCR circuit. In terms of L , C and R , the quality factor is given by equation 1-9.

$$Q = \frac{\omega_0 L}{R} \quad [1-9]$$

The equivalent impedance of the circuit in Figure 1.5 (as seen by V_s) is

$$Z_{eq} = Z_R + Z_L + Z_C$$

$$Z_{eq} = R + i(\omega L - \frac{1}{\omega C})$$

Therefore the current through the circuit can be written as:

$$I = \frac{V_s}{Z_{eq}} = \frac{V_s}{R + i(\omega L - \frac{1}{\omega C})}.$$

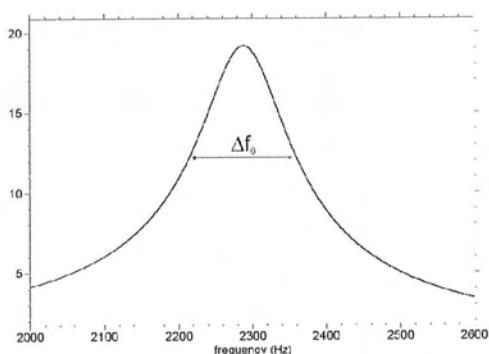


Figure 1.6 A resonance peak with the width, Δf_0 , defined to be 3 dB below the maximum.

In NMR it is the signal to noise ratio rather than just the signal level that is of significance and although the tuned circuit increases the signal level by a factor of Q so too does it increase the noise. However, this is only true at resonance. At other frequencies there will be considerable attenuation and so the effect of the tuned circuit is to boost the signal to noise ratio of the detected signal in the time domain (provided the detection bandwidth is larger than the linewidth of the NMR signal.)

1.4. Procedure

1.4.1. Instrument Setup

Review section 1.2, which provides information about the Terranova-MRI apparatus. To obtain the best performance from the EFNMR apparatus it is important to choose the location of the instrument carefully. The instrument should not be used within a couple metres of any ferrous objects (e.g. chairs and tables with metal parts). Switch off any source of low frequency electrical noise in the immediate vicinity (e.g. some fluorescent lights and CRT computer monitors). It is often beneficial to place the EFNMR probe on a wooden bench or stand. Also, the centre of the room (as far from the floor, ceiling and walls as possible) is frequently a good location.

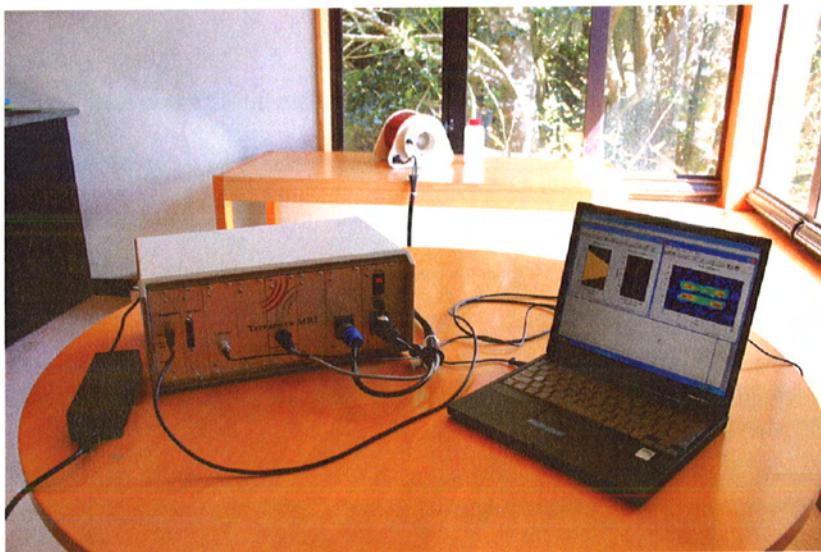


Figure 1.7 A photo of the entire Terranova-MRI system setup. Note that the probe is on a wooden table far from the spectrometer, the power supply and the PC.

Try and keep electrical equipment, especially the power supply, as far away from the probe as possible. Do **NOT** put the probe on top of the spectrometer, but rather as far from the spectrometer and power supply as possible. Figure 1.7 shows a typical arrangement of the Terranova-MRI system, where the power supply, spectrometer and PC are kept at a distance of several meters from the probe and the probe is set on a wooden table.

Identify the three major components of the system: the probe, the spectrometer and the power supply. Connect up the three components of the EFNMR probe to the spectrometer using the cable provided, as illustrated in Figure 1.8 and Figure 1.9.

STEP 1
Connect the polarising coil



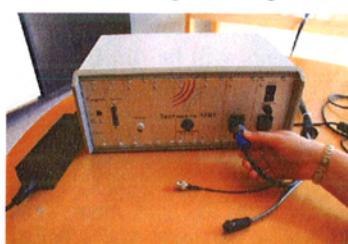
STEP 2
Connect the gradient coil



STEP 3
Connect the B_1 coil



STEP 4
Connect the polarising coil



STEP 5
Connect the gradient coil



STEP 6
Connect the B_1 coil



Figure 1.9 Connecting the Earth's Field MRI spectrometer.

STEP 7
Connect the power supply



Figure 1.10

STEP 8
Connect the USB cable

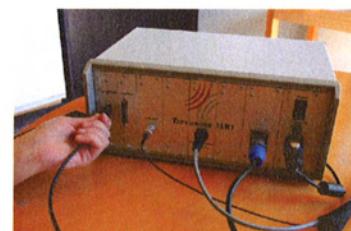


Figure 1.11

Connect the power supply to the spectrometer as depicted in Figure 1.10. Connect the PC to the spectrometer via the USB cable provided. The USB cable connects to the spectrometer as illustrated in Figure 1.11 .

Switch on the spectrometer. A red power light will illuminate on the far left panel of the spectrometer, below the USB connection. If the power light does not illuminate, check that the power supply is properly plugged into the mains power. Once the power supply is properly plugged in and the spectrometer is successfully connected to the PC, a green data light will illuminate above the red power light on the spectrometer. Note that the green light will only be illuminated once the USB driver has been installed on your computer. For more information on installing this driver refer to the Terranova-MRI User Manual.

Having successfully connected the system, it is time to consider some important factors regarding probe placement. The orientation of the probe is very important for the quality of experimental results and can also greatly affect the magnitude of detected noise. At your chosen location for the probe, measure the Earth's field direction using a conventional compass. Rotate the probe so that its longitudinal (x) axis is orthogonal to this direction (this optimises the action of the 90° and 180° pulses, increases the signal level slightly and is important for the magnetic field gradients used to perform the imaging experiments). In addition, the arrow marked on the end of the probe (indicating the axis of the z-imaging gradient) should be directed along the Earth's field axis, this can be determined using the supplied Magnaprobe™ 3 axis compass (see Figure 1.12). This may necessitate tilting the EFNMR probe slightly or in severe cases mounting the probe on a cradle so it can be rotated easily.



Figure 1.12. Photos of the EFNMR probe showing the correct orientation to optimise imaging results.

Now that the instrument is connected and properly oriented, the next step is to run some basic experiments to evaluate the system and the chosen location. These experiments will be run from the *Prospa* software on your PC. (Refer to the Terranova-MRI User Manual for instructions on installing *Prospa*.)

To start *Prospa*, double click on the shortcut on your desktop or run *Prospa* from the start menu. The *Prospa* window, pictured in Figure 1.13, will appear. At the top of the window you will find a list of menus.

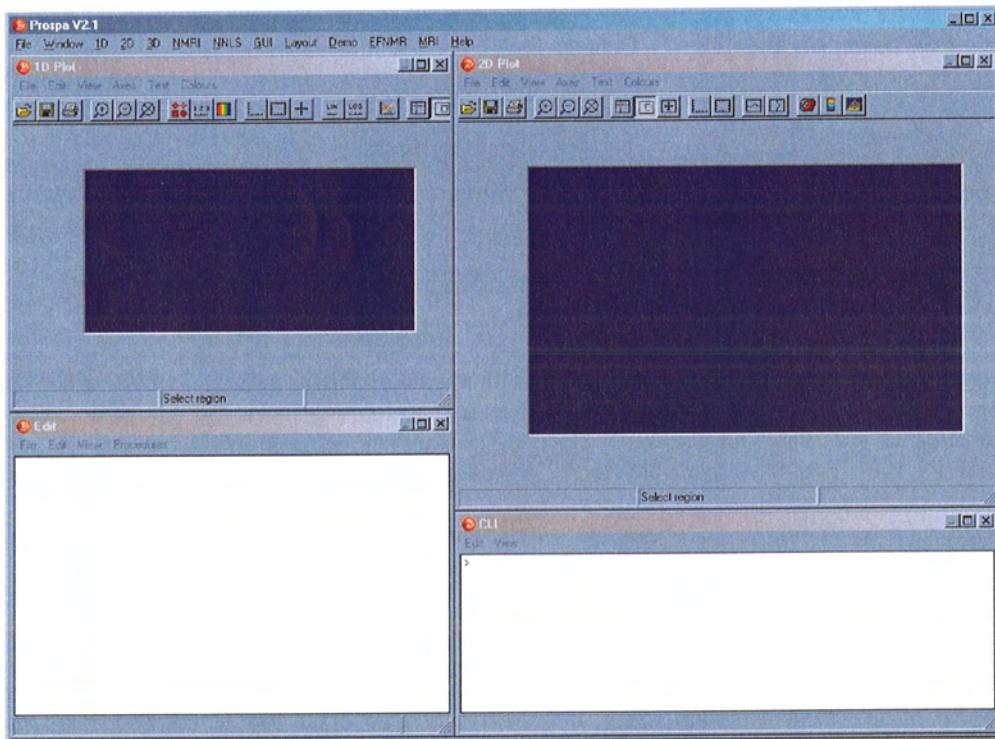


Figure 1.13 The *Prospa* window

The Earth's field NMR and MRI experiments can be accessed from the "MRI" and "EFNMR" menus. Select *PulseAndCollect* from under the EFNMR menu. The dialog shown in Figure 1.14 will appear.

All experiments will be run from dialog windows, such as the one shown in Figure 1.14. These windows allow the user to control a given experiment by choosing values for all experimental parameters and selecting the destination folder and file names for the output of the experiment. An experiment is executed by clicking the "Run" button. An experiment may be halted at any time by either clicking the "Stop" button or by pressing the "Esc" key. The former method allows the

apparatus to finish the current scan before aborting the experiment. The latter method halts the experiment immediately.

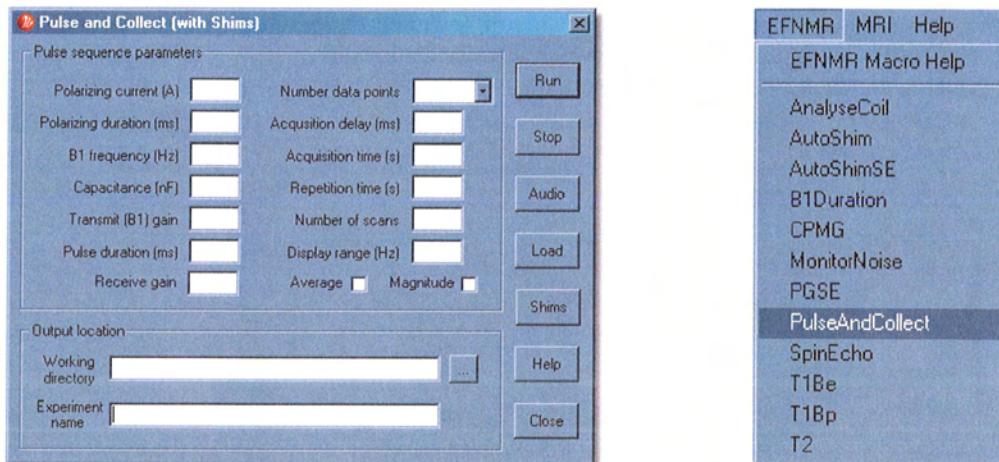


Figure 1.14 The Pulse and Collect Experiment Dialog

The first step to assessing the system and the choice of location is to characterise the B_1 coil so that the system can later be tuned to the resonant frequency of the observed nucleus.

From under the EFNMR menu, choose the experiment: *AnalyseCoil*. The dialog window in Figure 1.15 will appear.

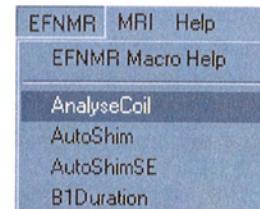


Figure 1.15 Analyse Coil Dialog Window

Choose a working directory by clicking the “...” button and navigating to an appropriate location on your PC. This is the location where the data from this experiment will be saved. The experiment name is the name of the folder, created within your working directory, to which the data from this experiment will be saved. The setting of a working directory and experiment name is a common feature of all experiments.

The B_1 coil is a tuned LCR circuit, as discussed in section 1.3.2. The frequency at which the coil resonates is given by equation 1-6 and is therefore determined by the equivalent capacitance, C , and inductance, L , of the circuit. The AnalyseCoil experiment characterises the B_1 transmit/receive coil by applying an impulse to the B_1 coil and detecting the response. This procedure is repeated over a range of capacitance values in order to determine the parameters of the B_1 coil. At each value of C , the resonance response is measured as a function of time. This time domain signal is Fourier transformed to yield the frequency response of the circuit. A typical experiment output is shown in Figure 1.16. On the far left is the time domain response of the coil. In the centre is the frequency response. In the frequency domain we observe a peak (reminiscent of that shown in Figure 1.6) at the resonance frequency of the coil. On the far right is a plot of resonant frequency as a function of the applied capacitance. This curve can be analysed, using equation 1-6, to determine the inductance of the coil as well as the parasitic capacitance of the coil, i.e. the capacitance in the LCR circuit due to the coil itself not the software controlled capacitors within the spectrometer. The coil inductance and capacitance can in turn be used to determine the self-resonance of the coil, i.e. the frequency at which the coil would resonate if no external capacitance was connected.

Execute the *AnalyseCoil* experiment by clicking “Analyse”. Record the output values for coil capacitance, inductance and self-resonance printed to the command line interface (CLI) window (pictured on the right). You will need to use some of these values for calculations later in the experiment. Close the *AnalyseCoil* window.

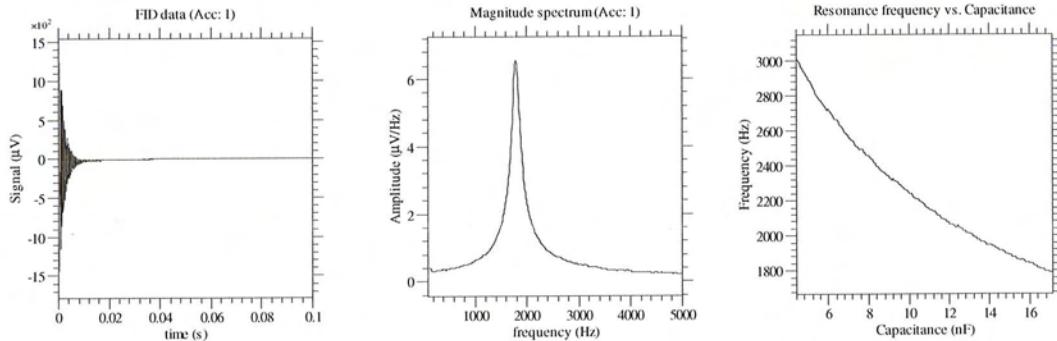
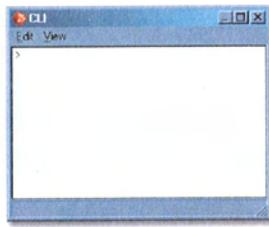


Figure 1.16. Sample output from the *AnalyseCoil* macro.

The next step in the setup procedure is the measurement of the external noise level detected by the coil. This noise level is very environmentally sensitive and so is very dependent on both the location and orientation of the probe. Select the experiment *MonitorNoise* from under the EFNMR menu. The window in Figure 1.17 will appear.

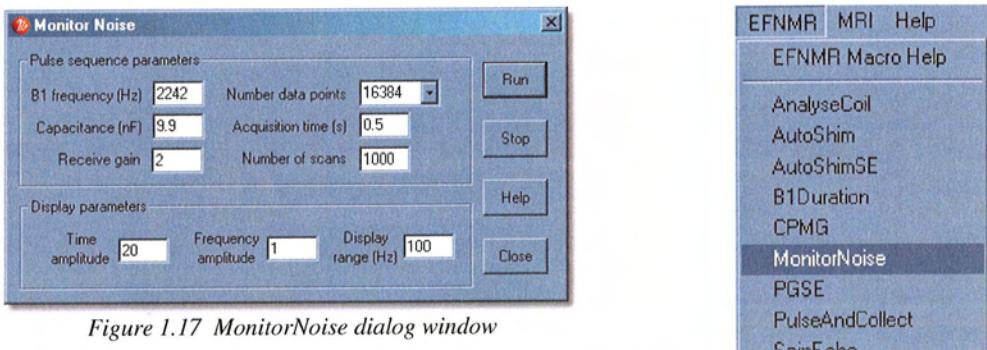


Figure 1.17. *MonitorNoise* dialog window

The *MonitorNoise* experiment acquires data in the absence of any NMR signal excitation and therefore only records noise. The output of this experiment (Figure 1.20) displays both the time domain and frequency domain noise. The root-mean-square (RMS) noise value, listed at the top of the time domain noise plot, is a measure of the noise level in the time domain.

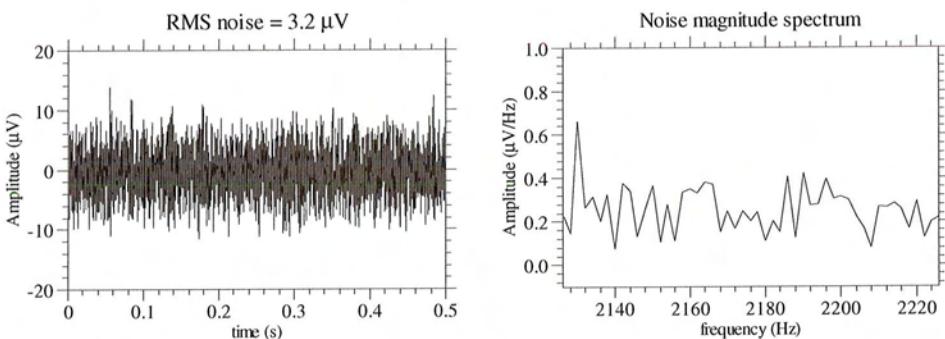


Figure 1.18. A typical output from the *MonitorNoise* macro in a region of low noise

Using the parameters shown in Figure 1.17, execute the *MonitorNoise* experiment by clicking “Run”. Observe the RMS noise value. A noise level less than 10 μV rms is acceptable, a noise level less than

5 μV rms is good and a noise level less than 3 μV rms is excellent. For noise levels above 10 μV rms, it will be challenging to acquire good quality NMR data, and MRI will be virtually impossible.

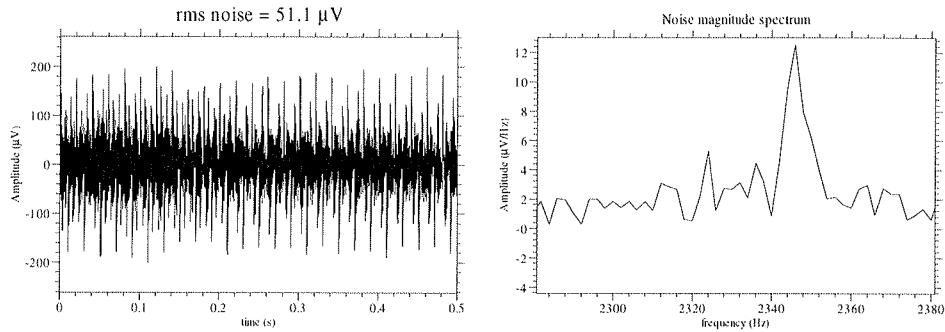


Figure 1.19 A MonitorNoise output with an unacceptable noise level.

If your noise level is not within the acceptable range you will need to find a new location for the EFNMR probe. With the *MonitorNoise* experiment running, move the probe to a new location and observe the noise level. Remember it is important to orient the probe correctly at each location using the 3D compass as shown in Figure 1.12! Repeat this procedure until a position is found where the noise level falls in the acceptable range (< 10 μV).

