Nuclear Magnetic Resonance F61/F62

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Prerequisites:

The knowledge of the following physical concepts is required for F61/F62:

- Classical mechanics: angular momentum, torque
- Electrodynamics: Magnetic dipole, interaction magnetic dipole with magnetic field, magnetization, precession, Larmor frequency, Helmholtz coils, Biot-Savart law
- Thermodynamics: Fermi-Dirac statistic, Boltzmann statistic
- Quantum mechanics: Perturbation theory

The physics lab experiment F61/F62 is organized into three parts. The measurements of the first two parts are carried out with a Bruker minispec p20 apparatus whereas the third part is measured with a newer Bruker minispec mq7.5 model. The experimental program of F61 and F62 is identical. Due to the organization of this lab experiment into three parts with two Nuclear Magnetic Resonance analyzers, F61 starts monday whereas the starting day of F62 is wednesday.

The three parts of this experiment are the following:

• Measurement of relaxation times

The spin-lattice relaxation time T_1 and the spin-spin relaxation time T_2 are measured. The spin-spin relaxation time is measured by the spin echo as well as by the Carr-Purcell method.

• Measurement of chemical shift

The chemical shift of protons is measured and used to identify five different substances.

• Imaging measurements in one and two dimensions

Nuclear Magnetic Resonance techniques are used for imaging measurements in one and two dimensions. The flow of oil in sand is measured as a time dependent process in one dimension. Two dimensional measurements are carried out to map the inner volume of substances which contain oil or water.

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Printer utilities

The display of the data is done with LabVIEW in all three parts of the experiment. Two printers are available for printing the LabVIEW front panels:

- fplaserjet: This black and white printer is located in the same room.
- schiele (og2): This printer is a color printer and is located at the other end of the hallway in the small room with the copy machine.

The LabVIEW programs display the data on graphics panels. You can save the display of a panel in encapsulated postscript format (eps) in the following way:

- right click on the panel which you want to save
- click on "Export Simplified Image", left click "Encapsulated Postscript (.eps)"
- left click on the folder symbol next to the empty string field, find the folder where you want to save the file and specify the file name, click "OK".
- click "Save", the file now exists in the specified folder. You can view the contents of the file by double clicking on it.

1 Basics of Nuclear Magnetic Resonance

Nuclear Magnetic Resonance (NMR) techniques are based on the fact that nuclei with spin J unequal zero possess a magnetic dipole moment

$$\vec{\mu} = \hbar \gamma \vec{J} \tag{1}$$

The quantity γ is called the gyromagnetic ratio of the nucleus. In the following, we restrict the discussion to the case of protons for which

$$\gamma_{Proton} = 2.6752 \cdot 10^8 sec^{-1} Tesla^{-1} \tag{2}$$

The magnetic dipole $\vec{\mu}$ interacts with an external magnetic field $\vec{B_0}$

$$\Delta E = -\vec{\mu} \cdot \vec{B_0} \tag{3}$$

This interaction results in a parallel μ_+ or antiparallel μ_- orientation of the proton magnetic dipole in the external field $\vec{B_0}$.



Figure 1: Proton magnetic dipole moment in external field B_0 .

A macroscopic sample containing N protons will populate the parallel and antiparallel state with occupation numbers N_+ and N_- where $N = N_+ + N_-$. The occupation numbers N_+ and N_- follow Dirac statistics, but can here be approximated by a Boltzmann distribution

$$N_{\pm} = N_0 e^{-\frac{E_0 \pm \Delta E}{kt}} \tag{4}$$

Here, N_0 is a normalization factor. The parallel state is energetically favorable, hence $N_+ > N_-$. The surplus of protons in the parallel state leads to a macroscopic magnetization \vec{M} .

The magnetization can be calculated as the sum of magnetic dipole moments per unit volume:

$$\vec{M} = \frac{1}{V} \sum_{i} \vec{\mu_{i}} = \frac{1}{V} (N_{+} - N_{-}) |\vec{\mu}| \vec{e_{z}}$$
(5)



Figure 2: Ground state magnetization \vec{M} along $\vec{B_0}$.

This sum can be evaluated and yields

$$\vec{M} = \frac{\mu N}{V} \sinh(\mu B/kT) \,\vec{e_z} \tag{6}$$

For weak fields ($\mu B \ll kT$), which is the case here, the sinh function (sinus hyperbolicus) can be expanded and the law of Curie follows:

$$\vec{M} = \frac{N}{V} \frac{\hbar^2 \gamma^2 I(I+1)}{3kT} \vec{B_0} \sim \vec{B_0}/T$$
(7)

The macroscopic state characterized by a magnetization \vec{M} parallel to the external magnetic field \vec{B}_0 as shown above minimizes the energy.

In general, the magnetization can have an arbitrary direction relative to the external field.



Figure 3: Magnetization M_{\parallel} and M_{\perp} .

The magnetization \vec{M} shown in Fig. 3 represents a macroscopic state different from the ground state. This state will therefore dissipate its excitation energy and reach the ground state asymptotically on a characteristic time scale. For further discussion, the magnetization \vec{M} is decomposed into components M_{\parallel} parallel and M_{\perp} perpendicular to the external field. In particular, M_{\parallel} can be antiparallel to the external $\vec{B_0}$ field.

The interaction between the external magnetic field \vec{B}_0 and the magnetization \vec{M} as expressed by eq. 3 results in a torque $\vec{\tau}$ acting on the magnetization.

$$\vec{\tau} = \vec{M} \wedge \vec{B_0} \tag{8}$$

Inserting the components M_{\parallel} and M_{\perp} into eq. 8 results in the torque acting only on M_{\perp} . Without relaxation processes, the rate of change of M_{\perp} is given by

$$\frac{d\vec{M_{\perp}}}{dt} = -\gamma \vec{M_{\perp}} \wedge \vec{B_0} \tag{9}$$

The quantity $\frac{d\vec{M_{\perp}}}{dt}$ is perpendicular to $\vec{M_{\perp}}$ and $\vec{B_0}$, hence it follows that $\vec{M_{\perp}}$ precesses around $\vec{B_0}$. The angular frequency ω is called the Larmor frequency ω_L .



Figure 4: Precession of M_{\perp} around $\vec{B_0}$.