1.4.2. The NMR Pulse Program

Now you are ready to acquire your first NMR signal. Experiments in NMR are often referred to as pulse programs or pulse sequences because they are made up of a series of pulses: polarising pulses, RF pulses and gradient pulses. The pulse sequence that you will use to acquire your first NMR signal is the pulse and collect experiment pictured in Figure 1.20. This experiment is executed in three stages: polarisation, excitation and detection.

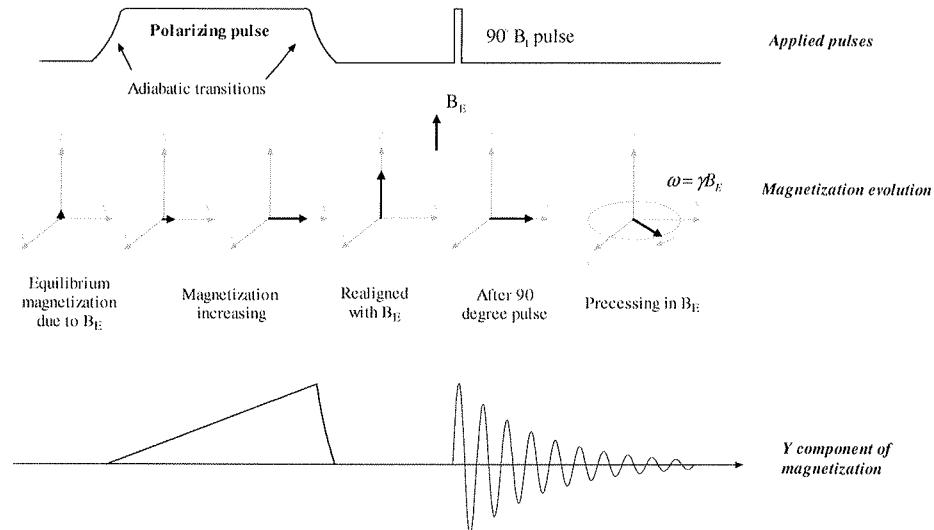


Figure 1.20 The pulse sequence diagram for the pulse and collect experiment.

In the first stage, a current (**polarisation current**) is passed through the polarisation coil to establish a magnetic field, many times stronger than the Earth's magnetic field, in the region inside the coil. This magnetic field will induce a bulk nuclear magnetisation in a sample placed within the EFNMR probe by aligning the nuclear magnetic moments of the sample spin-ensemble either parallel or anti-parallel to it. This bulk nuclear magnetisation is aligned with the applied polarisation field, B_p , as described in the background theory section (1.3.1). Typically this current is applied for about 4 or 5 seconds (**polarisation duration**) to maximise the bulk nuclear magnetisation in the sample. The current is then switched off adiabatically (in other words gradually on the timescale of the precessing magnetisation), so that the enhanced magnetisation is left lying along the Earth's field direction.

The second and third stages of the NMR experiment use the detection/excitation (B_1) coil, to excite and then collect the NMR signal. In order to excite and acquire a strong NMR signal, the LCR circuit, formed by the B_1 coil and the spectrometer, must be tuned to the Larmor frequency of the sample. This is achieved by adjusting the **capacitance** of the LCR circuit.

In the excitation stage, an RF pulse (in the ultra-low frequency (ULF) band) tuned to the Larmor frequency (**B_1 frequency**) of the sample is applied to the system. In the classical picture (see Section 1.3.1), this RF pulse rotates the bulk magnetisation vector, by a tip angle θ , into the transverse plane. The tip angle is linearly dependent on both the amplitude (**transmit (B_1) gain**) and duration (**pulse duration**) of the excitation pulse. A 90° pulse (**90 pulse duration**) fully rotates the bulk magnetisation vector into the transverse plane. A 180° pulse (**180 pulse duration**) inverts the bulk magnetisation vector. Only the transverse component of the bulk magnetisation vector is detected and therefore the 90° pulse results in the maximum signal and the 180° pulse results, ideally, in no signal. In order to optimise the signal to noise ratio (SNR) of the acquired signal, the 90° pulse is often used in most pulse and collect experiments; however, in theory any tip angle pulse can be employed. More sophisticated pulse sequences use both 90° and 180° pulses to manipulate the bulk magnetisation vector and can be used to measure the NMR properties of the sample. Such pulse sequences are beyond the scope of this experiment.

The third stage of this NMR experiment is the detection phase. The excited bulk magnetisation vector precesses in the transverse plane about the Earth's magnetic field vector at the Larmor frequency. This precessing magnetisation is in an excited state and therefore, over time, it will return to its equilibrium magnitude and orientation via a process called relaxation.

The precessing bulk magnetisation vector induces an emf in the detection coil according to Faraday's law (equation 1.10), which states that the induced emf is equal to the time rate of change of the magnetic flux.

$$\text{emf} = N \frac{d\phi}{dt} \quad [1.10]$$

After a short delay (**acquisition Delay**) this induced emf is sampled, amplified (**receive gain**) and recorded by the spectrometer as a function of time (**number data points; acquisition time**). Due to relaxation processes, this emf decays with time. The record of this decaying emf, measured in μV and sampled as a function of time, is called the free induction decay (FID). It is called "free" because it is measured in the absence of the B_1 excitation pulse and therefore in the absence of any perturbing forces.

After the experiment has been completed and an FID recorded, the *Prospa* software Fourier transforms the data from the time domain into the frequency domain. The signal in the frequency domain is called a spectrum and displays the frequency components of the signal. Ideally, the spectrum displays a single strong narrow peak at the Larmor frequency.

If desired, an experiment can be repeated several times (**number of scans**) and the individual FIDs can be averaged in order to improve the SNR (**average**). The time between the beginning of one experiment and the beginning of the next experiment is called the **repetition time**.

1.4.3. Initial Parameter Determination

In the pulse and collect dialog, Figure 1.14, there are a number of pulse sequence parameters that must be set by the user. These parameters are highlighted above in order to indicate which part of the experiment they relate to.

At the bottom of the *PulseAndCollect* dialog there is a section entitled "Output location". This section can be found on all of the experiment dialogs. It allows the user to explicitly state where the output of the experiment is to be saved. The **working directory** is the root directory where the data is to be saved. It can be chosen by clicking the "..." button and navigating to an appropriate folder on your PC. Within this directory, the macro will generate an experiment directory with a name given by the

experiment name parameter. The output data from the experiment, along with a list of all experimental parameters and any output plots, is saved to this folder.

Choose an appropriate working directory by clicking the “...” button and navigating to a folder on your PC. Choose a name for the experiment. For example you could call the experiment “firstFID”.

The pulse sequence parameters can be considered to belong in a number of different categories. There are hardware parameters which are chosen during your initial setup and then rarely changed during an experiment session. Additionally there are more experiment specific parameters which may be changed on a regular basis.

First we will choose some sensible initial values for the hardware parameters: capacitance, B_1 frequency, polarisation current, receive gain and transmit gain. The acceptable range of values for each of these parameters is listed in Table 1.2.

Table 1.2 Value Ranges for Hardware Parameters

Parameter	Acceptable Value Range
Capacitance (nF)	4.4 – 17.15
B_1 frequency (Hz)	1800 – 3000
Polarisation Current (A)	0 – 6
Receive Gain	0 – 10
Transmit Gain	0 – 10

Read the experiment description above, noting where each of these hardware parameters is mentioned. What does each parameter control? What are some of the factors which will determine how these parameters are chosen?

The capacitance of the system is controlled by the *Prospa* software through a series of relays within the spectrometer. An initial value for the capacitance can be estimated using the resonance condition for the LCR circuit. Calculate an approximate value for the Larmor frequency of the sample (equation 1-5), using an estimated value for the local Earth’s magnetic field strength. The gyromagnetic ratio for the hydrogen nucleus is $2.675 \times 10^8 \text{ T}^{-1}\text{s}^{-1}$. Using the resonance conditions for the B_1 coil (equation 1-6) and the previously determined values for your coil inductance and parasitic capacitance, calculate the necessary coil capacitance for resonance at your estimated Larmor frequency.

Enter the capacitance value (to the nearest 0.05 nF) and the B_1 resonance frequency (in Hz) into the relevant fields of the pulse and collect dialog.

The polarisation current is limited by resistive heating within the polarisation coil. This heating is potentially dangerous because it will lead to heating of both the apparatus and the sample. Power is proportional to the square of the current and therefore high currents passed through the polarisation coil for long periods of time at a high duty cycle will cause significant resistive heating. For example, 6 A applied with a duty cycle of 50% would be equivalent to a 45 W heater. We suggest that a maximum current of 6 A be applied for no more than 5 s with a duty cycle of 50%.

Enter some mid-range polarisation current, transmit and receive gain values.

Next we consider the pulse parameters. What does the polarisation duration control? Choose an initial value between 1000 and 4000 ms. What does the pulse duration control? The pulse duration can range from 0 to 50 ms. Choose an initial value of a couple ms. The parameters controlling signal acquisition include: number of data points, acquisition time, acquisition decay, number of scans and display range. The number of data points and the acquisition time determine the time spacing of the acquired FID points. Choose 8192 from the “number of points” text menu and enter an acquisition time of 1 s. For these parameters what is the time spacing between successively acquired data points?

What is the acquisition delay? Why do you think this delay might be necessary? For the first experiment, set this delay to a value of 20 ms.

Set the number of scans to 1. The number of scans and the average function will be explored later.

The frequency range of the spectrum is controlled by the sampling rate (the time between successively sampled points in the FID). For the typical sampling rates employed this is a very broad frequency range and so it is inconvenient to view the entire spectrum. The display range dictates the portion of the full spectrum which will be displayed upon completion of the measurement. The displayed portion of the spectrum will be centred about the B_1 frequency. For example, if the B_1 frequency is 2200 Hz and the display range is 200 Hz, the region of the spectrum between 2100 and 2300 Hz is displayed. (Note: no matter what display range is chosen, the full spectrum can be viewed using the zoom tools in the 1D plot window.) Choose a display range.

The final option in the *PulseAndCollect* experiment window is the “magnitude” checkbox. Following Fourier transformation, the NMR signal is a complex quantity. The phase of the spectrum can be changed such that the real part displays the absorption peak (the sample peak) and the imaginary part displays the dispersion spectrum (see the “phased” complex spectrum in Figure 1.21.) However, if the data is noisy it is difficult for the automatic phasing algorithm to correctly determine this ideal phase value and so the real part will contain both absorption and dispersion elements. This may make it difficult to distinguish the sample peak. Therefore during this initial setup stage it is useful to click the “magnitude” option and observe just the magnitude part of the NMR spectrum.

1.4.4. Acquire an FID

Place a large bottle (500 ml) containing tap water into the centre of the probe. Run the experiment by clicking the “Run” button in the pulse and collect dialog. Once the experiment has started you will hear relays clicking in the spectrometer. After a few seconds an FID and a spectrum will appear in the *Prospa* 1D plot window.

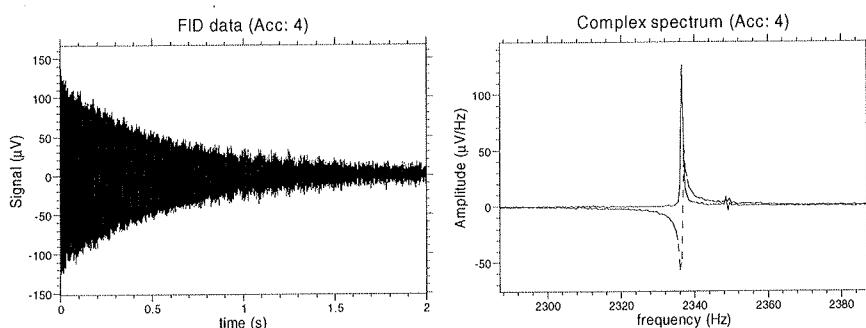


Figure 1.21 A typical good quality FID and spectrum. In the complex spectrum the solid line is the real part and the dotted line is the imaginary part.

Ideally you will observe an exponentially decaying sinusoidal signal in the FID window and a strong narrow peak in the spectrum window as is shown in Figure 1.21. However, there is a very good possibility that you will not see a strong FID signal on your first attempt.

It is sometimes difficult for a first-time user to differentiate between noise peaks and NMR peaks in a spectrum. Noise peaks tend to be very narrow, whereas the sample peak will have a finite width that is dependent on the rate of decay of the signal in the time domain (the faster the decay the broader the peak.) The best way to check if a peak is in fact an NMR signal is to acquire a signal with the sample in the probe and another with the sample removed from the probe. Compare the two frequency spectra. If the peak is a noise peak it will not change significantly when the sample is removed. However, if the peak is an NMR peak it will disappear when the experiment is repeated with the sample removed.

Note: the spectrum in the plot window is scaled automatically to the height of the largest peak and so it may not be immediately obvious how the peak height has changed between the two experiments. If this is the case you will need to measure the height of the peak in each spectrum. Click on the “allow

data display” tool  at the top of the 1D plot window. Click and hold the left mouse button within the area of the spectrum plot. Move the mouse until the crosshairs are at the top of the peak. The frequency and amplitude of the peak will be displayed in the bottom left corner of the plot window (Figure 1.22).

[x, y] = [0.238302, 5.81558e+009]

Figure 1.22 The (x,y) coordinates of the selected data point are displayed in the bottom left corner of the plot window.

It is possible that your signal may decay very quickly and so the signal will be very short in the time domain (persisting for 10s or 100s of ms) and therefore very broad in the frequency domain (10s of Hz). If this seems to be the case, reducing the acquisition time from 1s to a few hundred ms and increasing the display width may make it easier to identify the presence of a signal.

As stated previously, the position and orientation of the EFNMR probe can have a big impact on the quality of the observed FID. Operating the instrument with any ferrous objects in the near vicinity (like metal benches, screwdrivers etc...), can severely disrupt the homogeneity of the local Earth's magnetic field. This will make the decay of the FID much more rapid. A rapidly decaying FID broadens the spectral peak. Make sure that the apparatus is not on a metal bench (a wooden one is preferred) and that all ferrous materials are removed from the vicinity (from within 2-3 metres of the EFNMR probe). It also helps to have the instrument as far from the ceiling, floor and walls as possible.

If a signal is not observed on your first attempt, it is a good idea to check that your choice of parameters is not the problem. It is possible that the local value for the Earth's magnetic field is different from your estimate and so the chosen Larmor frequency may be incorrect. Acquire signals for a range of B_1 frequencies from 1800 Hz to 2500 Hz. For each frequency value, use an appropriate capacitance value. The appropriate capacitance value can be read from the output plot of the AnalyseCoil macro or else can be calculated from the inductance and parasitic capacitance of the coil, as measured by that macro. The range of frequencies excited by the coil is quite large and so a series of measurements with B_1 frequencies 100 Hz apart should be sufficient to ascertain if the problem is your choice of B_1 frequency.

The B_1 pulse duration and transmit gain determine the tip angle of the RF pulse. The largest signal is acquired from a 90° pulse. It is possible that your choice of B_1 pulse parameters is resulting in a pulse with a tip angle very different from 90°. Use a 2 ms pulse with a transmit gain of 2.

If a signal is still not observed, it is worthwhile moving the apparatus to a new position. Remember to correctly orient the probe and to check the noise level at each potential location. If finding a signal proves challenging, please refer to the user manual for instructions on how to use a more advanced experiment to find a signal. The goal is to acquire a signal that persists for at least 100 ms. Once such a signal is located, it can be optimised using the following procedure.

1.4.5. Optimising the NMR parameters

After finding a suitable location and minimising the noise as much as possible, the next step to acquiring a good quality FID is to tune the probe, i.e. adjust the capacitance of the resonant circuit so that it resonates at the Larmor frequency of the sample.

In order to tune the probe to the Larmor frequency of the sample we need a means of observing the actual resonance of the tuned circuit. After transmission of the B_1 excitation pulse, the B_1 coil resonates. This resonance decays in time and is called the coil ring-down. In order to acquire the NMR signal with this coil, without observing the coil ring-down, a delay of about 25 ms (acquisition delay) is inserted between the application of the excitation pulse and the acquisition of the first data point. If this delay is shortened, then the coil ring-down will be observed. The Fourier transform of the ring-down signal will appear as a sinc-like function in the spectrum. The centre of this sinc function is the resonance frequency of the probe.

Reduce the Acquisition delay parameter to 2 ms and set the display range to a large value such as 500 Hz. Acquire an FID by clicking “Run” in the *PulseAndCollect* window.

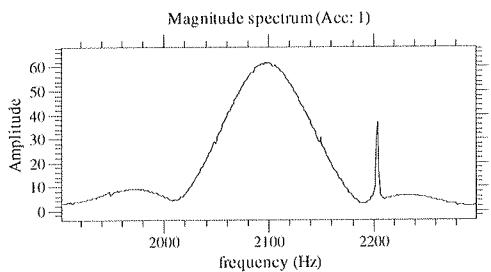


Figure 1.23. An example of the spectrum from a large water sample acquired by an incorrectly-tuned coil with an acquisition delay of 2 ms.

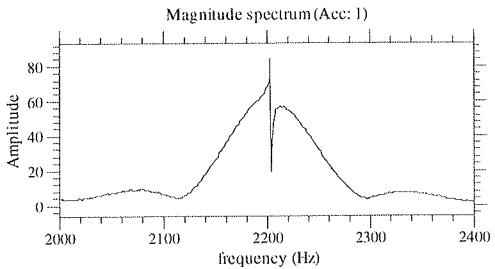


Figure 1.24. An example of the spectrum from a large water sample acquired by a correctly tuned coil using an acquisition delay of 2 ms.

In the example in Figure 1.23 the broad sinc-type peak is the coil response to the short B_1 impulse, while the narrow spike is the NMR signal. If the sample peak is very strong, it is possible that the sinc function, caused by the ring down of the coil, will be difficult to distinguish. If this is the case, reduce the polarisation duration. This will reduce the intensity of the sample peak.

The capacitance of the system is controlled by the *Prospa* software and is entered by the user into a text field in many of the experiment dialogs. To tune the coil, adjust the capacitance value and repeat the experiment until the centre of the sinc function coincides with the centre of the resonance peak (Figure 1.24). What is the form of the relationship between the capacitance and the resonant frequency of the coil? That is, if the coil resonance is too low, should you increase or decrease the capacitance to increase the resonant frequency?

Once the coil has been tuned, return the acquisition delay to 25 ms and acquire an FID. Has the signal level improved?

The next step in optimising the FID is to measure the frequency of the resonance peak in the spectrum and enter this as the B_1 frequency in the Pulse and Collect dialog. The exact Larmor frequency of the sample can be determined from the sample peak in the NMR spectrum that you acquired after tuning the coil.



Click on the “allow data display” tool at the top of the 1D plot window. Click and hold the left mouse button within the area of the spectrum plot. Move the mouse until the crosshairs are at the top of the peak. The frequency and amplitude of the peak will be displayed in the bottom left corner of the plot window. Record the frequency value, since this is your Larmor frequency. Enter this value as your B_1 frequency parameter in the Pulse and Collect window.

Calculate the local Earth’s magnetic field strength from your measured sample resonance (Larmor) frequency. How does it compare with your initial estimate? How does it compare with the local value using the website: <http://www.ngdc.noaa.gov/seg/geomag/jsp/IGRF.jsp>?

1.5. Further Questions

1. Why is the homogeneity of the local Earth’s magnetic field important? How does magnetic field inhomogeneity affect the FID and spectrum?

1.6. Appendix for the Instructor

The goal of this experiment is to setup the Earth’s field NMR apparatus and to acquire an NMR signal from a water sample. This experiment introduces the concept of the NMR apparatus as a tuned LCR circuit used for excitation and detection of the signal. In addition, the basic principles of a simple, single RF pulse NMR experiment are introduced. It is left to subsequent experiments to explore the NMR parameters and their effect on the quality of the signal.

The student is asked to determine the Larmor frequency based on an estimation of the local Earth's magnetic field. The gyromagnetic ratio for ^1H is $2.67 \times 10^8 \text{ s}^{-1}\text{T}^{-1}$. Therefore a reasonable estimate of the Larmor frequency for a region where $B_E = 54 \mu\text{T}$ is given by:

$$\omega = \gamma B_E = (2.675 \times 10^8 \text{ s}^{-1}\text{T}^{-1}) \times (54 \mu\text{T}) = 14445 \text{ s}^{-1} \rightarrow 2299 \text{ Hz}$$

An estimate of your local Earth's magnetic field value can be obtained, using your local longitude and latitude, from the following website:

<http://nssdc.gsfc.nasa.gov/space/model/models/igrf.html>.

Alternatively a Hall probe can be used to measure an approximate value for the local field in the chosen location. It is anticipated that indoors the actual value of B_E found by the student may vary significantly from the value given by the above web-site. The relatively weak magnitude of the Earth's field means that it will be easily disrupted by structural materials in the building or nearby objects.

A determination of the required capacitance value can be obtained using the resonance expression for the system (an LCR circuit) and the Larmor equation. The inductance of the coil is determined by the AnalyseCoil experiment. Therefore the required capacitance will be:

$$\omega = \frac{1}{\sqrt{LC}} \rightarrow C = \frac{1}{\omega^2 L} = \frac{1}{(2\pi \times 2299 \text{ Hz})^2 \times 0.50 \text{ H}} = 9.6 \text{ nF}$$

A general note on using the *Prospa* software package to save data. After each experiment is run, the output data, experiment parameters and any plots, will be saved into a folder in the working directory, named according to the experiment specified in the experiment dialog window. Therefore it is possible to review previous results by loading the corresponding plot file. (Use the "Load" command under the "File" menu in the plot window). Plot files have the extension *.pt1 and *.pt2 for 1D and 2D plots respectively. Another useful feature of *Prospa* is that plots can be copied and pasted from the plot window into another application, such as a word processing package. It may be useful for students, for the purposes of experiment documentation, to copy and paste their plots, at each step of the experiment, into a word processor. This can be achieved by pressing "CTRL-C" when the plot window is selected. This copies the plot to the clipboard. The plot can then be pasted by pressing "CTRL-V" when in the other application. This would be especially useful in the case of the AnalyseCoil macro.

Locating a position in which the EFNMR probe produces a sufficiently low noise level can be a time consuming endeavour. It is recommended that the instructor locates a suitable position prior to the student performing this experiment. Additional guidelines for finding a good signal and suggestions for suitable initial parameters can be found in the Terranova-MRI User Manual.

If a "good" signal still can't be found, check the parameters that are being used by the student. Suggested values are as follows: polarising current = 6 A, polarising duration = 4 s, B_1 frequency = 2200 Hz, capacitance = 10 nF, transmit gain = 2, pulse duration = 2 ms, receive gain = 7, number of data points = 8192, acquisition delay = 25 ms, acquisition time = 1 s, repetition time = 8 s, display range = 200 Hz, number of scans = 1 and magnitude option selected. As suggested in the experiment, the one incorrectly chosen parameter which will most often result in no observed signal is the B_1 frequency. Therefore if no signal is observed, try B_1 frequency values which range from 1800 to 2500 Hz, with a step size of 100 Hz. }

To identify an NMR signal, acquire data with the sample in the probe and one with the sample removed from the probe. If the peak is only present in the spectrum acquired with the sample in the probe then it is a sample peak not a noise spike. An example of this is shown in Figure 1.25 and Figure 1.26.

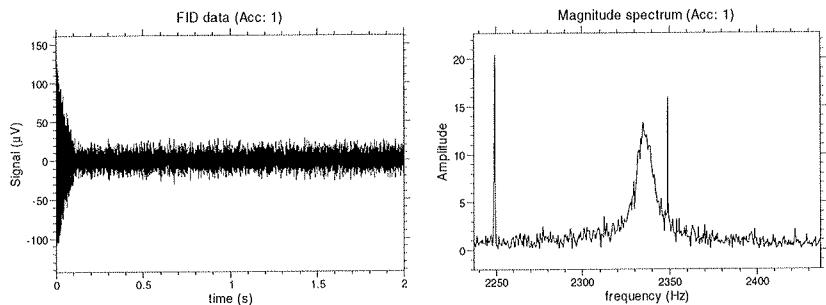


Figure 1.25 A signal acquired with the sample in the probe. Which is the sample peak?

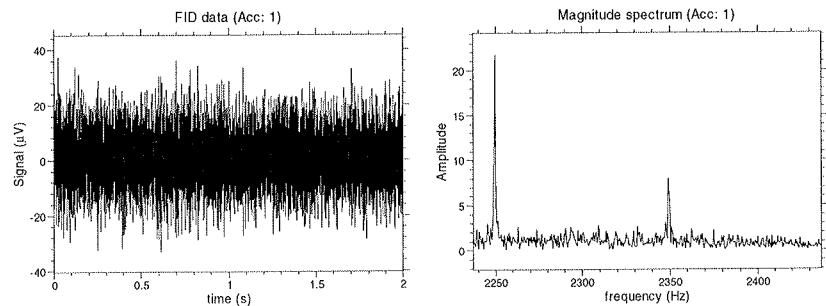


Figure 1.26 A signal acquired with the same parameters but with the sample removed from the probe. The remaining peaks are noise. The missing peak is the sample peak.

By the end experiment 1 the student should have been able to acquire a signal that is observable using the *PulseAndCollect* pulse sequence, i.e. one which persists in the time domain for approximately 100 ms or longer. Figure 1.27 demonstrates the minimum signal quality required in order to successfully proceed with Experiment 2.

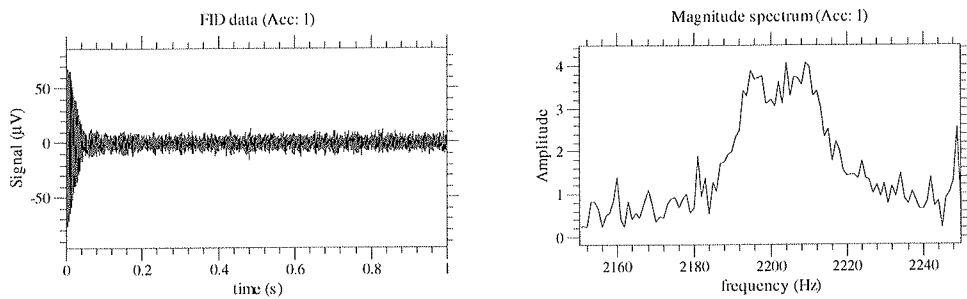


Figure 1.27 An example of a FID signal and spectrum prior to optimisation.

2. Shimming and Parameter Optimisation

2.1. Objective

The object of this experiment is to improve the quality of the NMR signal found in Experiment 1 through the use of shimming and parameter optimisation. This experiment introduces a protocol for optimising the NMR signal that will be used prior to all further experiments in order to ensure the highest quality results possible.

2.2. Apparatus

The Terranova-MRI Earth's field MRI apparatus, consisting of a spectrometer and a three-coil probe, will be used for this experiment. The sample will be a large (500 ml) bottle of tap water. All experiments will be run from the *Prospa* software package.

2.3. Background Theory

2.3.1. Magnetic field homogeneity and shimming

A very important consideration in the acquisition of a high quality NMR signal is the homogeneity of the underlying static magnetic field. In the case of EFNMR this static field is the Earth's magnetic field, B_E . This field is naturally highly homogeneous. However, the field is so weak ($\sim 50 \mu\text{T}$) that its homogeneity is easily disrupted by the proximity of ferrous objects or other sources of small magnetic fields.

The NMR signal arises from the phase coherence of an ensemble of precessing nuclear spins. The exponential decay of the signal is a consequence of the loss of phase coherence between the spins. One source of phase coherence loss is the relaxation process known as spin-spin relaxation (characterised by the T_2 relaxation time constant). Spin-spin relaxation is caused by the magnetic dipole coupling between two neighbouring spins. In the presence of a completely homogeneous field, spin-spin relaxation is the dominant source of phase coherence loss. However, in practice, the magnetic field is not entirely homogeneous and therefore the loss of phase coherence is not solely due to spin-spin relaxation but is rather a combined effect of spin-spin relaxation and magnetic field inhomogeneity. Local magnetic field inhomogeneities introduce a range of Larmor frequencies across the sample. Each spin will precess at the Larmor frequency associated with its position. The overall phase coherence of the ensemble depends on all nuclei precessing at the same frequency. Therefore this dispersion of frequencies will result in a loss of phase coherence and hence signal decay.

To describe the combined relaxation effects we define an effective spin-spin relaxation time constant, T_2^* , where γ is the gyromagnetic ratio of the observed nucleus and ΔB_0 is the magnetic field inhomogeneity.

$$\frac{1}{T_2^*} = \frac{1}{T_2} + \gamma \Delta B_0 \quad [2.1]$$

The observed signal as a function of time in a region with some magnetic field inhomogeneity is given by:

$$S(t) = S_0 \exp(-t/T_2^*) \quad [2.2]$$

where S_0 is the initial signal magnitude at $t = 0$. Therefore if T_2^* is very short, due to significant inhomogeneity, ΔB_0 , the signal will be greatly reduced and will decay very quickly in the time domain. This will also result in a very broad peak in the frequency domain.

B_0 field homogeneity can be greatly improved using a process called *Shimming*. Shimming is the process of iteratively applying weak position dependent magnetic fields across the sample until the applied fields cancel the underlying inhomogeneity of the static field. This is typically achieved by passing small currents through a collection of coils designed to generate magnetic fields of specific

2-2 Introductory Experiments

geometries such as a magnetic field that changes linearly along one axis. This is termed a field gradient. More complex shim coils will generate magnetic fields that simultaneously vary in more than one direction or else vary quadratically or cubically in a single direction.

The Terranova-MRI apparatus employs three shim coils, which correspond to field gradients along the x, y and z axes. These are called first order shims and have been found to be very effective for shimming in the Earth's magnetic field.

2.3.2. Pre-polarisation

Conventional high field NMR instruments require an extremely uniform magnetic field for the polarisation of the nuclear spins and for the detection of the resultant magnetisation. It is difficult and expensive to make a magnetic field of the required homogeneity (much less than 1 ppm (0.0001%) in some cases) and so sample sizes are usually quite small. However because the polarisation and detection fields are large the resulting signal to noise ratio (SNR) can also be large. Earth's field nuclear magnetic resonance (EFNMR) works because detection takes place in the highly uniform Earth's magnetic field. This means that large samples can be used – partially compensating for the loss of sensitivity due to the very low detection field. To improve the initial magnetisation, a relatively crude electromagnet with a field about 350 times larger than the Earth's field is used to polarise the sample.

The polarisation coil is used to generate a magnetic field many times stronger than the Earth's field for the purpose of increasing the bulk nuclear magnetisation of the sample. The bulk magnetisation is directly proportional to the magnitude of this polarisation field, through the following equation:

$$M = \frac{N\gamma^2\hbar^2 I(I+1)B_p}{3kT} \quad [2.3]$$

In the Terranova EFNMR system a maximum current of 6.0 A may be applied to the polarising coil, producing an 18 mT field (B_p) directed along the horizontal axis of the coil (for comparison the Earth's field is about 54 µT). Typically this current is applied for about 4 or 5 seconds to maximise the bulk nuclear magnetisation in the sample. The current is then switched off adiabatically (in other words gradually on the timescale of the precessing magnetisation), so that the enhanced magnetisation is left lying along the Earth's field direction. (Ideally orthogonal to the long axis of the apparatus.)

2.4. Procedure

2.4.1. Getting Started

In order to proceed with this experiment, you must first obtain an NMR signal from the large bottle of water (see Experiment 1). This signal must be observable using the pulse and collect experiment and persist for about 100 ms. Open the pulse and collect experiment and follow the instructions in Experiment 1 to locate such a signal.

2.4.2. Shimming

Once you have found a signal, click the "Shims" button in the pulse and collect dialog. The dialog in Figure 2.1 will appear. This dialog window controls the values of the shims used for the pulse and collect experiment. There are two sets of shim values visible in this window: "Current" and "Saved". Your shim values, once an optimum set have been determined, will be used by all experiments and so are stored in a common location. These are the "Saved" values. The common shim values are saved in your preferences folder, which can typically be found in the following location, where "username" is your Windows username:

C:\Documents and Settings\username\Application Data\Prospa\Preferences V2.0\Other Macros.

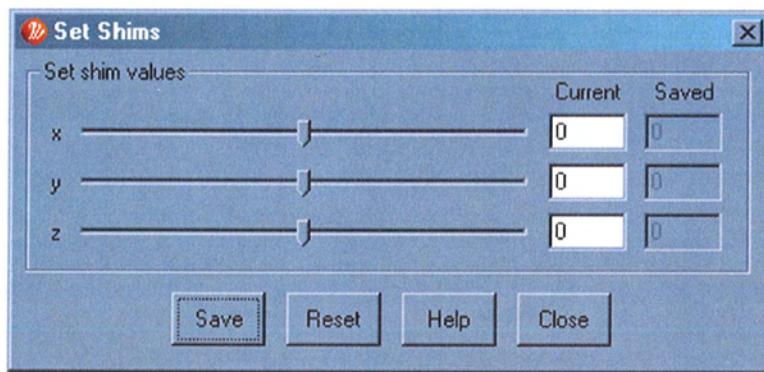


Figure 2.1 The set shims dialog window.

The “Current” values are the values used by the current experiment. These values can be saved to the common file by clicking “Save”. Alternatively, the saved values can be loaded at any time by clicking “Reset”.

Leaving the shim window open, acquire an FID with all shim values set to zero. Record the signal height both in the time domain and in the frequency domain. Estimate the linewidth in the frequency domain. [Note: initially it will be easier to observe the peak if the magnitude option is selected.] Change one of the shim values by several millamps and repeat the pulse and collect experiment. Does the signal improve? What is the peak height in the time and the frequency domain? What is the linewidth?

Continue to increase and decrease the shim values, running the experiment after each shim value change. Can you find some values that improve the signal? What do you think “improving” means in terms of the signal shape? What would be a good parameter to use to identify a “good” shim?

In order to find the ideal shim values, it is important to change the shim values in a systematic way. For example if an increase in the shim value on one axis improves the signal, continue to increase this value until the signal stops improving.

If the signal does not decay completely by the end of the acquisition time, i.e. if there is still significant signal at the end of the FID as shown in the time domain plot, increase your acquisition time to 2 or 3 seconds.

Obtain the best signal you can by manually changing the shim values. What is the peak height and linewidth of the sample peak in the spectrum? At this point you should remove the magnitude option and observe the complex spectrum because the linewidth of the real part of a phased spectrum is the true linewidth.

Make a note of your shim values. Save these values by clicking “Save” in the set shims window. Close the shim window and the pulse and collect window.

2.4.3. Auto-shimming

Having spent some time trying to achieve a good set of shim values, you will probably now appreciate the amount of time and effort that can be expended in the act of shimming. Fortunately, in some cases, it is possible to automate this procedure and thus reduce the time and effort involved in achieving a high quality signal.

From under the EFNMR menu choose “AutoShim”. This experiment performs shimming in an automated manner. The search for the ideal shim values is performed using a modified bisection approach.

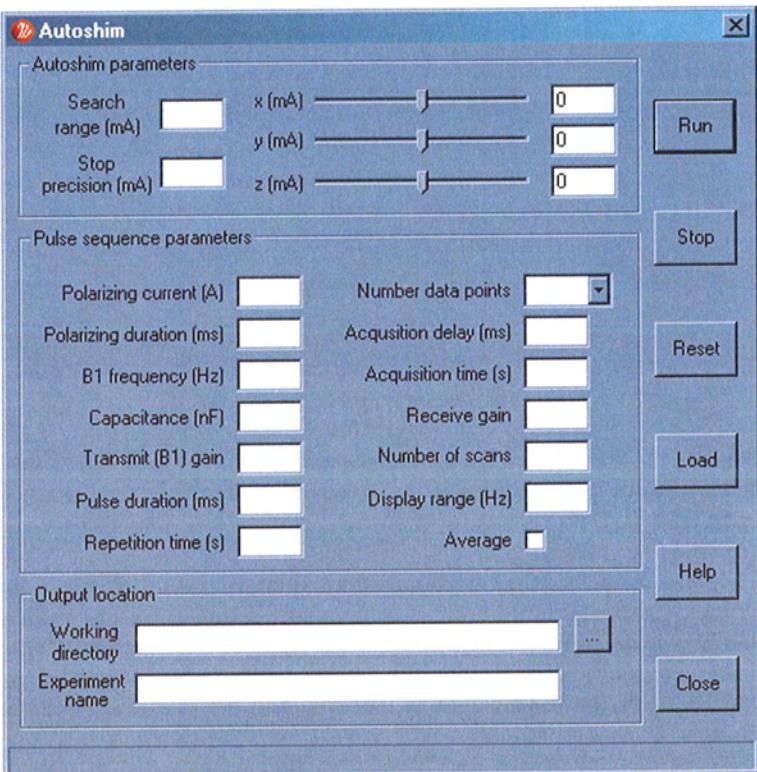


Figure 2.2 AutoShim dialog window

The inputs to this experiment are the initial shim values, a search range and a stop precision. The search range determines the range of values to try, i.e. from the initial value plus half the search range to the initial value minus half the search range. The stop precision tells the program when to stop the iteration, i.e. when the procedure search step size is reduced to equal to the stop precision.

This experiment uses the pulse and collect sequence. Enter the same values into the pulse sequence parameters as you used in the previous section. Choose an appropriate working directory and experiment name. Set the initial shim values to zero and the search range to something relatively large, such as 20 mA. Use a stop precision of 0.1 mA. Start the experiment by clicking “Run”.

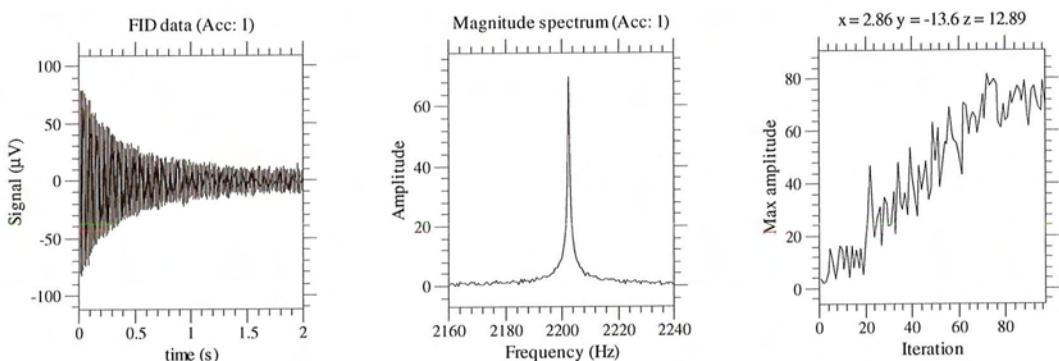


Figure 2.3 At the end of the shimming process. The plot on the right shows the history of the shimming process, the amplitude represents the height of the peak in the central plot.

A sample output of the AutoShim experiment is shown in Figure 2.3. The FID and spectrum for each step in the AutoShim process are shown in the left-most two plots. These plots are updated for each step in the iterative process. The plot on the right shows the progress of the shim. The amplitude of this plot represents the peak height in the frequency domain and is used as a measure of shim quality.

The AutoShim routine will take a while to complete, typically 10 to 20 minutes depending on the initial parameters and the stop precision. How do the shim values found by the autoshim routine compare to those you found in the previous section? How does the quality of the FID and spectrum compare?

The shimming parameters are very sensitive to changes in the environment. Therefore if the Terranova-MRI probe is moved in any way or if any large metal objects in the vicinity of the probe are moved (i.e. metal chairs) then the shimming process must be repeated. It is therefore useful, throughout an experimental session, to periodically check the shim by acquiring an FID and spectrum using the pulse and collect experiment.

2.4.4. Parameter optimisation

In this section you will explore the effect of the various NMR parameters on the FID and spectrum. Select the PulseAndCollect experiment from the EFNMR menu. Name your experiment: "Explore_FID". Your previous parameters will appear in the dialog window.

The "receive-gain" parameter controls the amount of amplification applied to the acquired signal. Increase the receive gain and observe the peak voltage on the FID. How does it change? Does the signal-to-noise ratio (SNR) of the FID change? Why or why not? (The SNR can be estimated by dividing the peak voltage at the beginning of the FID by the peak voltage at the end of the FID, once the signal has decayed and there is only noise remaining.)