By inserting an Ansatz $\vec{M} = (M_{\perp} cos(\omega_L t), M_{\perp} sin(\omega_L t), 0)$ into eq. 9, the Larmor frequency ω_L is deduced as

$$\omega_L = \gamma B_0 \tag{10}$$

A magnetization M_{\perp} perpendicular or M_{\parallel} antiparallel to $\vec{B_0}$ can be generated by applying a high frequency pulse to the ground state magnetization \vec{M} .



Figure 5: High frequency pulse along x-direction.

Consider the situation as shown in Fig. 5 above. The external static magnetic field \vec{B}_0 is pointing in z-direction. A coil is oriented along the x-direction. Applying a sinusoidal voltage of frequency ω_{HF} on the coil results in a solenoidal magnetic field

 $\vec{B_1}$ which is longitudinally polarized along the x-direction. Setting up the equations of motion it can be shown that the ground state magnetization \vec{M} precesses around the x-axis during the high frequency pulse. During a time interval Δt the angle α of precession is given by

$$\alpha = \gamma B_1 \Delta t \tag{11}$$

If the time interval is chosen such that $\alpha = 90^{\circ}$, then the ground state magnetization \vec{M} is rotated into a perpendicular component M_{\perp} along the y-axis. Such a pulse is called a 90° pulse. Similarly, the time interval of the high frequency pulse can be chosen such that the ground state magnetization \vec{M} is rotated by $\alpha = 180^{\circ}$. The resulting magnetization is then antiparallel to the static field \vec{B}_{0} .

2 NMR Signal

2.1 Hardware

Make yourself familiar with the hardware by reading Appendix A and B. Remove the styrofoam shielding of the magnet and have a look inside.

2.2 Signal generation

The probes are located within the volume of the coil shown in Fig. 5 when the glass tubes are inserted into the magnet (see Fig. 14 of Appendix B). The high frequency pulses of frequency ω_{HF} are generated in the electronic unit shown and explained in Appendix A. A transverse magnetization M_{\perp} precesses around $\vec{B_0}$ with the Larmor frequency ω_L . The difference between ω_{HF} and ω_L is on the order of a few hundred Hertz and is called the working frequency. Note that ω_{HF} is of fixed value whereas the Larmor frequency ω_L can be changed by turning the screw W1 of the magnet as explained in Appendix B. Due to the precession of M_{\perp} , the magnetic flux through the coil is modulated by the Larmor frequency. The coil therefore generates an induction signal of this frequency which is fed back to the electronic unit. This coil signal is subsequently mixed with the high frequency signal ω_{HF} . The mixing generates two signals, one with a frequency of the sum and one with the frequency of the difference, ie the working frequency. The corresponding signal of the working frequency is available for analysis.

2.3 Calibration high frequency pulses

Before you can start with the measurements you need to define Puls I and Puls II as 90° and 180° pulse, respectively.

Find the box with the label duplexer which is sitting on the table above the electronics unit. This duplexer box has three connections:

• Signal in/Puls out: This cable connects the duplexer box with the connector C illustrated in the Appendix B of the magnet.

- **Signal out**: This cable transmits the signal induced in the coil back to the electronic unit.
- Puls in: This connector takes the high frequency pulse of the electronic unit.

Proceed in the following order to see the high frequency pulses on the oscilloscope:

- Make sure that the electronic unit does not produce any pulses. The OPER-ATE button of S1 has to be OUT and the S2 switch has to be in OFF mode (see Appendix A for meaning of S1 and S2).
- Find the attenuator box with label ATT U1 on the **Puls in** connector of the duplexer box. Unplug the cable which connects this attenuator box with the electronic unit where the cable carries the label **Duplexer Puls in**. Find the box with label **ABSCHWAECHER 45,5 db**. Connect this box on the A side with the cable with label **Duplexer Puls in**. Take the cable on the B side of the 45.5 db attenuator and connect to the input of the oscilloscope.
- Generate pulses by pushing the OPERATE button into IN position and switch S2 into "*periodic*" mode. Select Puls I by pushing the Puls I button of the S1 switch. Generate 1000 pulses per second by pushing the S3 switch into the 1 ms position.
- Look at the high frequency pulse on the scope. Choose an appropriate time base and select the trigger mode by triggering on the input channel with positive slope and trigger threshold of about 100 mV. What is the modulation frequency of this pulse? Change the switch S6 to minimum value. Convince yourself that the pulse duration is now minimum. Increase switch S6 slowly. Check on the scope that the pulse duration increases correspondingly.
- Repeat the same for Puls II. Select Puls II by pushing the Puls II button of the switch S1. Check the pulse duration by adjusting the S7 switch.
- Disable pulse generation by pushing the OPERATE button of S1 into OUT position and the S2 switch into OFF position.
- Reconnect the cables properly.

In order to define the 90^0 and 180^0 pulses proceed in the following order:

- Insert probe 1 into the magnet.
- Select Puls I on the S1 switch.
- Select one pulse every 3 seconds by an appropriate setting of the S3 switch.
- Generate pulses by pushing the OPERATE button of S1 into IN position and the S2 switch into "periodic" mode. Look at the signal on the scope. You expect a signal which is modulated by the working frequency ν_w . Choose a time base for a working frequency of $\nu_w = 1$ kHz and select trigger mode external. Trigger on negative edge with a threshold of about -50 mV. Adjust the working frequency to 1 kHz by turning the screw W1 explained in Appendix B of the magnet.

- Increase the pulse duration of Puls I by increasing the value of switch S6. Watch the amplitude of the signal on the scope. When is Puls I a 90⁰ pulse ?
- Repeat the same procedure for Puls II. Change the pulse length of Puls II by varying the switch S7. When is Puls II a 180⁰ pulse ?

3 Part I: Relaxation time

3.1 Bloch equations

The Bloch equations describe the time evolution of the magnetization subject to relaxation. For further discussion, we introduce the rotating frame of the transverse magnetization. In this frame, the transverse magnetization is constant if no relaxation processes take place. The magnetization shown in Fig. 3 contains such transverse magnetization, and the time evolution in the rotating frame is defined by

$$\frac{dM_{\perp}(t)}{dt} = -\frac{M_{\perp}(t)}{T_2} \tag{12}$$

$$\frac{dM_{\parallel}(t)}{dt} = -\frac{M_{\parallel}(t) - M_0}{T_1}$$
(13)

These equations assume that the time evolution is dominated by a restoring force which is proportional to the deflection from equilibrium. The constant T_2 in eq. 12 is the spin-spin relaxation time whereas T_1 in eq. 13 is the spin-lattice relaxation time. M_0 denotes the ground state magnetization.

The time evolution of the magnetization \vec{M} in the laboratory system is related to the time evolution of magnetization $\vec{M^{rot}}$ in the rotating system by

$$\frac{d\vec{M}}{dt} = \frac{\partial \vec{M^{rot}}}{\partial t} + \vec{\omega} \wedge \vec{M} \tag{14}$$

The Bloch equations in the laboratory system are hence written in the following way

$$\frac{dM_{\perp}(t)}{dt} = \gamma (\vec{B} \wedge \vec{M})_{\perp} - \frac{M_{\perp}(t)}{T_2}$$
(15)

$$\frac{dM_{\parallel}(t)}{dt} = \gamma (\vec{B} \wedge \vec{M})_{\parallel} - \frac{M_{\parallel}(t) - M_0}{T_1}$$
(16)

3.2 Spin-spin relaxation T_2 by spin echo method

A transverse magnetization M_{\perp} represents a state which contains excitation energy. First, the interaction of the magnetic dipoles with the external magnetic field contributes according to eq. 3. This term is called the spin-lattice contribution. Second, the interaction energy of two magnetic dipoles depends on the orientation of the dipoles. The antiparallel orientation is energetically favorable. A transverse

magnetization hence contains the corresponding interaction energy when many magnetic dipoles are oriented parallel to each other. This term is called the spin-spin contribution.

The time evolution of transverse magnetization is determined by the solution of eq. 12 and is given by

$$M_{\perp}(t) = M_{\perp}^0 e^{-\frac{t}{T_2}}$$
(17)

A 90⁰ pulse will create a transverse magnetization M_{\perp} in y-direction. After the 90⁰ pulse the transverse magnetization will precess around the static field $\vec{B_0}$ with a Larmor frequency ω_L defined by eq. 10. Due to the inhomogeneity of the field, protons at different positions in the probe will precess with different Larmor frequencies. As a consequence, a phase difference between the protons will develop. This phase difference results in a dephasing of the transverse magnetization on a time scale much shorter than the relaxation time T₂. A 180⁰ pulse reverts the phases and can be used for correction.



Figure 6: Dephasing of transverse magnetization M_{\perp} .

Consider the pulse sequence 90^{0} - 180^{0} as illustrated in Fig. 6. A 90^{0} pulse at time t=0 generates a transverse magnetization M_{\perp} along the y-axis. Protons at different locations of the magnetic field will have slightly different Larmor frequencies due to the inhomogeneity of the field. Assume that the magnetic field is larger at location A than location B, hence $\omega_{A} > \omega_{B}$. Protons at location A precess with frequency ω_{A} whereas protons at location B precess with frequency ω_{B} in clockwise fashion. At time t= τ the protons at locations A and B have acquired a phase difference. The protons at location A are ahead of the protons at location B as illustrated in the

upper right corner. At time $t=\tau$ a 180^{0} pulse is applied. The effect of this pulse is to rotate the magnetic dipoles by 180^{0} around the x-axis. The dipole orientation of the protons at locations A and B is reversed after this 180^{0} pulse. The protons at location B are now ahead of the protons at location A as shown in the lower right corner. The time duration of the 180^{0} pulse is short as compared to the time τ and hence can be neglected. The protons continue to precess clockwise after the 180^{0} pulse. After another time interval τ , the protons at A and B are in phase again as illustrated in the lower left corner. All the magnetic dipoles in phase build up the magnetization M_{\perp} at time $t=2\tau$.

Select a pulse sequence I-II on pushbutton S1 and $\tau=10$ ms on switch S4 and a repetition rate of 1/3 Hz on S3. Insert probe 1 into the magnet and activate the pulse sequence. Watch the signal on the scope.



Figure 7: Pulse sequence 90^{0} - 180^{0} with $\tau = 10$ ms.

Fig. 7 displays the NMR signal of pulse sequence I-II with $\tau=10$ ms. Visible at early times t<5 ms is the decay of the transverse magnetization due to loss of coherence as explained above. At later times the spin echo builds up and reaches its maximum at time t= 2τ . An evaluation of the maximum of the spin echo envelope at the maximum is hence a measurement of the decay curve as defined by eq. 17 at time t= 2τ . This decay curve can be measured at different times by varying the parameter τ in the pulse sequence I-II.

The spin echo method as described above has limitations when measuring the decay curve at large times. For such measurements, 2τ is large hence also τ is large. The protons can move to different locations due to molecular diffusion before the 180° pulse is applied. The average Larmor frequencies in the time intervals $0 < t < \tau$ and $\tau < t < 2\tau$ can hence be different due to field inhomogeneities. The magnetic dipoles are in such case only approximately aligned along the negative y-axis at time t= 2τ as shown in Fig. 6 and hence only partial coherence is achieved. This limitation leads to reduced signals at large times with a correspondingly reduced value for the relaxation time T₂.

3.3 Spin-spin relaxation T_2 by Carr-Purcell sequence

The Carr-Purcell sequence minimizes the effects of molecular diffusion and field inhomogeneities and hence improves considerably the limitation of the spin echo method as described above. The Carr-Purcell sequence starts with a 90⁰ pulse to generate a transverse magnetization. A 180⁰ pulse is applied for correction after a small time interval τ . At time t=2 τ the system will be phase coherent. The system then starts to loose the coherence again due to field inhomogeneities. At time t=3 τ another 180⁰ pulse is applied hence the system is phase coherent at time t=4 τ . This pattern of phase coherence at even multiples of τ and generation of 180⁰ pulses at odd multiples repeats to large times as shown in Fig. 8. A measurement of the spin-spin relaxation time by the Carr-Purcell method hence yields a value which is closer to the true value of the system as compared to the measurement with the spin echo method.



Figure 8: Carr-Purcell sequence with $\tau = 10$ ms.

3.4 Spin-lattice relaxation T_1

The spin lattice measurement starts with the generation of a 180^0 pulse. Such a pulse produces a magnetization antiparallel to the external $\vec{B_0}$ field. This magnetization will undergo a time evolution as described by the solution of eq. 13

$$M_{\parallel}(t) = M_0 (1 - 2 e^{-t/T_1}) \tag{18}$$

The longitudinal magnetization $M_{\parallel}(t)$ does not produce a signal. The generation of a 90⁰ pulse at time t will, however, transform $M_{\parallel}(t)$ into a transverse magnetization which will induce a signal in the coil.