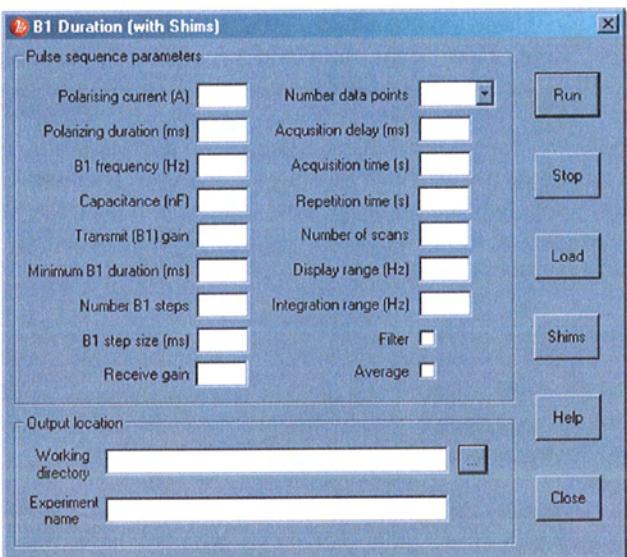
Now click the "Average" box and increase the number of scans to 9 or 16. The average function repeats the pulse and collect experiment for the number of scans and then averages the results. What happens to the FID peak voltage? The SNR? Try different numbers of scans. Explain your observations.

Next we will consider the B_1 excitation pulse. Change the length of the B_1 pulse. How does the FID and spectrum change? Change the transmit gain. How does the FID and spectrum change? These two parameters control the tip angle of the excitation pulse. Consider Figure 1.4. Recall that only the component of the bulk magnetisation which precesses in the transverse plane is detected; how do you expect the signal magnitude to change with tip angle? What would a 90° pulse do? A 180° pulse?

The tip angle can be calibrated by determining the duration required for a 90° and 180° pulse. Repeating the pulse and collect experiment over an array of durations and observing the change in signal magnitude can achieve this. Choose the "B1_Duration" experiment under the EFNMR menu. The dialog in Figure 2.4 will appear.

From this dialog you can run a series of pulse and collect experiments with an array of B_1 pulse lengths. The signal intensity produced by this macro is obtained by integrating the spectrum over the range of frequency values that define the resonance peak. By inspecting a single spectrum, obtained with a single pulse and collect experiment, choose the integration width. The remaining parameters are chosen as before and so will have been set by your previous experiments.

Based on your answer above regarding the relationship between tip angle and signal intensity, what do you expect as an output from this experiment? Run the experiment. The output plot displays the integral of the sample peak for each value of the pulse duration. How would you determine the 180° pulse from this plot? The 90° pulse? It may be necessary to re-run the experiment with a different range of pulse lengths to find the most accurate 90° and 180° pulse lengths. Make a note of the 90° and 180° pulse length values.

Figure 2.4 B_1 duration experiment dialog

Open the PulseAndCollect experiment. The final parameters to be examined are the number of data points and the acquisition time. These parameters define the dwell time, Δt , between adjacently sampled points. The dwell time in turn determines the range of frequencies, or spectral width, of the spectrum. The maximum and minimum frequencies observed in the spectrum are equal to

$$\omega_{\pm} = \pm \frac{1}{2\Delta t} \quad [2-4]$$

where

$$\Delta t = \frac{\text{acquisitionTime}}{\text{numberPts}} \quad [2-5]$$

Acquire an FID. Observe the time axis of the FID. How would you choose an appropriate acquisition time? It may be useful to increase and decrease the acquisition time to get a feel for what this parameter does.

Once you have chosen an appropriate acquisition time, acquire an FID with 16384 points. Observe the full spectrum (using the display all data button). What is the maximum frequency observed? Now reduce the number of acquired points. Given the expressions above for the frequency range and dwell time, Δt , what would you expect the new range of frequencies in the spectrum to be? Observe the change in the spectral width. Is it as expected?

What happens when the maximum frequency (ω_+) is less than the resonance frequency? Can you explain this effect?

2.4.5. Pre-polarising pulse duration

The last optimisation step is to determine the ideal polarisation pulse length for the water sample.

The purpose of the polarising pulse is to establish an enhanced bulk nuclear magnetisation in the sample that is oriented in the direction of the Earth's magnetic field. The magnitude of this nuclear magnetisation is dependent on the magnitude, B_p , of the polarisation field and the duration, τ_p , of the polarisation pulse. Following the polarisation of the sample, the net magnetisation vector is rotated into the transverse plane by an RF excitation pulse, where it precesses about the Earth's magnetic field direction and produces an induced emf in the detection coil. The magnitude of this induced emf (the NMR signal) is directly proportional to the magnitude of the net magnetisation in the transverse plane. This is in turn dependent on the net magnetisation established in the sample by the polarisation pulse. Therefore the NMR signal intensity is dependent on both τ_p and B_p .

Open the PulseAndCollect experiment from under the EFNMR menu. Choose a polarising current value of 6 A. Obtain a number of FIDs and spectra with different polarisation durations. Do not exceed a duration of 6 s. Record the peak heights displayed in the CLI window for each polarisation duration. How does the peak height change with τ_p ? What would be a reasonable choice for τ_p ? Keep in mind that shorter pulses are preferred because of heating concerns and also the total experiment time. The optimal τ_p is the time for which a small decrease in τ_p results in a significant signal decrease whereas a small increase in τ_p does not result in a significant signal increase.

2.5. Further Questions

1. Is it possible to have a 270°, 360° etc... RF pulse? What would be the effect of these pulses?
2. What phenomenon causes the observed relationship between the NMR signal amplitude and τ_p ?
3. How is prepolarisation achieved in hyperpolarised gas magnetic resonance imaging applications?
4. Why separate the polarisation and detection phases of an NMR experiment? What are some of the difficulties associated with encoding the NMR signal at high field strengths?
5. In a conventional NMR experiment where the polarisation and detection steps are both carried out under the influence of the same magnetic field, B_0 , the relationship between the NMR signal magnitude and B_0 is not linear. Why?

2.6. Appendix for the Instructor

The goal of this experiment is to optimise the EFNMR signal and to teach the student about the various EFNMR parameters and how they affect the EFNMR signal.

The first part of the experiment involves improving the homogeneity through shimming. A good signal persists for a long time, hundreds of ms or even seconds and has a narrow, large peak in the frequency domain. Good parameters for determining the shim quality are the height and line-width of the peak. The line-width is difficult to accurately measure with an automated procedure and therefore the autoshim method uses the peak height to indicate the shim quality.

The student should be able to improve the signal to such an extent that it persists for 100s of ms in the time domain and achieve a linewidth of less than a couple Hz in the frequency domain.

The next section of the experiment allows the student to explore the acquisition parameters and see their effect on the NMR signal.

The receive gain parameter amplifies both the signal and the noise and so does not improve the SNR (although extreme values will degrade the SNR or result in clipping).

Averaging FIDs increases the SNR by coherently adding the signal and incoherently adding the random noise. The SNR improvement should equal the square root of the number of accumulated scans.

A tip angle of 90° yields the maximum signal. Ideally a tip angle of 180° yields no signal although in practice it is the minimum signal not a complete null because of B_1 inhomogeneity effects. In the plot of signal amplitude versus B_1 pulse duration the maximum gives the 90° pulse duration and the minimum the 180°. For a transmit gain of 2.5 typical values are 1.5 ms for the 90° pulse and 2.7 ms for the 180° pulse.

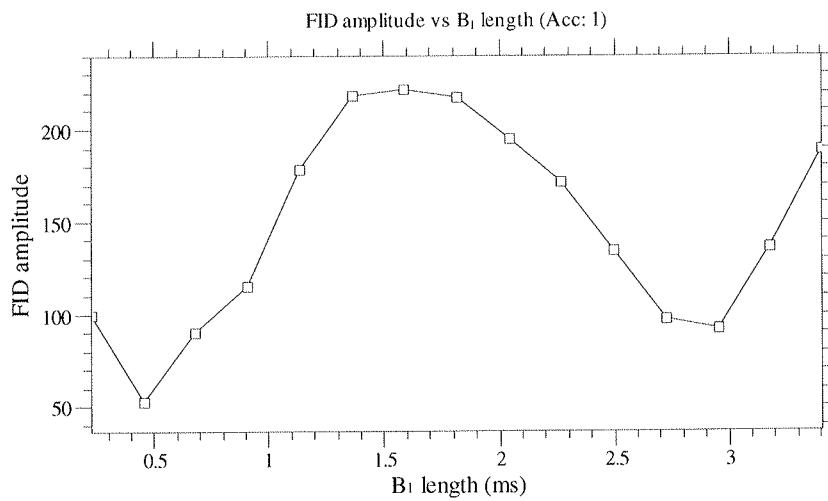


Figure 2.5. A sample output of the B_1 Duration experiment.

An appropriate acquisition time is a duration during which the signal decays to just below the noise within the window but where most of the time window contains signal. For example, if a signal persists for 800 ms, 1 s is an appropriate acquisition time.

If the Larmor frequency is outside the spectral width of the measurement, the frequency will not be measured properly because it violates Nyquist's theorem, which states that the sampling frequency must be twice the maximum detected frequency. If this theorem is violated there will be aliasing or fold-over, i.e. the sample peak will appear at an incorrect frequency in the window.

The final section deals with the pre-polarising pulse and how the signal magnitude is dependent on the polarising pulse duration. Due to resistive heating concerns it is important that the student not employ polarisation currents of more than 6 A and polarisation durations of more than 6 s. If the polarising coil heats up significantly, stop the experiment and wait a few minutes for it to cool down.

The student should observe that the relationship between signal and polarising pulse duration is not linear. The observed relationship; $1 - \exp(-\tau_p/T_1)$, is due to T_1 relaxation, which will be covered in a subsequent experiment. For a tap water sample a τ_p of 4 s is a reasonable compromise between coil heating and signal magnitude.



Terranova-MRI

Earth's field MRI & NMR teaching system

Earth's Field NMR Experiments

3. Spin-Lattice (Longitudinal) Relaxation: T_1 ✓

3.1. Objective

The object of this experiment is to demonstrate the concept of spin-lattice relaxation and to measure the spin-lattice relaxation time constant (T_1) of a water sample. Two methods will be used to measure this relaxation time, including measurements in both the polarisation field and in the Earth's magnetic field.

3.2. Apparatus

This experiment will use the Terranova-MRI EFNMR instrument, consisting of a three-coil probe, a spectrometer and a controlling PC. The experiments will be run from the *Prospa* software package. The samples used include a large (~500 ml) bottle of tap water and a large bottle of cooking oil.

3.3. Background Theory

3.3.1. Spin-Lattice Relaxation

The process of spin-lattice relaxation involves the transfer of energy between the spin system and the surrounding thermal reservoir, called the lattice. The relaxation time constant, T_1 , is a measure of the efficiency of this energy transfer and describes how quickly the equilibrium magnetisation is established. Spin-lattice relaxation is described by the phenomenological equation:

$$\frac{dM_z}{dt} = \frac{M_0 - M_z}{T_1} \quad [3.1]$$

where z is defined as a unit vector parallel to the dominant static magnetic field; z defines the longitudinal axis in the laboratory frame of reference. During an EFNMR experiment, the dominant magnetic field is either the polarisation field, B_p , during the polarisation pulse or the Earth's magnetic field, B_E , during signal excitation and detection. M_0 is the equilibrium magnetisation in the static magnetic field. Solving equation 3.1, the time dependence of the longitudinal magnetisation, $M_z(t)$, is found to be:

$$M_z(t) = M_z(0) \exp(-t/T_1) + M_0 [1 - \exp(-t/T_1)] \quad [3.2]$$

The T_1 time constant is dependent on molecular dynamics and can also depend on the strength of the static magnetic field. Therefore it is interesting in the context of the Earth's field NMR experiment to consider T_1 in the polarising field and in the local Earth's field.

In the polarising field the equilibrium magnetisation is proportional to B_p and will be denoted by M_p . At $t = 0$, before the application of the polarising pulse, the longitudinal magnetisation, M_z , is proportional to B_E and is therefore essentially zero. Accordingly, the spin-lattice relaxation magnetisation evolution equation during the polarising pulse can be written as

$$M_z(t) = M_p [1 - \exp(-t/T_1)] \quad [3.3]$$

Following the polarisation pulse, the evolution of the longitudinal magnetisation will be characterised by T_1 in the Earth's field. In this case, the initial magnetisation, $M_z(0)$, will be equal to M_p and the equilibrium magnetisation, M_0 , will be essentially zero. Therefore the time dependence of the longitudinal magnetisation in the Earth's field is given by

$$M_z(t) = M_p \exp(-t/T_1) \quad [3.4]$$

3.4. Procedure

3.4.1. Getting Started

Run through the setup procedure from Experiments 1 and 2 to acquire a good quality FID of a large tap water sample. This process should include:

- Shimming.
- Setting the B_1 frequency to the Larmor frequency of the sample.
- Tuning the probe.
- Determining the length of the 90° and 180° pulses.

3.4.2. Measuring T_1 in the Polarising Field

The purpose of the polarising pulse at the beginning of a pulse and collect experiment is to establish an enhanced nuclear magnetisation in the sample. This polarisation of the sample is established via the process of spin-lattice relaxation, which is characterised by the T_1 time constant. The time dependence of the signal magnitude during the polarisation pulse can be defined by the following equation:

$$S(\tau_p) = S_p [1 - \exp(-\tau_p/T_1)] \quad [3.5]$$

where S_p is the equilibrium signal magnitude in the polarising field in the limit as the polarisation time, τ_p , goes to infinity.

The T_1 of any given sample in the polarising field can be measured using the polarise, pulse and collect experiment (Figure 3.1) with an array of different polarising pulse durations.

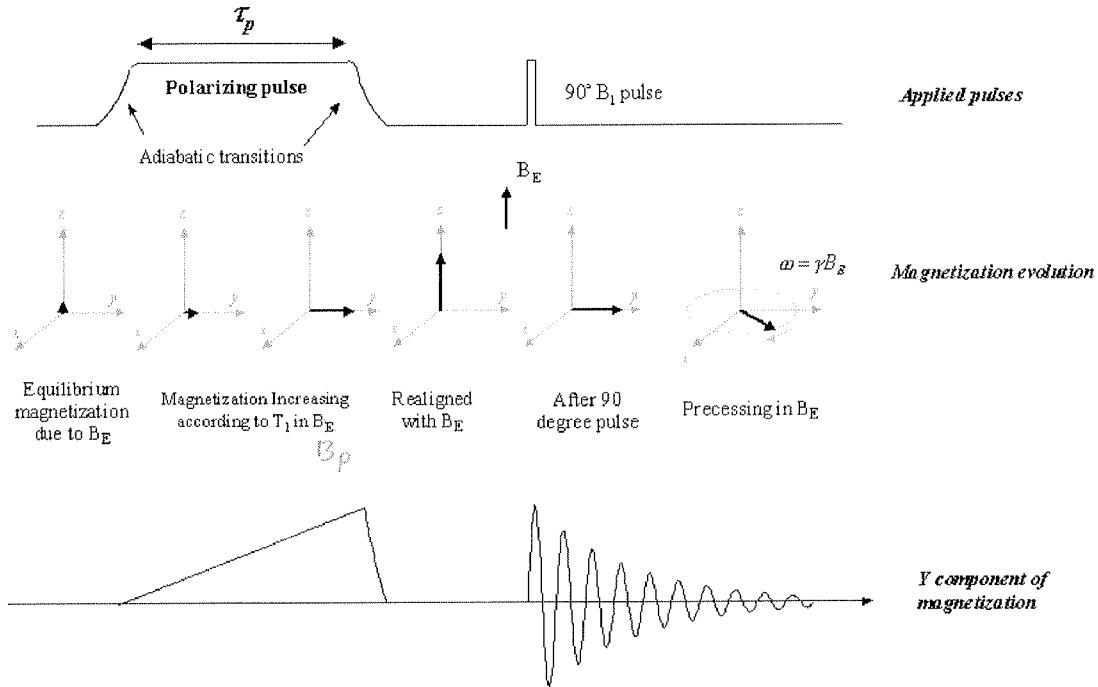


Figure 3.1 The T1Bp pulse sequence.

Open the “T1Bp” experiment dialog from under the EFNMR window. The dialog box in Figure 3.2 will appear.

Enter the parameters: polarising current, B_1 frequency, capacitance, transmit gain, receive gain, 90° acquisition delay, number of data points, acquisition time, repetition time and display range according to your setup values. Enter the value for the 90° B_1 pulse length obtained during the instrument setup. It is often beneficial to use 4 (or more) scans in order to obtain good S/N in this experiment. Remember to check the “average” option if using more than one scan. Choose an integration width

that includes the entire breadth of the peak. Choose an appropriate experiment name and working directory.

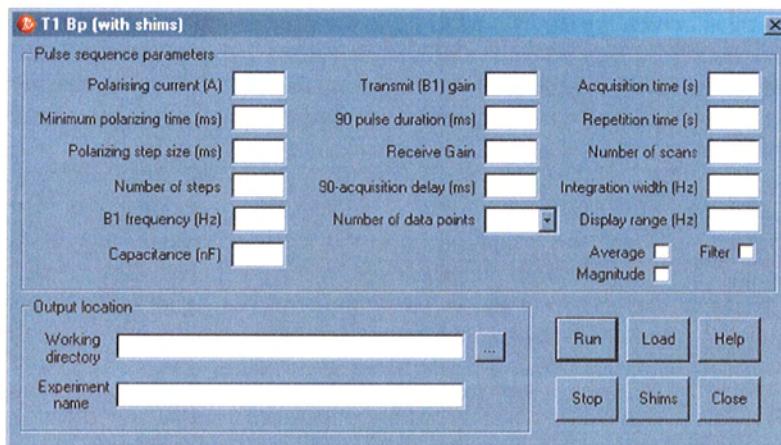


Figure 3.2. T_1 Measurement Dialog Box (in B_p)

The remaining parameters to be chosen include the minimum polarising time, the polarising step size and the number of steps. These parameters determine the array of polarisation pulse durations to be used in this experiment. Use an array of at least 9 or 10 steps starting at around 500 ms. The upper limit on the pulse duration would ideally be a few multiples of the anticipated T_1 value. However, there are other concerns that must be considered when choosing the strength and duration of the polarisation pulse.

There is a danger of resistive heating in the polarisation coil if large currents are driven through with high duty cycles. This heating is potentially dangerous because it can lead to heating of both the apparatus and the sample. Power is proportional to the square of the current, $P = I^2R$, and therefore high currents passed through the polarisation coil for long time periods at a high duty cycle will cause significant resistive heating. For example, a current of 6 A applied for 4 s with a duty cycle of 50% (i.e. 4 s on and 4 s off) to a coil with a resistance of 2.7 Ω results in an average power dissipation of 49 W, 1-2 W of which is deposited in the sample. We suggest that a power dissipation of no greater than 100 W be employed.

Disconnect the cable from the polarising coil on the probe. Measure the resistance of the polarising coil. This can be done by putting a multi-meter between pins 1 and 2 of the outermost coil connector on the EFNMR probe. Re-connect the polarising coil.

The duty cycle of your experiment can be calculated as the ratio of the polarisation pulse duration to the total time for a single experiment (the repetition time). A suggested maximum current is 6 A and maximum polarisation duration is 5 s. Calculate the coil power dissipation for a polarising current of 6 A and a duty cycle of 50%.

Choose polarisation duration parameters that yield an array of values up to a maximum of 5 s. Run the T_1 experiment.

The output of this experiment is two-fold. A list of polarisation duration values and the corresponding signal magnitude, obtained by integrating the spectrum under the peak (within the integration width) are printed to the CLI window and these values are plotted on the far right in the 1D plot window. The data is fitted to equation 3.5 in order to determine a value for T_1 . What is your value for the T_1 of water in B_p ? (If you wish to fit the data independently, the values printed to the CLI window can be copied and pasted into a separate program with plotting and fitting capabilities.)

3.4.3. Measuring T_1 in Earth's Magnetic Field

The previous experiment explored the T_1 relaxation process during the application of the polarisation pulse. After the polarisation field is switched off adiabatically (slowly on the time scale of the precessing magnetisation) an enhanced magnetisation vector is aligned with the direction of the Earth's

3-4 Earth's Field NMR Experiments

magnetic field. This magnetisation is no longer in an equilibrium state because the dominant static magnetic field is now the Earth's magnetic field, rather than the polarisation field. The equilibrium magnetisation, now proportional to B_E , is much smaller. Therefore the net magnetisation will relax from the enhanced value, which is proportional to B_p , to the equilibrium value, which is proportional to B_E and thus essentially zero. This relaxation process is caused by spin-lattice relaxation. The time constant describing this relaxation process is T_1 ; however, this T_1 value is dependent on the Earth's magnetic field, B_E , and may differ from the T_1 value in the polarisation field for some samples.

In order to measure T_1 in the Earth's field we will use the conventional pulse and collect experiment, with an added delay between the polarisation pulse and the 90° excitation pulse. During the delay between the polarising pulse and the excitation pulse the net longitudinal magnetisation will decay according to equation 3.4. The amount of signal excited by the 90° pulse will be directly proportional to the amount of available magnetisation and therefore the observed signal magnitude will decay as a function of the delay, t , in the same fashion as the magnetisation.

$$S(t) = S_p \exp(-t/T_1) \quad [3.6]$$

The pulse sequence diagram for measuring T_1 in the Earth's field is shown in Figure 3.3.

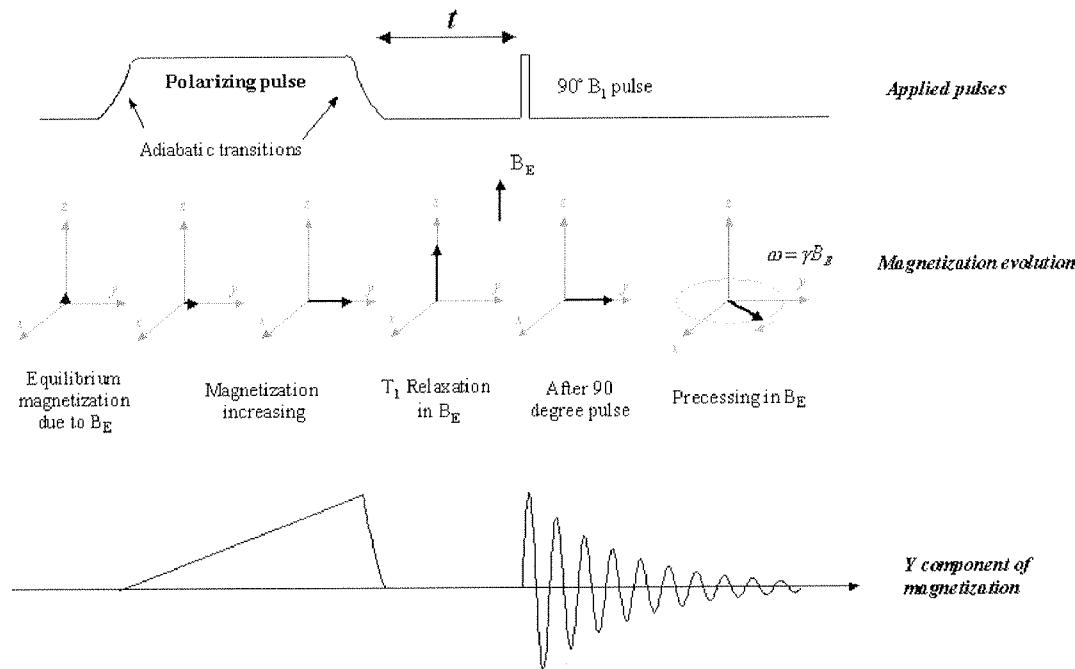


Figure 3.3. Pulse sequence diagram for measuring T_1 in B_E

Choose the “T1BE” experiment from under the EFNMR menu.

Use the same basic acquisition parameters as in the previous T_1 experiment and use a polarisation duration of 4 s and a polarisation current of 6 A. Choose an array of delay values that will be sufficient to characterise an exponential decay with a time constant equal to the T_1 you obtained in the polarising field. Use 8 – 10 steps. Run the experiment. (If, as the experiment runs, the delays chosen prove to be insufficient to describe the decay, stop the acquisition and choose a more appropriate array of delay values.)

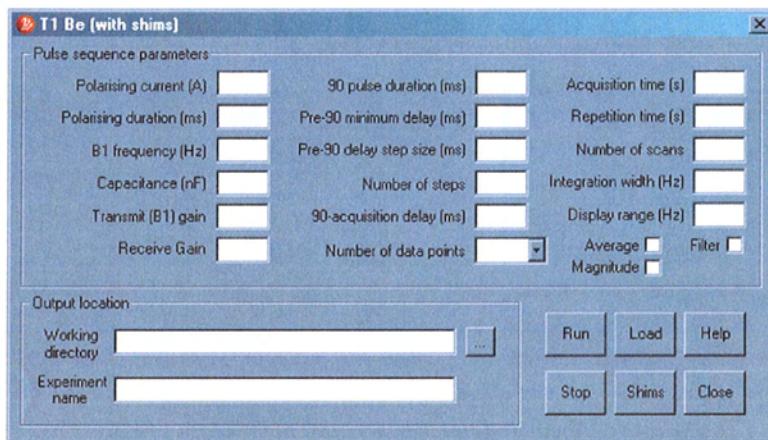


Figure 3.4 T_1 measurement dialog (in B_E)

The output of this experiment, like the previous T_1 experiment, will be printed in the CLI window and plotted to the 1D plot window. What is the value of T_1 in the Earth's field obtained by fitting the data to equation 3.6? How does it compare with the value in the polarisation field?

3.4.4. The Spin-Lattice Relaxation of Oil

Repeat the B_p and B_E T_1 measurements for a sample of cooking oil, such as soy bean oil. How do the T_1 time constants compare to those of the tap water sample? Why might this be the case?

3.5. Further Questions

1. What is field-cycled NMR Relaxometry?
2. What would the expected results be for an inversion recovery experiment carried out on a conventional high field NMR spectrometer?

3.6. Appendix for the Instructor

The purpose of this experiment is to introduce the idea of T_1 relaxation and to teach the student how to measure T_1 in both the polarising field and the Earth's field.

A T_1 measurement in the polarising field will result in an echo amplitude which will increase from zero to an equilibrium value. In the following example the T_1 was found to be 2.28 s.

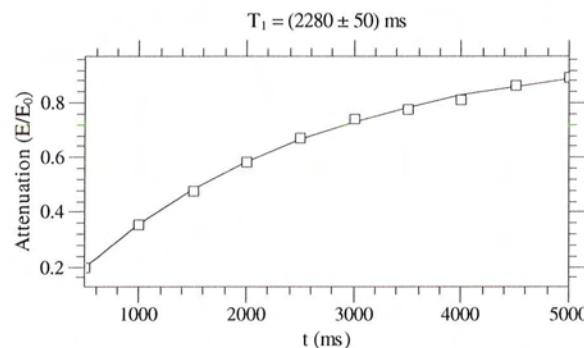


Figure 3.5 T_1 Analysis for data acquired in the polarising field.

A T_1 measurement in the Earth's field will result in a signal magnitude that decreases from the enhanced signal magnitude (due to the polarisation) to effectively zero. In the following example, the T_1 value was found to be 2.32 s.

3-6 Earth's Field NMR Experiments

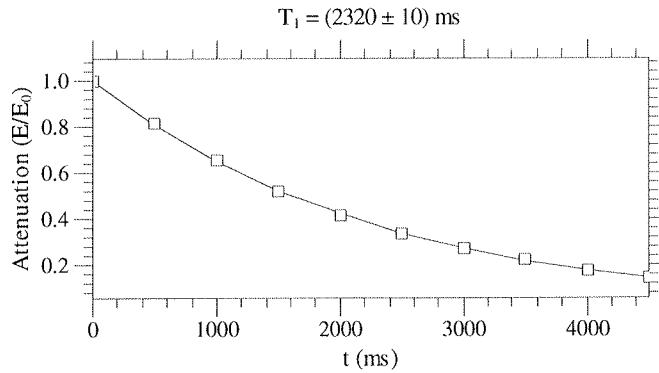


Figure 3.6. T_1 Analysis for data acquired in the Earth's field.

In the case of bulk water there is very little difference between T_1 at B_E and at B_p .

The following are T_1 measurements for a soy bean oil sample. The reduced mobility of the ^1H nuclei results in a significant reduction in T_1 , when compared with the bulk water samples.

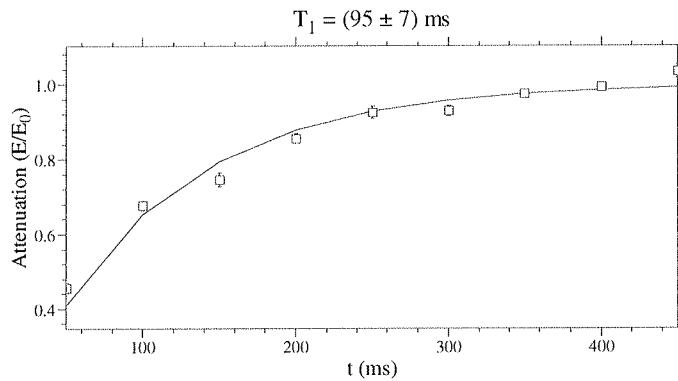


Figure 3.7 T_1 in B_p for a sample of soy bean oil.

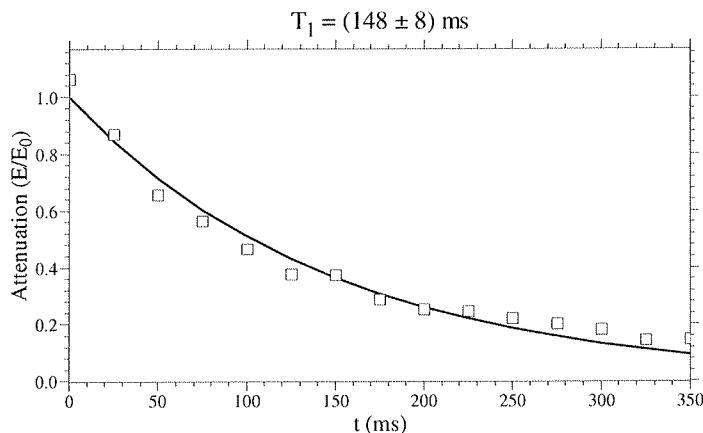


Figure 3.8 T_1 in B_E for a sample of soy bean oil.

Note that the relaxation values you obtain will be very temperature dependent.

4. Spin-echoes and Spin-Spin (T_2) Relaxation ✓

4.1. Objective

The object of this experiment is to learn about spin-spin relaxation in NMR and to observe the relationship between the spin-spin relaxation time constant, T_2 , and the effective spin-spin relaxation time constant, T_2^* . The concept of a spin-echo will be introduced and used to demonstrate the difference between T_2 and T_2^* . The T_2 time constant for a bulk water sample will be measured using a spin-echo experiment.

4.2. Apparatus

The Terranova-MRI EFNMR system, consisting of the EFNMR probe, spectrometer and a controlling PC, will be used for this experiment. All experiments will be run from the *Prospa* software package. The sample is a 500 ml bottle of tap water.

4.3. Background Theory

4.3.1. Spin-Spin and Effective Spin-Spin Relaxation

The NMR signal arises from the phase coherence of an ensemble of precessing nuclear spins. The exponential decay of the signal is a consequence of the loss of phase coherence between the spins. One source of phase coherence loss is the relaxation process known as spin-spin relaxation (characterised by the T_2 relaxation time constant). Spin-spin relaxation is caused by the magnetic dipole coupling between two neighbouring spins. In the presence of a completely homogeneous field, spin-spin relaxation is the dominant source of phase coherence loss. Therefore the sampled signal, as a function of time is given by:

$$S(t) = S_0 \exp(-t/T_2) \quad [4.1]$$

where S_0 is the initial signal magnitude at $t = 0$. For a bulk water sample, the T_2 time constant is typically on the order of a couple seconds and therefore the NMR signal from such a sample should, ideally, persist for a number of seconds. However, in practice, the magnetic field is not entirely homogeneous and therefore the loss of phase coherence is not solely due to spin-spin relaxation but is rather a combined effect of spin-spin relaxation and magnetic field inhomogeneity. Local magnetic field inhomogeneities introduce a range of Larmor frequencies across the sample. Each nucleus will precess at the Larmor frequency associated with its position. The overall phase coherence of the ensemble depends on all nuclei precessing at the same frequency. Therefore this dispersion of frequencies will result in a loss of phase coherence and hence signal decay.

To describe the combined relaxation effects, an effective spin-spin relaxation time constant, T_2^* is defined, where γ is the gyromagnetic ratio of the observed nucleus and ΔB_0 is the magnetic field inhomogeneity.

$$\frac{1}{T_2^*} = \frac{1}{T_2} + \gamma \Delta B_0 \quad [4.2]$$

Therefore the observed signal as a function of time in a situation with significant magnetic field inhomogeneity is given by:

$$S(t) = S_0 \exp(-t/T_2^*) \quad [4.3]$$

where S_0 is the initial signal magnitude at $t = 0$. The phase coherence loss due to spin-spin relaxation is essentially irreversible, whereas the coherence loss due to magnetic field inhomogeneity *can* be reversed by generating a so-called *Hahn-echo* (or spin-echo).

4.4. Procedure

4.4.1. Getting Started

Run through the setup procedures from Experiments 1 & 2 to acquire a good quality FID of a large tap water sample. This process should include:

- Shimming.
- Setting the B_1 frequency to the Larmor frequency of the sample.
- Tuning the coil to the Larmor frequency of the sample.
- Determining the length of the 90° and 180° pulses.

4.4.2. Spin-Echoes

For the next section of this experiment the spin-echo pulse sequence in Figure 4.1 will be employed.

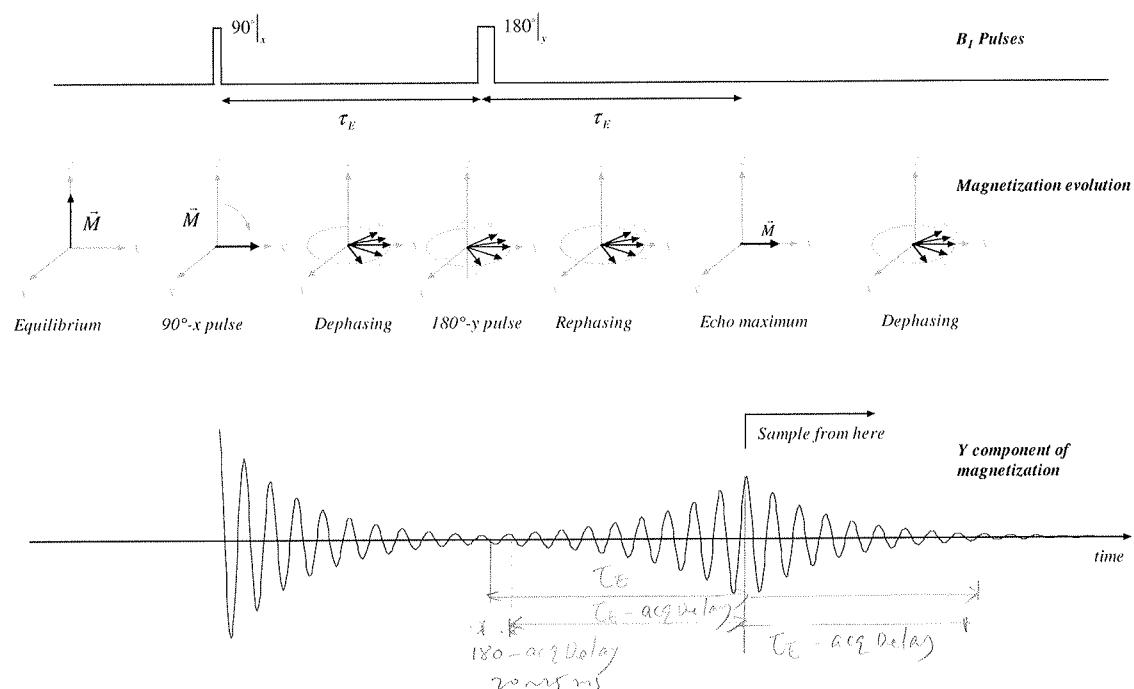


Figure 4.1. The spin-echo pulse sequence diagram.

The spin-echo sequence manipulates the phase coherence of an ensemble of excited spins. The first step in the experiment is the polarisation of the sample with a polarising pulse from the polarising coil, as is done in the simple pulse and collect experiment. This step is not shown in the above diagram.

The second step is to excite the sample with a 90° pulse. This results in the rotation of the bulk magnetic field vector into the transverse plane. In the subsequent delay time-period, τ_E , the magnetisation de-phases from the combined effects of spin-spin relaxation and magnetic field inhomogeneity. The former, caused by the random motion of the spins, is essentially irreversible whereas the latter can be reversed. Reversing the de-phasing due to magnetic field inhomogeneities is the goal of this basic spin-echo experiment.

At a time τ_E following the first 90° pulse, a 180° pulse is applied to the sample. This pulse flips the magnetic field vectors about a given axis in the transverse plane. During the subsequent time period, τ_E the magnetisation re-phases and forms what is known as a spin-echo. Only the de-phasing that occurred as a result of magnetic field inhomogeneity will be re-focused.

Open the spin-echo experiment from under the EFNMR menu. The following dialog will appear.

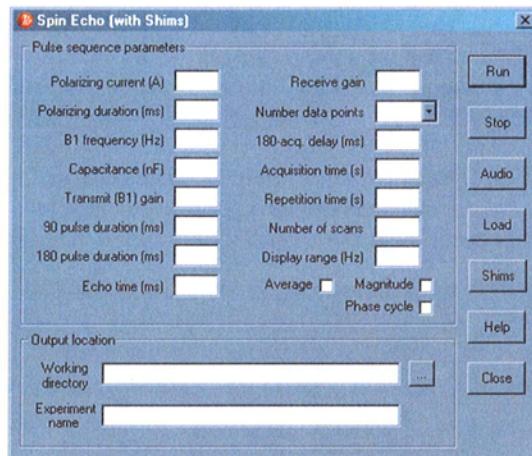


Figure 4.2 Spin-echo experiment dialog

The delays for this experiment must be chosen carefully, with attention given to how they interrelate. The time between the 90° pulse and the 180° pulse is called τ_E , the echo-time. The centre of the spin-echo is at a time, τ_E , after the 180° pulse. In Figure 4.1 the sampling commences at the centre of the echo and therefore the delay between the 180° pulse and the first sampled data point is τ_E . However, we would like to observe the entire echo and so we wish to sample as soon as possible after the 180° pulse. The acquisition delay, *acqDelay* (**180-acq. delay**), following the 180° pulse, must accommodate the ring down of the coil and should thus be at least 20 to 25 ms. If data is acquired for a time, t_{acq} (**acquisition time**), then the echo will fall in the centre of the acquisition window only if equation 4.4 is fulfilled.

$$t_{acq} = 2(\tau_E - \text{acqDelay}) \quad [4.4]$$

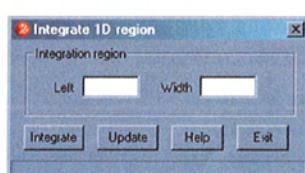
The echo time must be chosen to be long enough to allow the user to view the entire echo and to allow for the complete relaxation of the signal excited by the 90° pulse. Inspect the signal, in the FID window, from a pulse and collect experiment. How long does it take for the signal to decay? Choose an echo time, τ_E , which is a bit longer than the signal persists in the FID window. Choose an acquisition delay of 25 ms and use these parameters to calculate an appropriate acquisition time. Run the experiment. You should observe an echo signal in the FID window. In order to see the echo clearly, try averaging 4 or 9 scans.

4.4.3. Magnetic Field Inhomogeneity and the FID

In this experiment the spin-echo sequence will be used to demonstrate the difference between T_2 and T_{2^*} relaxation and to measure T_2 . However, first we shall return to the pulse and collect experiment to observe the effect of magnetic field inhomogeneity on the FID and spectrum.

Acquire an FID of the tap water sample, using the pulse and collect experiment and the 90° pulse. Estimate the peak voltage of the FID signal. The FID signal decay can be described by an exponential decay with a single decay time constant. Estimate this time constant by finding the time it takes for the signal to decay to approximately $1/e$ (37%) of its initial value. Estimate the width of the peak in the spectrum and choose a frequency range for integrating the sample peak. Integrate under the sample peak in the spectrum to determine signal amplitude. *Aside:* The spectrum peak can be integrated using the 1D macro “integrate1D”, which is found under the “1D” menu in the main *Prospa* window (see

Figure 4.3.) In the 1D plot window, use the “Allow Region Selection” tool to select a region around the sample peak.