The signal from a pulse sequence $180^{0}-90^{0}$ with a value $\tau=20$ ms is shown in Fig. 9. The envelope of the signal at time $t=\tau$ is proportional to the longitudinal magnetization M_{\parallel} at time $t=\tau$. The decay curve of the longitudinal magnetization $M_{\parallel}(t)$ can hence be measured at different times by varying the value of τ in the pulse sequence $180^{0}-90^{0}$.



Figure 9: Pulse sequence $180^{\circ}-90^{\circ}$ with $\tau=20$ ms.

3.5 Measurements of relaxation times

3.5.1 Measurement T_2 by spin echo method

Measure about 8-12 points of the decay curve given by eq. 17 by using the pulse sequence I-II and by choosing appropriate τ values. For these measurements use the LabVIEW application "*T2_meas.vi*". You can find this program by clicking on the link "*LabVIEW day 1,2*" on the desktop of the PC.

The information of this measurement is stored in a file. Find the link to the folder "ss10" on the desktop of the PC. Here, "ss10" denotes summer semester 2010. For later semesters corresponding folders will exist. In this folder, create a new folder and rename this folder by your starting day of the experiment. Specify this folder in the "folder" field of the vi and choose a name for the data file of the measurement. The analog signal will be sampled with the scanning frequency. A scanning frequency of 10 kHz results in 10 data points per oscillation period of the signal which has a frequency 1 kHz. Run the application.

This application contains the following subprograms:

1. Single measurement:

Enter the number of data points to be recorded (10 points per ms of data acquisition time). Start this subprogram by clicking on "OK". The data points are displayed on the left. Enter the spin echo time in the field "spin echo time" and a time value of 6 in the field "del T(+-)". Run the subprogram again. On the right, the data points within an interval \pm del T around the spin echo time are displayed. The difference between maximum and minimum value within the time interval specified are calculated and shown above the plot. Proceed to many measurements if the parameters are set correctly such that the spin echo is centered in the display on the right.

2. Many measurements:

The signal to noise ratio can be improved by averaging over many measurements. Enter 10 in the field "number", then click "OK". The program displays the average and an associated error when the 10 measurements are made.

3. Write data to file, update plot:

Write this data point to the file by clicking "OK". The program shows the updated content of the file and the updated plot. You might have to rescale the horizontal axis of the plot for displaying all the data points.

4. Don't write data, update plot, no fit:

If a data point has been entered erroneously, then the information of this data point can be erased by editing the file and by removing the corresponding line. Click "OK" for updating the plot and the display of the file content.

5. **Fit**:

Click "OK" for fitting the relaxation time when you have measured all the data points. The fit value of T_2 and an associated error are shown.

3.5.2 Measurement T_2 by Carr-Purcell sequence

The measurement of the T_2 relaxation time by the Carr Purcell sequence is done with the LabVIEW application "*CP_meas.vi*".

The program has two modes:

- single measurement: This mode is active when the button "single meas" in the top left corner is selected and highlighted in green. In this mode, one measurement is made. Choose the parameter τ for the Carr-Purcell sequence such that about 8-10 spin echos are visible. Enter a scanning frequency of 10 kHz and define the number of data points such that all spin echos are measured. Enter the location of the spin echos in the bottom panel in the column "peak". Enter the number of peaks above the column.
- many measurements: This mode is active when the button "single meas" is not highlighted in green. The signal to noise ratio can be improved by averaging over many measurements. Enter 10 in the field "number of measurements" and run the vi. The program now takes 10 measurements and shows the average value with an associated error for each spin echo. After the 10 measurements the data points are plotted and the fit value of the T_2 relaxation time is shown with an associated error. You might have to rescale the axis of the plot for displaying all the data points.

3.5.3 Measurement T_1

The measurement of the T_1 relaxation time is done by "*T1_meas.vi*". This vi follows the same structure as the one for the T_2 measurement by the spin echo method ("*T2_meas.vi*") except that the fitting routine is now defined by eq. 18.

3.6 Systematics of relaxation times

Proceed with the measurements of the relaxation time T_2 by spin echo and Carr-Purcell sequence and relaxation time T_1 for both probes 1 and 3. Write your measurements with errors in a table and discuss the systematics of your results.

4 Part II: Chemical shift

The Larmor frequency ω_L is given by eq. 10. If protons are bound in a molecule then the total magnetic field $\vec{B_{tot}}$ determines the Larmor frequency. This total field consists of the external field $\vec{B_0}$ and a contribution $\delta \vec{B}$ due to the electron orbitals.

$$\delta \vec{B} = -\sigma \vec{B_0} \tag{19}$$

The contribution $\delta \vec{B}$ is proportional to the external field $\vec{B_0}$. The proportionality factor σ represents the magnetic shielding of the external field $\vec{B_0}$. This shielding factor characterizes the molecule and each nucleus within the molecule. If the modified Larmor frequency is measured then this shielding factor can be determined.

$$\omega_i = \omega_L (1 - \sigma_i) \tag{20}$$

Here, ω_L represents the free Larmor frequency and ω_i the frequency modified by the chemical shift.

For identifying substances by the chemical shift, a reference substance is added.



Figure 10: Chemical shifts δ_i of compounds relative to TMS.

In Fig. 10, the proton chemical shift in organic compounds relative to the reference substance Tetra-Methyl-Silan (TMS) is shown in units ppm (part per million).

$$\delta_i = \sigma_i - \sigma_{TMS} = \frac{\omega_L - \omega_i}{\omega_L} - \frac{\omega_L - \omega_{TMS}}{\omega_L} = \frac{\omega_{TMS} - \omega_i}{\omega_L}$$
(21)

4.1 Measurement chemical shift

Measure the chemical shift in the five probes A,B,C,D and E. Associate each probe with one of the substances listed in Fig. 11.



Figure 11: Five substances for identification.

Each probe exists with a label '+', for example there is a probe A and a probe A+. The label '+' signifies that the reference substance TMS is contained in the probe. The Larmor frequency of the reference substance TMS can be identified by comparing the measurement of the probe with the measurement of the probe plus reference. The evaluation of the Larmor frequency relative to the reference gives the shielding factor δ_i expressed in eq. 21.

Use the measured chemical shifts δ_i to identify the five substances of Fig. 11 based on the information listed in Fig. 10. Toluol and p-xylol have the same chemical shifts. Use the intensity of the measured frequency response to separate these two substances.

Use the LabVIEW application " $F61_scan_2_5Hz.vi$ " to measure the frequency response. This program has two modes:

- Messen: This mode is active when the boolean switch "Messen" is selected and highlighted in green. In this mode, the signal is sampled during some time interval and the measured time signal is displayed in the topmost panel. The Fourier transformation of the time signal is calculated in steps of 2 Hz and displayed in the second panel from the top. In addition, the information of the Fourier transform is saved in a file. The location and the name of this file have to be specified in the top right corner of the vi. In general, it is good practice to choose a file name which contains the information of the measured probe. Run the vi. You can check the information of the data file by opening it with a text editor. The file contains two columns. The first column represents the frequency values in steps of 2 Hz and the second column the corresponding intensity in arbitrary units.
- **Peak fit**: This mode is active when the boolean switch "*Peak fit*" is selected and highlighted in green. In this mode, the information in the data file is read

back and the intensity maxima can be fitted with a Gaussian fit. With these fits, the position of the peaks can be determined much more accurately than the discrete Fourier step of 2 Hz. The information of the data file is displayed in the bottom panel. Select a window where an intensity maximum is visible. Specify the low edge of the window in the field "W1-low" and the high edge in the field "W1-high". The values of the low and high edge have to be entered as multiples of 2 Hz. Run the vi. The location of the fitted maximum is displayed in the field "peak1 [Hz]". The units of this field are Hertz. The field "ppm1" displays the same information but in units of ppm.

A maximum of four peaks can be fitted simultaneously.

For the measurements of chemical shift, a working frequency of $\nu_w = 500$ Hz is needed. Check the working frequency on the oscilloscope and adjust if necessary. Select the 90⁰ pulse with a repetition rate of 1/3 Hz to generate the response of the probe. To get familiar with this measurement, insert probe 3 into the magnet. Make a measurement. You should see intensity in the Fourier transform at around 500 Hz.

- Over what interval is this intensity distributed ?
- What determines this width ?
- Use the pressure air as explained in Appendix B to put probe 3 into rotation. Repeat the measurement. Has the width of the frequency response changed ?

Proceed with the measurements by putting the probes A-E into the magnet. Use the pressure air for accurate measurements.

- Identify the substances.
- Do the peaks you measure correspond to what you expect ?

5 Part III: Imaging with NMR

The imaging measurements are made with the new Bruker NMR analyzer mq7.5. In order to carry out measurements which contain information on the position of the produced NMR signal, a magnetic field is necessary which has a position dependence. In practice, gradient fields are used for one, two and three dimensional imaging measurements. The gradient fields are superimposed to the static field \vec{B}_0 which defines the z-direction.

The gradient fields $\vec{B^x}, \vec{B^y}, \vec{B^z}$ are defined by the properties that the field vector points in z-direction and that the strength of the field is linearly increasing as function of the corresponding coordinate.

$$\vec{B}_{0} = (0, 0, B_{0})$$
$$\vec{B}^{x} = (0, 0, G^{x} \cdot x)$$
$$\vec{B}^{y} = (0, 0, G^{y} \cdot y)$$
$$\vec{B}^{z} = (0, 0, G^{z} \cdot z)$$

A gradient field can be produced by an arrangement of coils. For three dimensional imaging, three coil systems are needed which can switch the gradient fields $\vec{B^x}, \vec{B^y}, \vec{B^z}$ independently. In the Bruker NMR analyzer mq7.5, the three coil systems are mounted on an insert which is fixed by two screws in between the two pole pieces which define the static field $\vec{B_0}$.

Open the cover of the magnet unit and have a look inside. You will see the tip of one of the coil systems. On the desktop of the PC, you find a link "Bruker_pict". In the corresponding folder you find some pictures of the coil insert which carries the three coil systems.

5.1 One dimensional imaging

In one dimensional imaging, the position dependence of the NMR signal is generated by applying a gradient field which has a dependence in the corresponding direction, and the Larmor frequency hence becomes position dependent. In the following, we discuss one dimensional imaging along the z-direction.

5.1.1 Frequency coding

In one dimensional imaging along the z-direction, a gradient field $\vec{B^z}$ is superimposed to the static field $\vec{B_0}$. The Larmor frequency hence becomes z-dependent

$$\omega_L(z) = \gamma (B_0 + G^z \cdot z) = \omega_L^0 + \omega_z \tag{22}$$

For further discussion, we consider the rotating frame of the transverse magnetization M_{\perp} . In this frame, M_{\perp} is given by eq. 17 and denoted by M_{\perp}^{rot} . The transverse magnetization at a given z-position precesses with a Larmor frequency defined by eq. 10 and can thus be written as

$$M_{\perp}(z,t) = M_{\perp}^{rot}(z,t) e^{i\gamma(B_0 + G^z z)t}$$
(23)

The produced NMR signal is defined by

$$S(t) \propto \int_{V} M_{\perp}(z,t) \ e^{-i\omega_{HF}t} dV = \int_{V} M_{\perp}^{rot}(z,t) \ e^{i(\omega_{L}^{0}+\omega_{z}-\omega_{HF})t} dV$$
$$= e^{i\Omega t} \int_{z} \left[\int_{x} \int_{y} M_{\perp}^{rot}(z,t) dx dy \right] e^{i\omega_{z}t} dz$$
(24)

If the signal acquisition time T is much smaller than the relaxation time T_2 , then the transverse magnetization $M_{\perp}^{rot}(z,t)$ is constant during readout and can be set as $M_{\perp}^{rot}(z)$. The measured NMR signal S(t) is therefore, apart from the phase factor $e^{i\Omega t}$, the one dimensional Fourier transform of the transverse magnetization $M_{\perp}^{rot}(z)$. The quantity of interest, the magnetization $M_{\perp}^{rot}(z)$, can therefore be deduced by a one dimensional Fourier transformation of the measured NMR signal S(t).