The “left” and “width” parameters in the Integrate 1D window should update according to the region chosen. If it does not do this automatically, click “Update”. Once these parameters are correct click “Integrate”. The integration results will be printed to the command line interface (CLI) window.

Figure 4.3. Integrate 1D dialog

4-4 Earth's Field NMR Experiments

The next step is to deliberately increase the inhomogeneity of the static field and observe the effects. This can be accomplished by changing the shim settings from the optimal values found during the experiment setup to non-ideal values.

In the pulse and collect experiment window, click “Shims”. The shim dialog will appear (Figure 4.4). The “Saved” values are the values determined during your instrument setup. The “Current” values are the values used during the given experiment.

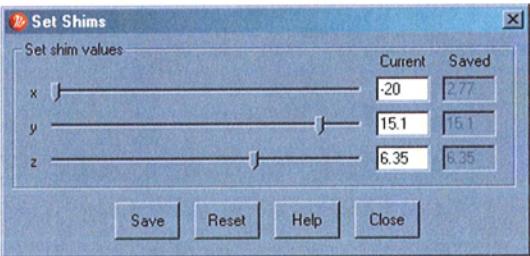


Figure 4.4 The set shims dialog with the x axis shim set to a non-ideal value

Move the x axis slider to a value very different from your ideal saved value. Leaving the shim dialog open, run the pulse and collect experiment. How do the FID and the spectrum change relative to your last measurement? Estimate the peak voltage and decay time constant value of the FID signal. Estimate the width of the peak in the spectrum. Integrate under the sample peak in the spectrum to determine signal amplitude. How do the values compare with the well-shimmed case? Explain your observations using the definition for T_2^* .

Close the set shim dialog without saving the current values. Close the pulse and collect dialog.

4.4.4. T_2 and T_2^* Relaxation

In this section of the experiment we will explore the differences between T_2 and T_2^* . Recall the relationship between these two relaxation times:

$$\frac{1}{T_2^*} = \frac{1}{T_2} + \gamma \Delta B_0$$

T_2 is a consequence of the irreversible de-phasing via spin-spin relaxation, resulting from the random motions of the spins. T_2^* combines the irreversible spin-spin relaxation effects (T_2 term) with the reversible magnetic field inhomogeneity de-phasing effects (ΔB_0 term).

Acquire a spin-echo with 4 or 8 averages. Remember to use a pulse and collect FID to select an appropriate echo time and to calculate an appropriate acquisition time. Record the echo amplitude value (printed in the CLI window), that has been obtained by integrating the area under the spectral peak. Repeat the same spin-echo experiment with non-ideal shim values (as done above with pulse and collect). The non-ideal shim values will introduce inhomogeneity into the local Earth's field. In theory, how will this change T_2 ? T_2^* ? In what way does the echo differ? How do the echo amplitudes compare? How do these results compare with the change in signal amplitude obtained for a simple pulse and collect experiment after the same length of time?

Repeat the echo experiment for a number of longer echo times. Record the echo amplitudes as a function of echo time, both in the shimmed and de-shimmed case. Plot the echo amplitudes (both in the shimmed and de-shimmed case) as a function of echo time. What kind of relationship between echo time and echo amplitude do you observe? Is there a difference between the two plots? What relaxation time dependence is there on echo amplitude (T_2 or T_2^*)? What relaxation time dependence, T_2 or T_2^* , is there on the signal in the FID?

The spin-echo experiment is advantageous because in areas of high magnetic field inhomogeneity it can be used to refocus the signal that decays too quickly to be observed with a pulse and collect experiment. Why?

Run a pulse and collect experiment. Repeat the experiment with non-ideal shim values, repeating the experiment with continuously ‘worse’ shim values until the FID is reduced to just noise. Open the spin-echo experiment and enter the same shim values into its set-shim dialog. Acquire a spin-echo with a short echo time (100 ms) and an appropriate acquisition time. Employ 16 averages. Is any signal observed? What does the spectrum look like? (It may be necessary to observe a wide frequency range of several hundred Hz in order to see anything interesting.) The centre of the echo occurs at a time $2\tau_E$ following the signal excitation (the 90° pulse). What does this result suggest about the relaxation time dependence of the signal magnitude at the centre of the spin-echo?

Close the set-shim dialogs without saving the shim values.

4.4.5. T_2 Measurement

T_2 is measured using a succession of spin-echo experiments with incrementally longer echo times. The plot of echo amplitude as a function of echo time will be an exponential decay with a characteristic decay time constant, T_2 . That is, the echo amplitude will be described by:

$$E(\tau_E) = E_0 \exp(-2\tau_E/T_2) \quad [4.5]$$

where E is the amplitude of an echo acquired with an echo time, τ_E , and E_0 is the echo amplitude in the absence of a T_2 decay. The factor of two appears in the exponential because the centre of the echo occurs at a time $2\tau_E$ after the 90° excitation pulse.

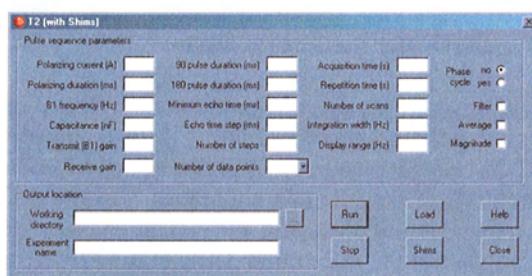


Figure 4.5 T_2 experiment dialog

Open the T_2 experiment from the EFNMR menu. This macro will repeat the spin-echo experiment for an array of echo times. Sampling will commence at the centre of the echo, so the full echo will not be observed. The output of the macro will be the echo amplitudes at each echo time. The echo amplitude is determined by integrating the sample peak in the spectrum. The integration interval is defined in the T_2 dialog. Observe a spectrum from the water sample and choose an appropriate narrow frequency range around the sample peak for this integration.

To accurately measure T_2 , using short echo times on the order of 50 ms, it is necessary to purposely disrupt the homogeneity of the Earth’s magnetic field such that the FID decays quickly. Why do you think this is necessary? This is accomplished, as above, by de-shimming.

The T_2 for a bulk water sample is on the order of 2 s. Choose an array of echo times for the T_2 experiment accordingly. 50 ms is the shortest echo time that should be employed. What limits the minimum echo time? What is the longest echo time that should be employed to accurately fit the exponential T_2 decay if the T_2 of the sample is 2 s?

Run the experiment. The echo amplitude data is printed to the CLI window and is also plotted in the 1D plot window. The data in the 1D plot window is fitted to equation 4.1 and a value for T_2 is reported in the plot title. If you wish to independently fit the data, copy the output echo amplitude data from the CLI into a spreadsheet program with plotting and fitting capabilities. Plot the echo amplitude vs. twice the echo time and fit the echo data to the exponential function (equation 4.1) in order to determine a value for T_2 .

4.5. Further Questions

1. How would you measure the T_2^* of a sample?
2. How is the spin-spin relaxation time, T_2 , of a sample used to advantage in Magnetic Resonance Imaging (MRI)?

4.6. Appendix for the Instructor

In this experiment, spin-echoes are used to demonstrate the difference between T_2 and T_2^* relaxation times and are also used to measure T_2 . The difference between T_2 and T_2^* can be seen in the following relation:

$$\frac{1}{T_2^*} = \frac{1}{T_2} + \gamma \Delta B_0$$

T_2^* is reduced by the presence of magnetic field inhomogeneities, ΔB_0 . This is demonstrated in this experiment by de-shimming, i.e. changing the shim values away from the ideal values found during the setup of the instrument. This will disrupt the homogeneity of the local Earth's magnetic field and thus reduce T_2^* but have no effect on T_2 .

The FID signal from a pulse and collect experiment decays with the T_2^* time constant. This is because the magnetic field inhomogeneities across the sample will cause the spins to precess at slightly different resonance frequencies. This spread of frequencies results in a loss of phase coherence and hence a decrease in the observed signal. The greater the inhomogeneity, the more phase coherence, and hence signal, will be lost and so the shorter the decay constant, T_2^* .

FID acquired in the presence of a inhomogeneous field: the student should observe that the FID decays more rapidly and the observed peak amplitude of the FID and signal amplitude obtained from integrating the spectrum, will decrease dramatically from that observed in the homogeneous field case. The decrease in the peak amplitude of the FID is a consequence of the delay between excitation and acquisition. During this delay signal is lost due to the T_2^* relaxation. In addition, the width of the peak in the spectrum will broaden because of the more rapid decay of the FID signal.

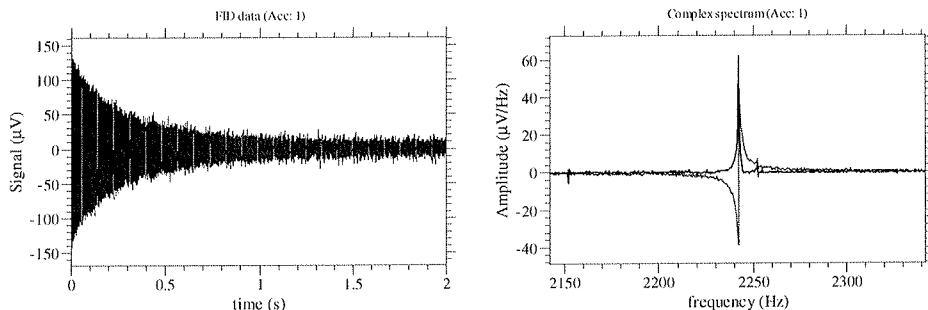


Figure 4.6 An example of an FID and spectrum acquired in the a field with good homogeneity.

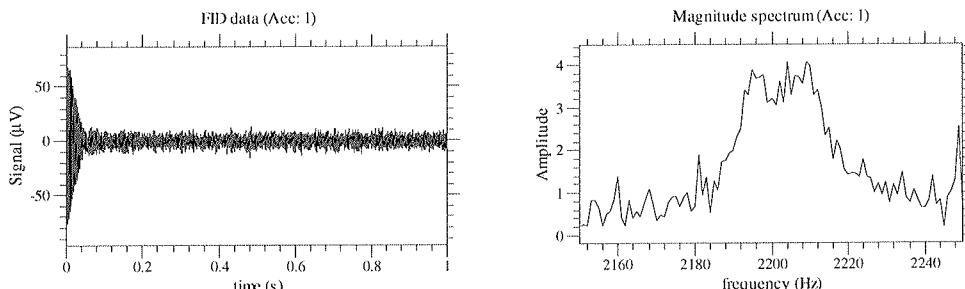


Figure 4.7 An example of a very short FID signal, caused by significant magnetic field inhomogeneity, and the corresponding broad peak in the NMR spectrum.

Spin-echo: the spin-echo amplitude is dependent on T_2 , not T_2^* because the de-phasing caused by the magnetic field inhomogeneities is reversed by the spin-echo pulse sequence. Therefore in the de-shimmed case the student should observe that the sides of the echo decay more rapidly (this is determined by T_2^*) but that the echo amplitude (in the time domain) and the integral under the peak (in the frequency domain) is not changed significantly.

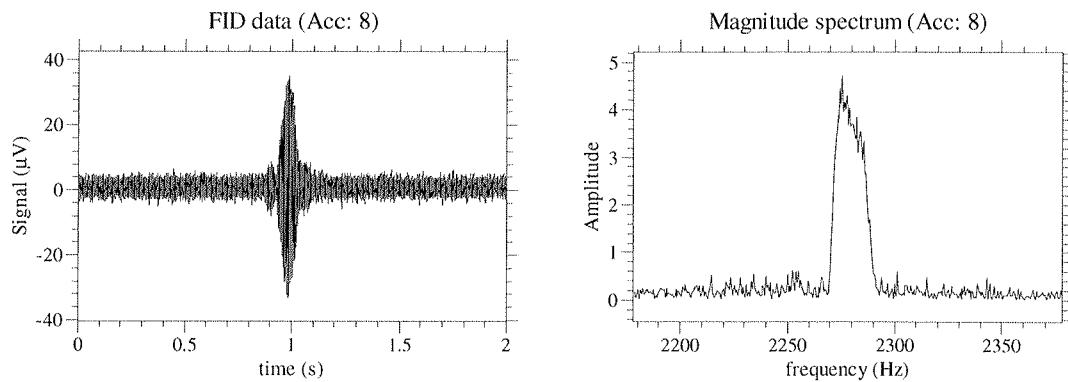


Figure 4.8 A spin-echo signal acquired in a highly inhomogeneous field.

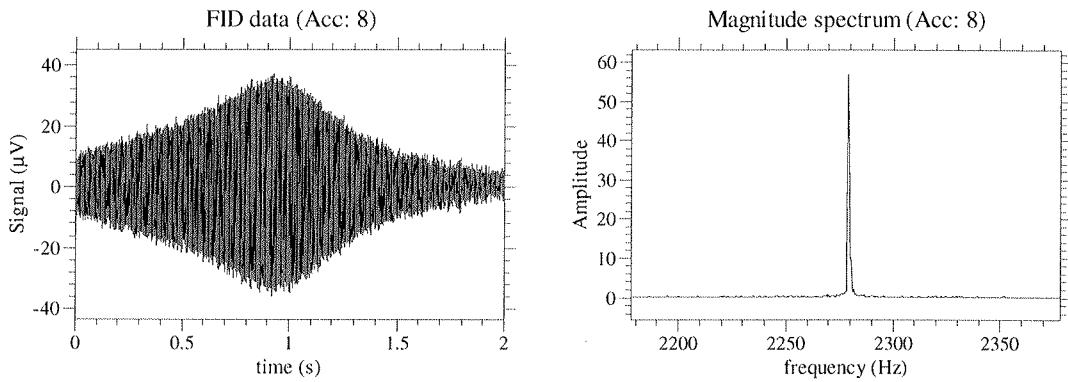


Figure 4.9 A spin-echo signal acquired in the presence of a reasonably homogeneous field.

At longer echo times the echo amplitude should decrease in the same manner for experiments in the shimmed and de-shimmed case because the decay of the echo centre is dependent on T_2 , which does not change in the presence of magnetic field inhomogeneity.

It is likely that the students will not observe perfect results because of the non-ideal nature of the 180° pulse. If this pulse is not exactly 180° then the refocusing of dephasing spins due to inhomogeneities in the field is not complete and so the echo amplitude will still have a small dependence on ΔB_0 . Therefore there will be some change observed between the experiments in the shimmed and de-shimmed cases. Regardless, the difference between the effect of de-shimming on an FID from the pulse and collect experiment and on the spin-echo signal should be very marked and thus the student results should demonstrate the utility of the spin-echo technique.

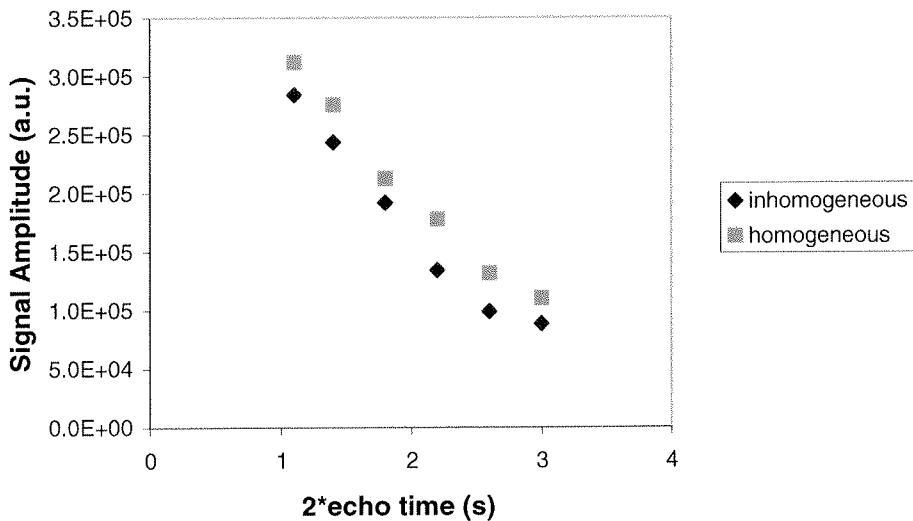


Figure 4.10 The amplitude of the spin-echo over a range of echo times acquired in a homogeneous and an inhomogeneous region, respectively.

Spin-echo T_2 Measurement: The minimum echo time is limited by the ring-down of the coil. The maximum echo time employed to accurately measure T_2 should be several times T_2 . For example with a T_2 of 2 s, the maximum echo time should ideally be around 6 s to fully capture the T_2 decay. However, a much shorter maximum echo time is often sufficient to capture the T_2 of the decay.

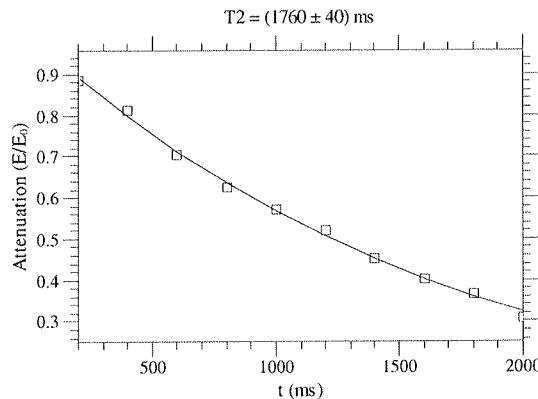


Figure 4.11 Typical results for a measurement of the T_2 of a tap water sample. These results were obtained in de-shimmed conditions.

It is often necessary to purposely disrupt the homogeneity of the Earth's magnetic field in order to accurately measure T_2 because, following the 90° pulse, the excited magnetisation will decay according to T_2^* . For short echo times, it is possible that this signal will not decay fully before the 180° pulse. Additionally, if the 180° is non-ideal then it may act as a partial excitation pulse and a partial re-focusing pulse. Both the signal from the 90° pulse and any non-ideal behaviour of the 180° pulse will interfere with the T_2 measurement. If the field homogeneity is purposely "spoiled" via de-shimming, then these signals decay rapidly and do not interfere with the echo formed after the 180° pulse.

Important !!

5. Multiple-echo experiments: CPMG



5.1. Objective

The object of this experiment is to achieve single-shot spin-spin relaxation time constant (T_2) measurements using the Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence. This experiment will introduce the basic principles of multiple-echo experiments and explore the effects of the relative pulse phases on echo amplitudes. Some interesting post-processing methodologies such as filtering and zero filling will also be touched upon.

5.2. Apparatus

This experiment uses the Terranova-MRI EFNMR instrument, consisting of a three-coil probe, a spectrometer and a controlling PC. The experiments are all run from the *Prospa* software. The sample is a large bottle of tap water.

5.3. Background Theory

5.3.1. Carr-Purcell-Meiboom-Gill (CPMG) Pulse Sequence

In a spin-echo NMR experiment (Figure 5.1) a 180° RF pulse is used to re-focus transverse magnetisation de-phased due to local magnetic field inhomogeneities.

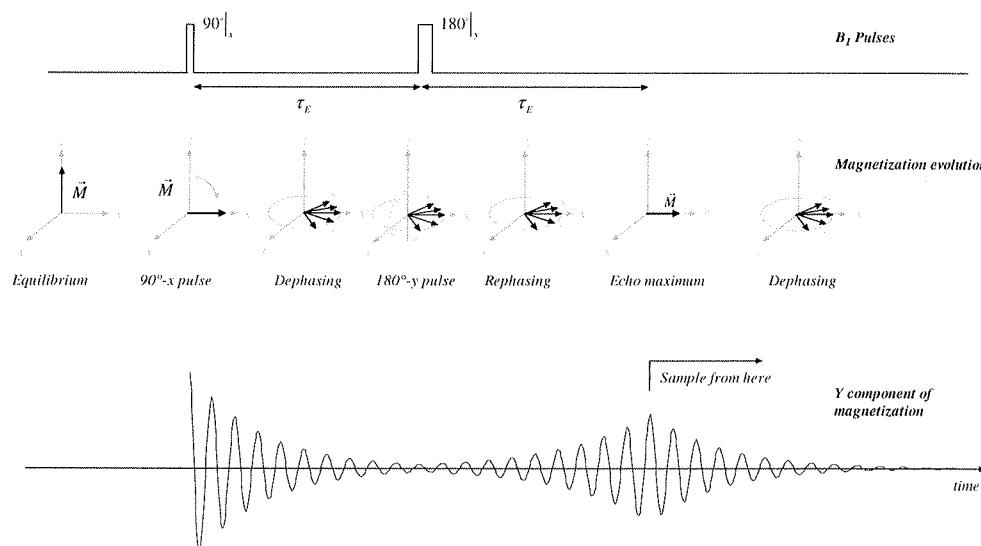


Figure 5.1. The spin-echo pulse sequence diagram.