In practice, the NMR signal S(t) is sampled at times $t_n = n\Delta t$ and hence yields a set of N data points

$$S_1 = S(\Delta t), \ S_2 = S(2\Delta t,), \dots, \ S_N = S(N\Delta t)$$
 (25)

A discrete Fourier transformation of the N data points S_N will result in N data points M_N defined by

$$M_1 = M_{\perp}(\Delta z), \ M_2 = M_{\perp}(2\Delta z), \dots, \ M_N = M_{\perp}(N\Delta z)$$
 (26)

with Δz defined by

$$\Delta z = \frac{Z}{N} = \frac{2\pi}{\gamma G^z N \Delta t} \tag{27}$$

Here, Z denotes the full range of the position measurement.

5.1.2 Phase coding

In the frequency coding approach described above, the NMR signal is measured as a function of time. During the readout time, the Larmor frequency at a given position z is determined by the applied gradient field, and the transverse magnetization precesses due to the gradient field by an amount

$$\Delta\phi(z) = \phi(z) - \phi(z=0) = (\gamma G^z z)t = \omega_z t \tag{28}$$

During the readout the position information is contained in the phase angle as described by eq. 28. The phase angle can, however, also be varied by applying a

gradient field which is of constant duration but whose strength is increased in steps of ΔG^z . This equivalent approach is called phase coding.

Phase coding is achieved by applying a gradient field $B^z = G^z z$ during a time interval T_{Ph} before readout of the NMR signal. The phase acquired at position z during this time interval T_{Ph} is given by

$$\varphi(z) = (\gamma G^z T_{Ph}) z = k_z z \tag{29}$$

The parameter $k_z = \gamma G^z T_{Ph}$ is the position frequency. After the phase coding gradient is switched off, all components of the transverse magnetization precess with the original Larmor frequency $\omega_L^0 = \gamma B_0$ but with a different phase angle which depends on the z-position. In phase coding, the different components all contribute to the NMR signal with the same frequencey but with different phases. The NMR signal is now defined by

$$S(k_x, t_0) \propto \int_V M_{\perp}(z, t_0) \ e^{-i\omega_{HF}t_0} dV = e^{i\Omega t_0} \int_z \left[\int_x \int_y M_{\perp}^{rot}(z, t_0) dx dy \right] e^{ik_z z} dz \quad (30)$$

The measurement of the phase coded NMR signal consists in making N measurements with different position frequencies $k_n = n\Delta k$ $(1 \le n \le N)$. As expressed in eq. 30, the phase coded NMR signal only needs to be measured at time t_0 . After the measurements, a set of N data points exists

$$S_1 = S(\Delta k, t_0), \ S_2 = S(2\Delta k, t_0), \dots, \ S_N = S(N\Delta k, t_0)$$
 (31)

As in frequency coding, the magnetization $M_{\perp}^{rot}(z)$ is deduced by a one dimensional discrete Fourier transformation of the measured data points S_N .

The two methods decribed above, frequency coding and phase coding, are mathematically equivalent. They differ, however, in the time required for the data acquisition. In frequency coding, the data points $S_n = S(t_n)$ as expressed in eq. 25 can be recorded in one sequence. In phase coding, N sequences are necessary to record the data points S_N as expressed in eq. 31.

5.1.3 One dimensional imaging measurements

The Bruker minispec mq7.5 is fully computer controlled by a graphical user interface. Your tutor will show you how to start the interface. The available application programs are listed in the top right corner of the interface in the folder "minispec Applications". Double click on the application "profile" for a one dimensional imaging measurement. A graphics window "profile.sig" will open which contains the display of the measurement.

To start the measurement, click on the blue icon "Measure" (bottom left corner). A menue "Application Configuration Table" will open. In this menue, the gradient directions are displayed on the left whereas the profile parameters are shown on the right. Choose the y-gradient for a measurement in vertical direction. The pulse parameters contain default values which in general don't need to be modified. Click on the "OK" button after you marked the box "y-gradient". The "profile" application is programmed such that a measurement is made every 10 seconds. This measurement is made when the timer displayed in the lower left corner has reached zero. Click on "STOP" for stopping the measurement. The data points of every measurement are stored in a data file. The file name contains a label which denotes the number of the measurement. These files "testProfilxx.txt" (xx = 1,2,3,..) are written into the folder "C:/minispec/imagingdata". You can find this folder by clicking on the link "minispec datafiles" on the desktop.

You have to save the data files of the measurements which you want to display. Click on the link on the desktop "Shortcut to ss10". Here, "ss10" denotes the summer semester 2010. For later semesters, a corresponding link will exist. In that folder, create your own folder and rename this folder to the day when you started the experiment. Copy the files you want to display to your own folder and rename the files such that you recognize the measurement by the file name.

5.1.4 One dimensional data display

A LabVIEW application "many_profiles.vi" is available for reading back data files for display. You can find this LabVIEW application by clicking on the link "LabVIEW day3" on the desktop. With the application "many_profiles.vi", you can superimpose many measurements by reading back the corresponding data files from your folder. In general, these files have a common prename, a label denoting the number of the measurement and a common postname. Enter the corresponding information in the front panel of "many_profiles.vi". The labels of the different files are entered in the box "file names". The strings "common prename", "file names" and "common postname" are concatenated for defining the full name of the file which is read. In addition you need to enter the number of files which should be read. If you want to read back only one file, then the string "file names" has to be empty such that the strings "common prename" and "common postname" concatenate to the full file name.

5.1.5 One dimensional measurements

Make the following one dimensional measurements in vertical direction::

- Take the glass tube which is filled with about 15 mm of oil. What signal do you expect? The ring on the glass tube should be positioned such that the bottom of the tube corresponds to the zero position of the measurement. If necessary, adjust the position by turning the ring with respect to the glass tube and move it into the correct position.
- Take the glass tube which is filled with about 50 mm of water. In this measurement, you can see the effects of the nonlinearity of the gradient field. From this measurement, estimate in which vertical range the probe has to be located for minimizing the effect of nonlinearity of the gradient field.

- Take the glass tube which contains the teflon structure immersed in oil. What signal do you expect? (Teflon material does not produce a NMR signal)
- Take an empty glass tube, fill it with about 15 mm of sand and add approximately 4 mm of oil on top. Put the glass tube into the magnet and measure until you cannot see any changes from one measurements to the next. Superimpose some of the measurements in the LabVIEW display in order to illustrate the time development of the system. Can this process of oil mixing with sand be understood as a diffusion process?

5.2 Two dimensional imaging

Two dimensional imaging measurements can be made by a two dimensional Fourier method. In this approach, a slice is first selected by an appropriate combination of gradient field and high frequency pulse. Within the slice, two dimensional position information is derived by a combination of frequency and phase coding.

5.2.1 Slice selection

A slice in z-direction is defined by the property that the z-coordinate of all the points within the slice satisfy $z_1 \leq z \leq z_2$. If a gradient field B^z is switched on, then every z-position is characterized by a Larmor frequency as defined by eq. 22. The positions z_1 and z_2 are characterized by frequencies ω_1 and ω_2 , respectively. If the high frequency pulse $F(\omega)$ contains only frequencies ω such that $\omega_1 \leq \omega \leq \omega_2$, then the slice $z_1 \leq z \leq z_2$ will be selectively excited. Such a pulse $F(\omega)$ corresponds to a sinc pulse in the time domain

$$f(t) \propto \frac{\sin(at)}{t} \tag{32}$$

After the sinc pulse of duration t_p , the slice $z_1 \leq z \leq z_2$ contains excitation energy. The Larmor frequency is different for subslices at different z-positions when the slice selection gradient is switched on. The different subslices are therefore out of phase after the high frequency pulse. This dephasing can be compensated by switching on the slice selection gradient with reversed polarity for a time interval $t_p/2$ after the high frequency pulse. This additional gradient is called the slice refocussing gradient.

5.2.2 Two dimensional Fourier method

Within the selected slice, the two dimensional position information can be derived by a combination of phase coding in x-direction and frequency coding in y-direction. The imaging sequence is repeated N times for different values of the phase coding gradient $G_n^x = n\Delta G^x$ $(1 \le n \le N)$. In each sequence, the NMR signal is read out at times t_m with the readout gradient G^y switched on. For each combination (k_n, t_m) of the parameters $k_n = n\gamma\Delta G^x T_{Ph}$ and $t_m = m\Delta t$ a data point $S(k_n, t_m)$ exists. From this matrix of $N \times M$ data points, the two dimensional matrix of the image can be derived by a two dimensional Fourier transformation.



Figure 12: Timing diagram for the two dimensional Fourier method.

Fig. 12 shows the timing diagram of the high frequency pulse and the slice selection, the phase and frequency coding gradient for the two dimensional Fourier method. The high frequency pulse is applied while the slice selection gradient is switched on. After this pulse, the slice refocussing gradient is switched on with reversed polarity. The phase and frequency coding gradients are then switched on to record the two dimensional matrix of data points.

5.2.3 Two dimensional imaging measurements

Select the application "msse" for the two dimensional measurements with the Fourier method. The parameters for such a measurement are listed in a Configuration Table. The Bruker user interface lists a row of icons at the top of the graphical window. The icon for the Configuration Table is located almost in the center of this row. Click on the icon, a graphical window "APPLICATION CONFIGURATION TABLE" will open. On the left, a column is listed with Imaging Options. Here, you have to define the orientation of the slice. The slice can be "Horizontal", "Vertical Back to Front" or "Vertical Left to Right". Choose the orientation by clicking on the corresponding box. On the right, the following Image Parameters are listed:

- *Field of View [mm]*: The physical dimension of the measurement. The glass tube has an outer diameter of about 24 mm, hence the measurement will show an edge of about 4 mm on the side if the Field of View is defined as 32 mm.
- *Image Size [points]*: This value defines the number of pixels in the slice and is set to 64. **Do not change this value**, the data points are stored as a 64x64 matrix.

- *Slice Thickness [mm]*: The thickness of the slice. For materials with a good NMR signal like oil the minimum slice thickness is 2 mm. For materials with a weak NMR signal the minimum slice thickness is larger. If the slice thickness is defined to be too thin, then the NMR signal is too weak and the program will not find valid data acquisition parameters.
- Center Slice Position [mm]: The location of the center of the slice along the gradient direction. For slices which are defined to be vertical, the center slice position zero is defined by the vertical axis of the glass tube. For a horizontal slice, the center slice position zero is defined by the height zero as shown in the one dimensional measurement made with the "profile" application.
- *Number of Slices*: This program can in principle record multiple slices simultaneously. The data format assumes that only one slice is recorded, hence this parameter has to be set to one.
- Inter Slice Distance [mm]: Only of relevance when multiple slices are recorded.
- Echo time [ms]: Set to 10 ms. Do not change this value.
- Repetition time [s]: Set to 0.5 s. Do not change this value.
- *Number of Averages*: The number of measurements over which the average is taken. Choose one measurement for checking whether the data acquisition parameters are set correctly. Choose more than one if you want to have a measurement with good contrast.

Click on the "OK" button in the bottom left corner when all the Image Parameters are set correctly. To start the measurement, click on the blue icon "*Measure*" in the bottom left corner of the minispec interface. A graphical window "*TEXT INPUT BOX*" will now open. Here, you have to define the location and the name of the file where the data points are stored. Enter your folder name and define the name of the file such that you recognize this measurement when reading back the file for display. Click "OK" when done. The program optimizes the slice refocussing gradient (see Fig. 12) and then goes through the cycles of data acquisition. The program will show "*Data exported*" when finished. You will now find two files in your folder. The file names contain some digits at the end added by the program with the information of the date and the time. Remove these digits for easier further processing of the files.

5.2.4 Two dimensional data display

A LabVIEW application "*nmr_2d.vi*" exists for displaying the data of your measurement. You can find this program by clicking on the link "*LabVIEW day3*" on the desktop. Enter your folder name and the file name which you want to display. You can print out the display by sending it to the color printer "schiele".

Storing the display in a graphics file is possible by a WINDOWS screen shot (press *"Print Scrn"* on the keypad) and by importing into a graphics program (Paint, "Ctrl-V").

5.2.5 Two dimensional measurements

Make the following measurements with the two dimensional Fourier method:

- Take the glass tube with the 15 mm oil and define a horizontal slice. What picture do you expect? When reading back the data file with the LabVIEW program, do you see what you expect? How does the image look when you define a vertical slice left to right? Do you see what you expect?
- Take a peanut with shell and put it into an empty glass tube. Choose the measurement parameters such that you can see the two peanuts within the shell. Take a peanut out of the shell. Adjust the measurement such that the air gap is visible in between the two halves of the peanut.
- Take a pecan nut and try to resolve the inner structure of this nut.
- Take a piece of celery and map the C-shaped form by using a horizontal slice.