The spin-echo pulse sequence consists of two RF pulses, a 90° pulse followed, after an echo time period (τ_E), by a 180° pulse. The echo forms during the echo time period following the 180° pulse, such that the centre of the echo occurs at a time τ_E after the centre of the 180° pulse. This methodology re-focuses any NMR signal de-phased due to local magnetic field inhomogeneities; however, de-phasing due to spin-spin relaxation is irreversible and so the echo amplitude is weighted by the spin-spin relaxation decay term, characterised by the spin-spin relaxation time constant T_2 .

In the Carr-Purcell (CP) sequence multiple re-focusing pulses are used to generate a train of spin-echo signals. The CP pulse sequence diagram is shown in Figure 5.2. The first 90° excitation pulse excites the signal. After a time period τ_E , a 180° re-focusing pulse is employed to generate the first echo. Subsequent 180° re-focusing pulses are applied at intervals of $2\tau_E$ to generate additional echoes. An echo forms at a time τ_E following each 180° pulse. As mentioned above, the amplitude of each echo will be weighted by the T_2 decay. Therefore a plot of the echo amplitudes as a function of time (where $t = 0$ is the centre of the first 90° excitation pulse) will provide a record of the T_2 decay of the sample.

5-2 Earth's Field NMR Experiments

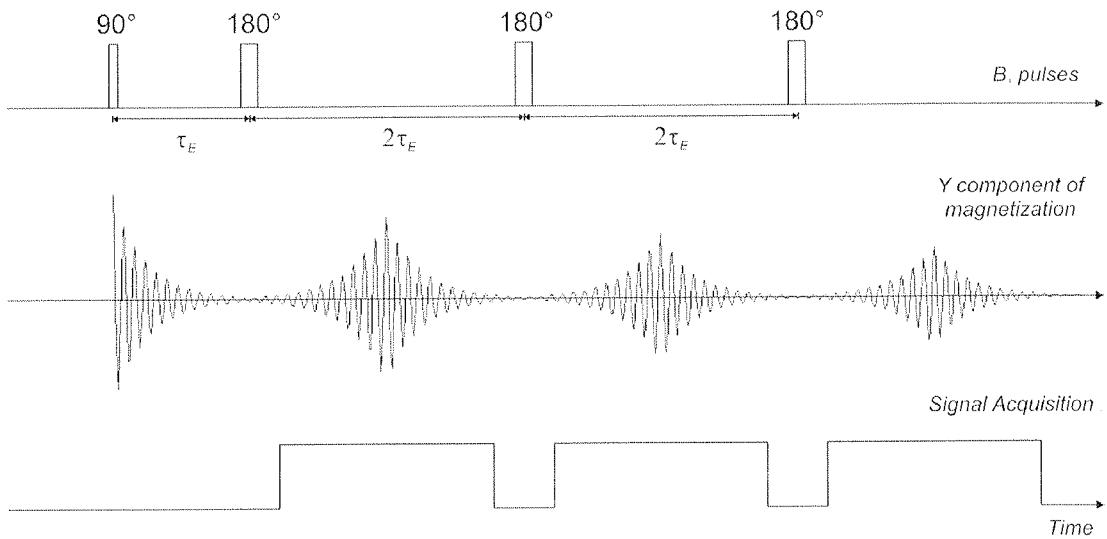


Figure 5.2. The Carr-Purcell (CP) multiple-echo pulse sequence

In the CP sequence each echo is sampled within an acquisition window centred about the middle of the echo. Each acquired echo can be Fourier transformed to yield an NMR frequency spectrum. The echo amplitude can be calculated from the spectrum as the integral under the spectral peak corresponding to the sample. Alternatively, the echo amplitudes can be determined directly from the time domain data as the intensity of the single point at the centre of the echo. The advantage of integrating the spectrum over the time domain method is that the integral rejects all noise outside of the integration range.

The CP sequence is very sensitive to non-ideal tip angles. If the 180° pulses are not ideal (i.e. if the pulse is either inhomogeneous such that the tip angle varies across the sample, or the tip angle of the pulse is slightly larger or smaller than 180°) then the re-focusing of magnetisation de-phased due to local magnetic field inhomogeneities will be incomplete. Thus the amplitude of each echo will be weighted by the imperfections in the refocusing pulses as well as by the T_2 relaxation of the sample. Pulse errors in the CP experiment are cumulative, i.e. the errors in the second echo are the result of not only the non-ideal nature of the second 180° pulse but also the first.

Compensation for echo amplitude errors resulting from non-ideal re-focusing pulses can be achieved by introducing appropriate phase shifts between the B_1 excitation and re-focusing pulses. The Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence is an adaptation of the CP sequence that compensates for errors due to non-ideal 180° pulses using a particular phase scheme. This pulse sequence will be explored in this experiment.

5.3.2. Time Domain Filters

Time domain filters are commonly employed as a post-processing device for manipulating the characteristics of the frequency domain data. The example to be employed in this experiment is a sine-bell-squared filter for echo data. This filter is defined by the following expression:

$$\cos^2\left(\frac{\pi(t - \tau_2)}{N}\right) \quad [5.1]$$

where N is the number of points in the filter. This function is depicted graphically in Figure 5.3.

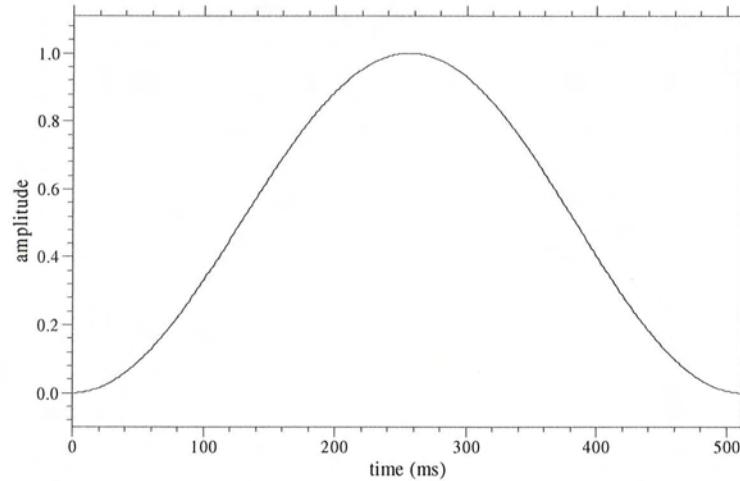


Figure 5.3 A plot of a sine-bell-squared time domain filter.

Consider an acquired echo signal such as that presented in Figure 5.4. The signal is acquired over a time period of 1 second. In many applications a short echo time, τ_E , is desired. In such cases sampling with a one second acquisition window is not feasible. If the signal in Figure 5.4 is only sampled for the short time period highlighted by the red box the edges of the signal will be severely truncated. Through the convolution theorem of Fourier transforms, it is known that the truncation of the signal at the ends of the sampling window will result in a convolution of the ideal narrow spectrum with a sinc function. This will introduce undesired oscillations into the spectrum. This effect can be countered through the use of a time domain filter, such as the sine-bell-squared filter pictured above.

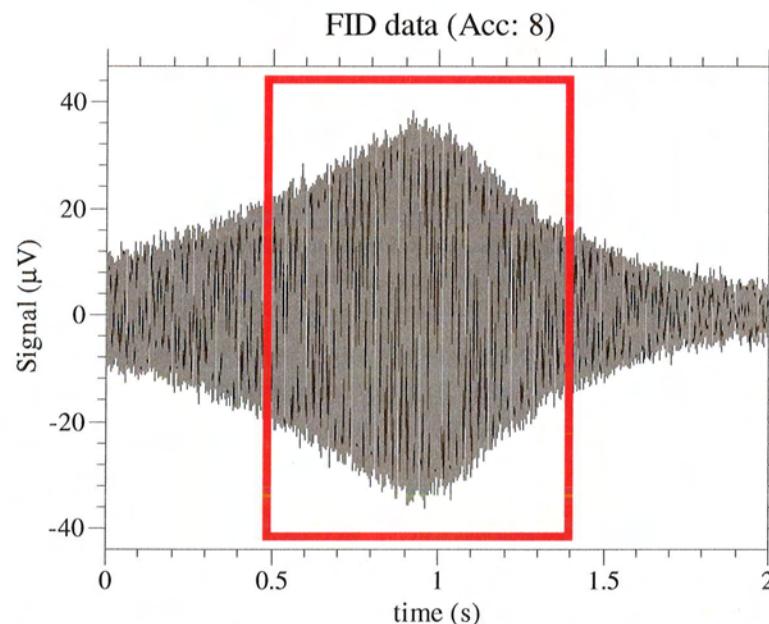


Figure 5.4 An example of a spin-echo data set. The red box highlights the area of the signal that would be sampled if only a short acquisition window was possible.

Applying the sine-bell-squared filter to the echo data within the red box in Figure 5.4 will artificially smooth the edges of the echo signal such that it progresses smoothly from the echo maximum at the centre to zero at the edges. A demonstration of the application of a sine-bell-squared filter is presented in Figure 5.5. The spectrum of the truncated echo signal contains a significant sinc-like oscillation, making it difficult to obtain a meaningful peak integral. In the spectrum calculated from the filtered echo, the sinc-like oscillations are smoothed; however, it is important to note that the filter broadens the spectral peak.

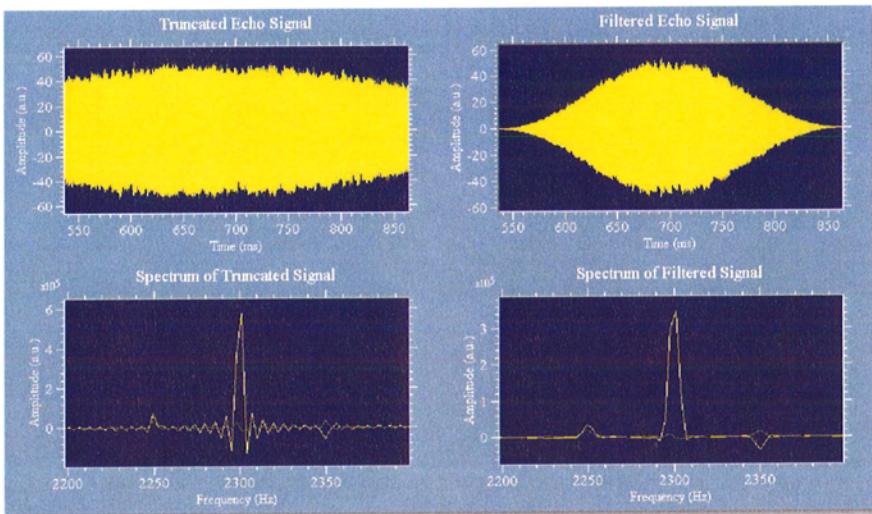


Figure 5.5 A demonstration of the application of a sine-bell-squared filter in the time domain. (The peaks at 2250 and 2350 Hz are due to external interference.)

5.4. Procedure

5.4.1. Instrument Setup

Run through the setup procedures described in Experiments 1 and 2 to acquire a good quality FID from a large tap water sample. This process should include:

- Shimming.
- Tuning the probe.
- Setting the B_1 frequency to the Larmor frequency of the sample.
- Determining the length of the 90° and 180° pulses.

5.4.2. Explore Spin-echo Parameters and Processing Options

In the first step of this experiment we will review the parameters of a spin-echo experiment. Recall the spin-echo pulse sequence shown in Figure 5.1. (Note that the polarisation pulse is omitted.) A 90° excitation pulse is followed, after a time τ_E called the echo time, by a 180° pulse which re-focuses magnetisation de-phased due to local magnetic field inhomogeneities. The refocusing of this signal, after a subsequent echo time period, is called a spin-echo.

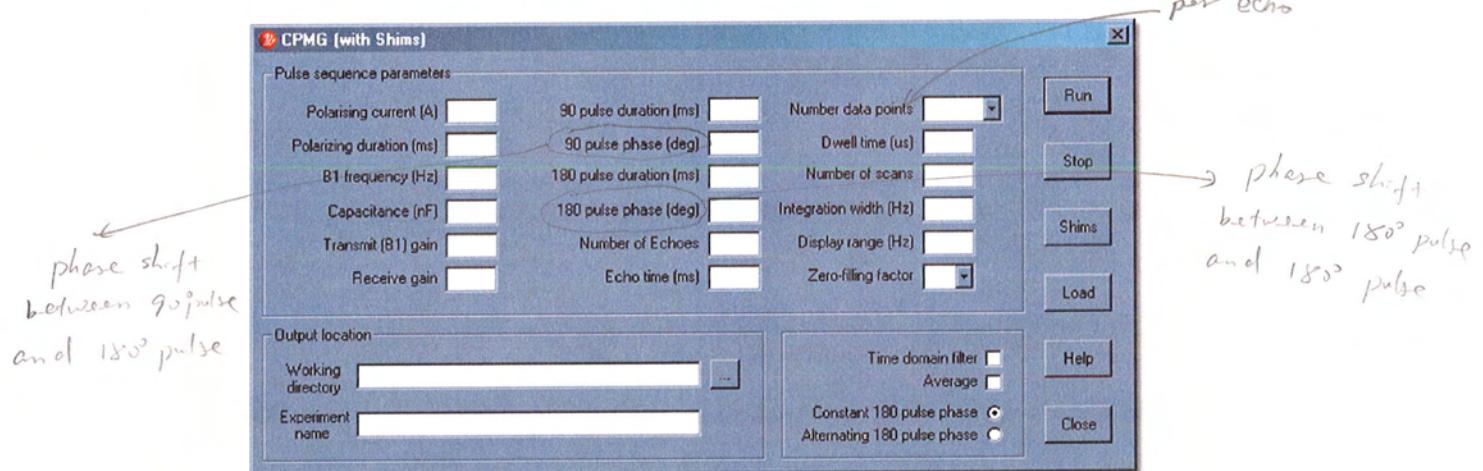


Figure 5.6 The CPMG experiment dialog window

Open the “CPMG” experiment from under the EFNMR menu. The dialog in Figure 5.6 will appear. Many of the parameters in this experiment will be familiar from previous experiments; however there are some new parameters that require some explanation.

The parameters in the first column on the left are familiar from previous experiments and should have been determined during the instrument setup. Enter the appropriate values.

In the central column of parameters fill in the 90° and 180° pulse durations. Two new parameters, the phase of the 90° and 180° pulses, are now present. These parameters take values between -360° and $+360^\circ$ and will be used later in the experiment to alter the relative phase of the excitation and re-focusing pulses.

The echo train parameters, "Number of Echoes" and "Echo time (ms)", determine the number and time spacing of the re-focusing (180°) pulses. The total number of acquired data points cannot exceed 32768. Therefore the number of acquired data points per echo, multiplied by the number of echoes, must be less than or equal to this number. In this section of the experiment, a single echo will be used. The echo time is constrained by the acquisition parameters and the ring-down of the coil as in the single-echo spin-echo experiments. This will be discussed further in the section on the acquisition parameters.

The acquisition parameters for the CPMG experiment are slightly different from the previous EFNMR experiments. Instead of choosing an acquisition time, the user chooses a dwell time, Δt . Dwell time is defined as the time between successively acquired data points in the time domain. Δt takes values that are multiples of $10 \mu\text{s}$. The acquisition time is equal to the dwell time multiplied by the number of points acquired per echo. Note that the "Number of points" parameter refers to the number of points acquired per echo. The "Number of scans", "Integration width (Hz)" and "Display range (Hz)" have the same meaning as in the T_2 experiment.

The acquisition parameters must be chosen carefully in a multi-echo experiment. The echo-time must be long enough to accommodate both the ring-down of the B_1 coil and the signal acquisition window. Therefore the echo time must be greater than half of the acquisition time (number of points \times dwell time) plus the coil ring-down (typically about 25 ms). Initially, choose an echo time of 110 ms, a dwell time of $10 \mu\text{s}$ and 16384 points. Using these parameters what is the acquisition time? What is the delay between the centre of the 180° pulse and the beginning of echo acquisition?

*refined
Fig 4.1*

The zero-filling factor will be discussed later. Choose 1 for now.

The CPMG dialog provides the option of applying a time domain filter. This will be discussed later. For now leave this box unchecked.

The average function has been discussed in previous experiments. For now we will be using only a single scan and so this box can remain unchecked.

The CPMG dialog provides the choice of two pulse sequences. In the first option: "Constant 180 pulse phase" all of the re-focusing (180°) pulses have the same phase. In the second option: "Alternating 180 pulse phase" the phase of the re-focusing (180°) pulses alternates by π (180°). For this initial section choose the "Constant 180 pulse phase" option.

Run the CPMG experiment. What does your echo signal look like? Is the echo signal truncated? Increase the acquisition time (increase the number of points and/or the dwell time) and re-run the experiment until the entire echo can be observed. (Remember to make an appropriate increase to the echo time to accommodate the longer acquisition times.) What is the drawback to using such long echo times? At short echo times, what is the consequence in the spectrum of a truncated echo signal?

Read the background theory section on time domain filters (section 5.3.2). Experiment with the application of a sine-bell-squared filter to truncated echo signals acquired with short echo times. What are the benefits of the filter? What are the drawbacks?

Re-call the relationship between acquisition time, t_{acq} , and spectral resolution, Δf :

$$\Delta f = \frac{1}{t_{acq}} = \frac{1}{\Delta t \times N},$$

where N is the number of points per echo and Δt is the dwell time. What is the spectral resolution using the short echo time parameters? This can be augmented through the process of zero filling. Zero filling increases the apparent acquisition time by padding the edges of the echo with zeroes. This does not add any new information to the spectrum but acts as an interpolation type step.

Experiment with the available zero-filling factors. What are the spectral resolutions for the zero-filling factor of 1, 2 or 4?

5.4.3. Multiple Echo Acquisition with the CP Sequence

Increase the number of echoes to be acquired. Remember to decrease the number of points per echo such that the total number of points acquired is less than or equal to 32768. Experiment with different numbers of echoes and different echo times. How do the echo amplitudes change with the number of echoes and with echo time? What is the expected relationship between echo amplitude and time? What would cause the relationship between echo amplitude and time to be different from the anticipated result?

5.4.4. The Carr-Purcell-Meiboom-Gill (CPMG) Sequence

The Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence corrects for non-ideal re-focusing pulse errors through a clever choice of the relative phase between the B_1 excitation and the re-focusing pulses. In this sequence the phase of all of the re-focusing pulses is the same, therefore the “Constant 180 pulse phase” pulse sequence will be used. The pulse phases are controlled through the pulse parameters in the CPMG experiment dialog. Using multiples of 90° only, run the experiment for a range of 90° and 180° pulse phases and observe the results. What is the relative phase between the 90° and 180° pulses that corrects for echo amplitude errors?

Echo error compensation can also be achieved through alternating the phase of the re-focusing pulses by 180° . Run the multiple-echo experiment using the “Alternating 180° pulse phase” pulse sequence and a range of 90° and 180° pulse phases. For what relative phase value is compensation achieved?

5.4.5. Single-shot T_2 measurements using CPMG

If pulse phases are chosen such that there is compensation for echo amplitude errors due to non-ideal re-focusing pulses, the CPMG experiment can be used to measure T_2 with a single experiment. Run the CPMG experiment using a short echo time (for example 110 ms). Experiment with the number of echoes until the entire T_2 echo amplitude decay can be observed over the time-scale of the experiment. This will typically be between 16 and 32 echoes. The echo amplitudes, calculated as the integral under the spectral peaks, are printed to the CLI (command line interface) window as well as being plotted on the far right in the 1D plot window. To fit the data independently, copy these values and the corresponding times ($t = 0$ is set as the centre of the first 90° excitation pulse) to a spreadsheet application. Plot the echo amplitudes as a function of time and fit an exponential decay to the data. What is the T_2 of the water sample? Compare this value with the results of a single-echo T_2 measurement.