6 NMR applications

NMR techniques are commonly used in many areas of basic and applied research as well as in industrial applications. A few of these applications are listed below and some links are given where further information is available.

- Spectroscopy of ³¹P: Nuclear Magnetic Resonance techniques can be applied for spectroscopy of nuclei with spin not equal to zero. Of particular interest is the spectroscopy of ³¹P due to the role of the adenosintriphosphat molecule (ATP) in the energy balance of living cells. Energy can be gained by transforming ATP into adenosindiphosphat (ADP). ATP contains three ³¹P nuclei whereas ADP contains only two. The ³¹P nuclei can be identified in these molecules by their chemical shift. For a particular application of ³¹P spectroscopy see http://www.nmr.uni-duesseldorf.de/main/menergie.html
- *Magnetic Resonance Imaging*: The application of NMR techniques in medical research or for medical diagnosis is known as Magnetic Resonance Imaging (MRI). YouTube lists a multitude of video clips of MRI pictures for the diagnosis of knee injuries and brain tumors (http://www.youtube.com).
- *Biomagnetism*: The Bio Imaging Laboratory at the University of Antwerpen concentrates its research activity on high resolution in vivo MRI. (http://webh01.ua.ac.be/biomag/)
- Surface quality control: Special hand held devices have been developed for measurements in the near surface volume, as is required for example in quality control for production processes of car tires. (http://www.brukeroptics.com/profiler.html)
- Digital fish library: The Digital Fish Library (DFL) at the University of California San Diego gives access to the three dimensional information of many fish species which have been scanned by NMR techniques. (http://www.digitalfishlibrary.org)

A Appendix A: Minispec p20 Electronic Unit

The electronic unit of the minispec p20 is connected to electrical power through a transformer. This transformer is located under the table. The power cable of the transformer should be disconnected from the power net when not in use. When disconnected, the transformer power cable is attached to a hook in the wall. Connect the transformer power cable to the power outlet, then switch on the transformer by pushing the button indicated by a red arrow with label ON/OFF (OFF is when button is OUT, ON is when button is IN). When you are finished with the measurement, proceed in reverse order: First push the ON/OFF button into OFF position, then disconnect the power cable of the transformer and attach it to the hook.



Figure 13: The p20 electronic unit.

The front panel of the electronic unit has different switches which have the following functions:

S1: A row of 8 push buttons for selecting the function of the minispec p20. From left to right these buttons are labeled

- Stand by: Don't touch this button ! This button should always be in IN position.
- **OPERATE:** When pushed IN, the electronics generates pulses. To stop generating pulses, push this button into OUT position.
- **Puls I:** This button selects Puls I.
- **Puls II:** This button selects Puls II.

- I I: This button selects a pulse sequence Puls I Puls I (not used in F61/F62).
- II I: This button selects a pulse sequence Puls II Puls I.
- I II: This button selects a pulse sequence Puls I Puls II.
- **CP:** This button selects a Carr-Purcell sequence.

S2: In *"single"* mode, this switch specifies that your selection done in S1 is generated only once. In *"periodic"* mode, your selection of S1 will be generated many times in time intervals specified by the S3 switch.

S3: This switch specifies the time interval at which your pulses are produced when S2 is in "*periodic*" mode. This switch S3 consists of an inner and outer knob. The outer knob in black is in logarithmic scale and has settings 100 μ s, 1 ms, 10 ms, 100 ms, 1 s and 10 s. The inner knob in red is in linear mode and multiplies the setting defined by the black knob. Turn the red knob counterclockwise until it locks in position 1. Every click in clockwise rotation increases the multiplicative factor by one unit up to the maximum value of 9.

S4: This switch is active when a I-II, II-I or CP sequence is selected by S1. The value of S4 specifies the time interval between the two pulses of the sequence. The switch S4 consists of an inner and outer knob. The outer knob in black is in logarithmic scale and has settings 10 μ s, 100 μ s, 1 ms, 10 ms, 100 ms and 1 s (**Do not use the 1 s setting, it doesn't work !)**. The inner knob in red is in linear mode and multiplies the setting defined by the black knob. Turn the red knob counterclockwise until it locks in position 1. Every click in clockwise rotation increases the multiplicative factor by one unit up to the maximum value of 9.

S5: The switch S5 has three positions and is a multiplicative factor to the value defined by S4. In upper position, the factor is one. In middle position, the factor is two whereas in lower position the factor is four.

S6: This knob defines the time duration of Puls I. Turn the knob counterclockwise until it is in position 1. This minimal value of S6 defines the minimum duration of Puls I. Turning the knob clockwise increases the time duration of Puls I.

S7: This knob defines the time duration of Puls II. Turn the knob counterclockwise until it is in position 1. This minimal value of S7 defines the minimum duration of Puls II. Turning the knob clockwise increases the time duration of Puls II.

B Appendix B: Minispec p20 Magnet

The minispec p20 magnet is sitting on its own table and is connected to the p20 electronics by a cable. This cable transmits the high frequency pulses generated by the electronics to the coil inside the magnet. In addition, the cable transmits the signal induced in the coil back to the electronics. The magnet is shielded by styrofoam material for minimal temperature variation when in use. Remove the top cover of the styrofoam to have a look into the magnet assembly.



Figure 14: The p20 magnet.

The magnet consists of the following pieces:

- M: The two pole pieces consist of iron material. The two pieces are mounted with an air gap in between where the probes are put for the measurements.
- B: The iron configuration produces a magnetic field of approximately 0.5 Tesla in the air gap. This \vec{B} field points from one pole shoe to the other.
- F: An iron frame holds the two pole shoes in position. This iron frame is used as return yoke for the \vec{B} -field lines.
- W1,W2: Two movable iron screws inside the frame are used to change the magnetic fringe field inside the frame. Since the moving of the screws changes the magnetic field configuration, the magnetic field in the gap can be adjusted. In particular, the screw W1 is adjustable from the outside by turning a knob. (W2 can only be changed with a screw driver, you shouldn't touch W2).
- **P**: The position P indicates where the glass tubes with the probes are positioned for the measurements.
- C: Connector for the cable.
- A: The needle for pressure air can be adjusted such that the glass tubes are put into rotation for the measurement of chemical shift. Adjust the orientation of the needle such that it points onto the small plastic ring on the tubes.

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