5.5. Further Questions

1. Explain how the CPMG ($90_x - 180_y - 180_y \dots$) and alternating 180° pulse phase ($90_x - 180_x - 180_x \dots$) experiments compensate for non-ideal re-focusing pulse errors.
2. Consider the role of diffusion in a CPMG experiment. If there is significant diffusion on the time scale of the experiment, how do you think this will affect your measurement of T_2 ? Under what conditions will diffusion change your measured T_2 value? Can you think of a way to check the effects of diffusion on your measurement of T_2 ?

5.6. Appendix for the Instructor

The objective of this experiment is to introduce the student to multiple-echo experiments and to demonstrate the benefit of the CPMG sequence which compensates for non-ideal re-focusing pulses.

The CPMG experiment will be used to achieve a single-shot T_2 measurement of water. This method will be compared with the conventional single-echo T_2 measurement.

For a dwell time of 10 μs and 16384 points per echo the acquisition time is 0.16384 s. These parameters, along with an echo time of 110 ms, leave a delay of 28.08 ms between the centre of the re-focusing pulse and the beginning of signal acquisition.

$$110 - \frac{1}{2} \times 163.84$$

Due to the short acquisition window the echo signal should appear truncated. This truncated echo causes oscillations in the spectrum. A long acquisition time can be used to acquire the entire echo but this requires a longer echo time and hence a loss in S/N due to T_2 relaxation. Employing a sine-bell-squared filter (Figure 5.5) will smooth out the spectrum but will also broaden the peak.

The frequency resolution for the parameters of $\tau_E = 110$ ms, $\Delta t = 10$ μs and $N = 16384$ is 6.1 Hz. Employing zero-filling factors of 1, 2 and 4 will result in frequency resolutions of 6.1 Hz, 3.05 Hz and 1.525 Hz, respectively.

Using the CP multiple echo sequence the echo amplitude should decrease according to the T_2 relaxation of the nuclei. However, the decrease in echo amplitude with time will most likely be much quicker than can be attributed solely to T_2 relaxation because of the cumulative effects of non-ideal re-focusing pulses.

The student should find that a 90° phase shift between the excitation B_1 pulse and the re-focusing B_1 pulse will result in compensation for non-ideal 180° pulses. This is the CPMG sequence. The student should find, using the alternating 180° pulse phase sequence that a relative phase of 0° between the excitation and re-focusing pulses results in the desired compensation. Some example data sets are presented below (Figure 5.7, Figure 5.8 and Figure 5.9).

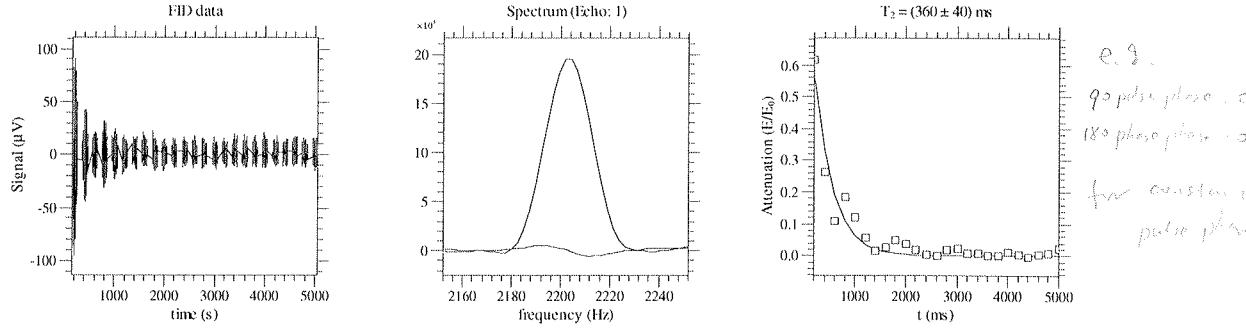


Figure 5.7. The original CP experiment employing constant phase re-focusing pulses. There is no (0°) phase shift between the excitation (90°) and re-focusing (180°) pulses. Non-ideal tip angles have a dramatic effect on the observed echo amplitudes.

$$90^\circ_x - 180^\circ_x - 180^\circ_x$$

In the final section of the experiment the student will use the CPMG experiment to acquire a single-shot T_2 measurement. Sample results are shown in Figure 5.8 and Figure 5.9. Results should be comparable to a single-echo T_2 experiment.

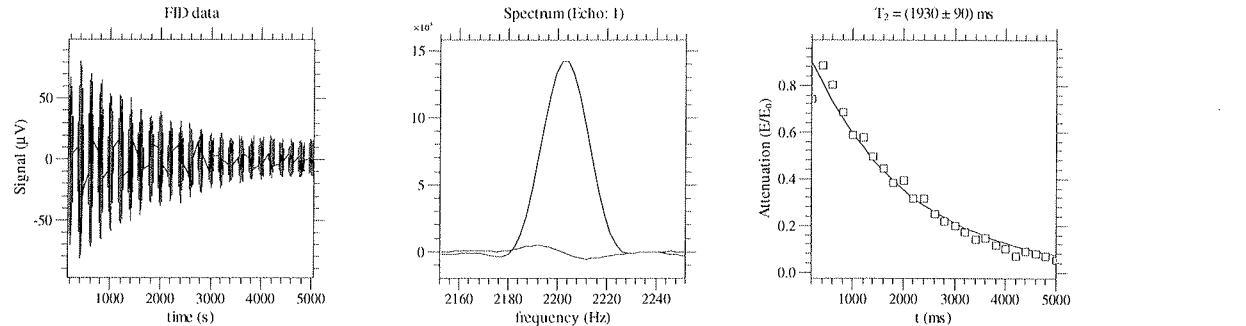


Figure 5.8 A sample single-shot T_2 measurement of water using the CPMG pulse sequence. The relative phase between the excitation (90°) and the re-focusing (180°) pulses is $\pi/2$ (90°).

$$\begin{aligned} 90 \text{ pulse phase: } & 90 \\ 180 \text{ pulse phase: } & 0 \end{aligned} \quad \text{for constant } 180 \text{ pulse phase}$$

$$90^\circ_x - 180^\circ_y - 180^\circ_y$$

5-8 Earth's Field NMR Experiments

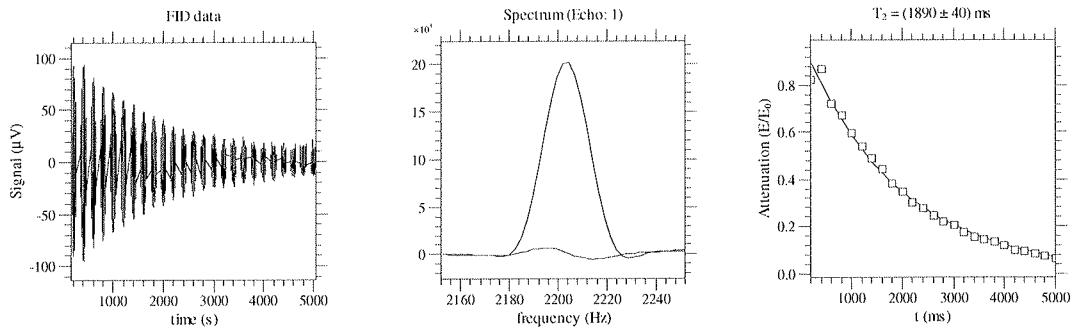


Figure 5.9 A sample single-shot T_2 experiment using the CPMG experiment with re-focusing (180°) pulses having phases alternating by π radians (180°). The relative phase between the excitation (90°) and re-focusing (180°) pulses is 0.

90° pulse phase: 0°
 180° pulse phase: 180° for Alternating 180° pulse phase

$$90^\circ_x - 180^\circ_y - 180^\circ_y - 180^\circ_y - 180^\circ_y$$

6. Relaxation Time Contrast

6.1. Objective

The object of this experiment is to determine the relationship between the concentration of the paramagnetic contrast agent, CuSO₄, in a water sample and the characteristic relaxation times of the ¹H NMR signal.

6.2. Apparatus

This experiment will use the Terranova-MRI EFNMR instrument, consisting of a three-coil probe, a spectrometer and a controlling PC. The experiments will be run from the *Prospa* software. The samples used will be a collection of large sample bottles containing 500 ml of distilled water and concentrations of CuSO₄ ranging from 250 μM to 4000 μM. A large bottle of tap water will also be required for instrument setup.

6.3. Background Theory

6.3.1. Contrast in NMR Imaging

Contrast in an NMR image provides a means of distinguishing between regions within a sample. Positive contrast means that the signal intensity is higher in the region of interest. Negative contrast means that the signal is reduced in the region of interest.

The magnitude of the data in an NMR image is a record of the number of spins in a given region of the sample. Therefore NMR images naturally display contrast between regions with different spin densities. While this is very useful in some applications, there are many situations where the regions of interest within the sample contain similar or identical spin densities. Therefore alternate contrast mechanisms need to be introduced into the imaging experiment to distinguish between these homogeneous regions.

Contrast can be introduced into an image by adapting the NMR experiment to exploit differences in the NMR properties of the sample, such as relaxation times. These properties can be exploited to achieve contrast in the image by either highlighting existing differences between the regions of interest in the sample, or by introducing differences between otherwise homogeneous regions through the addition of a contrast agent.

A contrast agent is a substance that alters, in a well-defined way, the NMR properties of spins in contact with the agent. Contrast agents can augment the sensitivity and/or specificity of an NMR imaging experiment. Some contrast agents create positive contrast and others generate negative contrast; however, many contrast agents can create either positive or negative contrast depending on the chosen NMR experiment.

6.3.2. Paramagnetic Relaxation Time Contrast Agents

There are many types of contrast agents employed in NMR imaging. Paramagnetic relaxation time contrast agents are an example of one class of contrast agents. Paramagnetic contrast agents possess unpaired electrons and have a positive magnetic susceptibility. The local magnetic fields generated by the paramagnetic contrast agent effectively decrease the T_1 and T_2 relaxation times of neighbouring spins. In general a decrease in T_2 will result in a decrease in the signal magnitude whereas a decrease in T_1 results in an increase in the signal. For low concentrations of the contrast agent the T_1 effects typically dominate to generate positive contrast. Larger concentrations of the contrast agent will typically result in negative contrast. The extent of positive or negative contrast can be controlled, to a certain extent, through adjustment of the various NMR imaging experiment parameters.

6.4. Procedure

6.4.1. Getting Started

Run through the setup procedures from Experiments 1 and 2 to acquire a good quality FID from a large tap water sample. This process should include:

- Shimming.
- Tuning the probe.
- Setting the B_1 frequency to the Larmor frequency of the sample.
- Determining the length of the 90° and 180° pulses.

6.4.2. ^1H NMR of H_2O doped with CuSO_4

In this section of the experiment the effects of the contrast agent, CuSO_4 , on ^1H NMR of H_2O will be explored. For this portion of the experiment you will require a large sample of water and a large sample of water doped with approximately $3000 \mu\text{M}$ (i.e. $3.0 \times 10^{-3} \text{ mol/l}$) of CuSO_4 .

Open the PulseAndCollect experiment from under the EFNMR menu. Use all of the parameters chosen during the instrument setup. Acquire an FID and spectrum from the pure water sample. Integrate the sample peak in the spectrum. Record the limits of the integration and the results. Repeat the experiment with a short polarisation time of 500 ms. Integrate the spectrum over the same frequency range as previously used. Record the results. How do the integrals compare? Can you explain the difference?

Replace the pure water sample with the doped water sample. Acquire an FID with the parameters chosen during the instrument setup. Integrate the spectrum over the same frequency range as used previously for the pure water sample. Record the result. Repeat the FID experiment with the short 500 ms polarisation time and integrate the spectral peak. Record the result. How does this result compare with the other integral results? How does adding CuSO_4 to the water sample change the signal observed at a short polarisation time? Why do you think this is the case?

6.4.3. Relaxation Time Measurement of CuSO_4 solutions

In this part of the experiment we will do some quantitative measurements of relaxation times in order to better understand the effects of the contrast agent CuSO_4 on the ^1H NMR signal.

Prepare at least 5 samples of CuSO_4 dissolved in 500 ml of distilled water. Choose concentrations of CuSO_4 between $250 \mu\text{M}$ and $5000 \mu\text{M}$ such that they are approximately equally spaced on a logarithmic plot.

For each sample, measure the T_2 and T_1 relaxation times. (In the latter case in both the polarising field and the Earth's field). For directions on how to do this, refer to Experiments 3 to 5. Record the relaxation times for each sample.

Plot the concentration of CuSO_4 against relaxation time on a log-log plot. Do a separate plot for each of the three relaxation times. What relationship do you find between concentration of the contrast agent and the relaxation time of the sample for T_2 , $T_1(B_p)$ and $T_1(B_E)$?

Consider these results. How can they be used to explain the effects observed in section 6.4.2?

In what way will the change in T_1 and T_2 , observed when more and more contrast agent is added to the water sample, affect the observed signal? How could the experiment be modified to highlight the changes in T_1 and T_2 ?

6.5. Further Questions

1. What are some examples of contrast agents currently used in MRI? What are they used for?

2. What is a T_2 weighted image in MRI? What is a T_1 weighted image in MRI? What kind of contrast would a chemical such as CuSO_4 create in a T_2 weighted image? In a T_1 weighted image?

6.6. Appendix for the Instructor

The object of this experiment is to introduce the idea of contrast agents to the student and to have the student observe the effects of a paramagnetic contrast agent, CuSO_4 . Copper (II) Sulphate (CAS = 7758-98-7; EC-No. = 231-847-6; HS Code = 2833 25 00) is available from most chemical suppliers.

The goal of the experiment is for the student to understand that the paramagnetic contrast agent decreases both T_2 and T_1 . This experiment also aims to show the student how, with the right manipulation of the experimental parameters, the decrease in T_1 can actually result in an increase in signal despite the corresponding decrease in T_2 .

In the first section of the experiment the student should observe that for a pure water sample, decreasing the polarisation time from 4 s to 500 ms significantly decreases the observed signal. However, in the case of the water sample doped with CuSO_4 , the student should observe that the signal does not decrease substantially between the long and the short polarising time. At the short polarisation time it should be observed that the signal from the doped sample is in fact much greater than that of the pure water sample. This effect is explained by the fact that the contrast agent (CuSO_4) shortens T_1 in the doped water sample. Therefore, the doped water sample becomes polarised much more quickly and so requires a much shorter polarisation time to become fully polarised.

The student should also observe a T_2 effect between the pure water and the doped water samples. In the case of pure water, the FID signal should be observed to persist for much longer than the FID acquired from the doped water sample. This implies that the contrast agent decreases T_2 in addition to decreasing T_1 .

Table 6.1 Sample results for relaxation time measurements of water samples doped with CuSO_4

Sample	$[\text{CuSO}_4]$ (μM)	$T_1 - B_p$ (s)	$T_1 - B_E$ (s)	T_2 (s)
1	4016	0.184 ± 0.008	0.149 ± 0.002	0.18 ± 0.03
2	1992	0.41 ± 0.02	0.347 ± 0.005	0.39 ± 0.03
3	996.2	0.56 ± 0.03	0.485 ± 0.006	0.56 ± 0.03
4	543.8	0.83 ± 0.02	0.73 ± 0.01	0.79 ± 0.01
5	250.6	1.68 ± 0.03	1.43 ± 0.02	1.39 ± 0.01

In the second portion of this experiment the student is asked to measure the relaxation times for a range of water samples doped with CuSO_4 . The suggested CuSO_4 concentrations are 250 μM , 500 μM , 1000 μM , 2000 μM and 4000 μM . The student should find a linear relationship between the concentration of CuSO_4 and the relaxation times on a log-log plot. (See sample results in Table 6.1, Figure 6.1, Figure 6.2 and Figure 6.3). Both T_1 and T_2 decrease as the concentration of CuSO_4 increases. This finding is consistent with the results observed in the first section of this experiment.

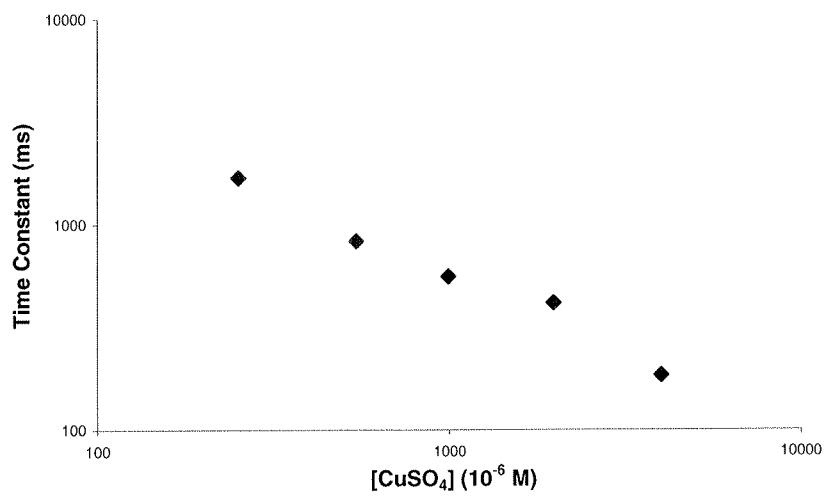


Figure 6.1 A log-log plot of T_1 (in B_p) as a function of the concentration of CuSO_4 .

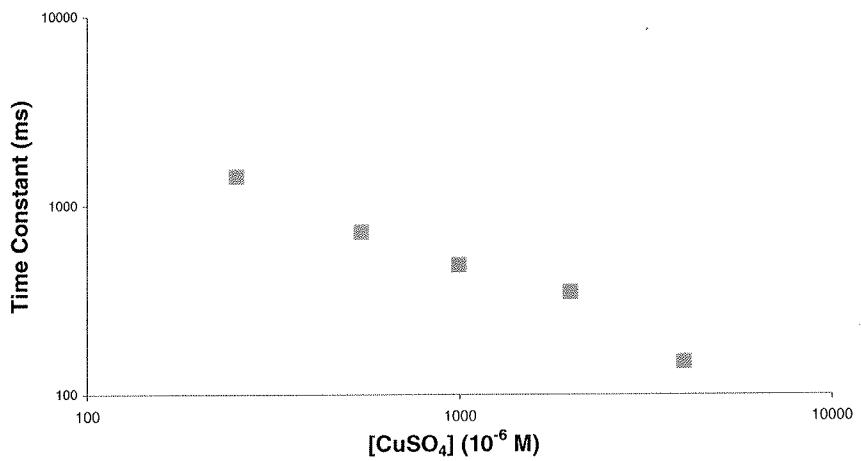


Figure 6.2 A log-log plot of T_1 (in B_E) as a function of the concentration of CuSO_4 .

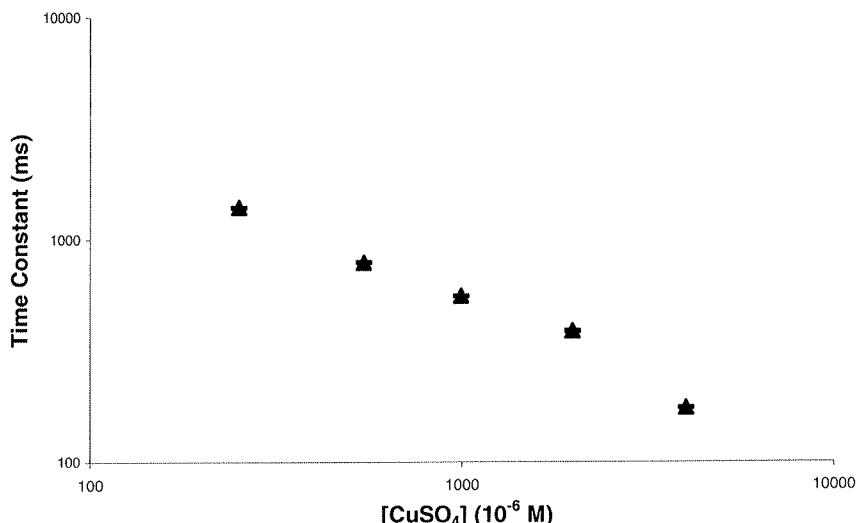


Figure 6.3 A log-log plot of T_2 as a function of the concentration of CuSO_4 .