TU Dresden

Advanced practical course

Lab report

Nuclear Magnetic Resonace

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Contents

1 Introduction

1.1 Motivation

Nuclear Magnetic Resonance is a physical phenomenon that can be observed while placing an ensemble of nuclei into a static magnetic field and stimulate it with a high-frequent alterning field. A necessary condition for this effect is that the atoms of the sample have a *nuclear spin* different from zero. It is the central concept that is used for *NMR-Spectroscopy*, a standard methodology for the investigation of the structure and interaction of complex molecules and solid state bodies by measuring local magnetic fields, and the *magnetic resonance tomography* which is an imaging technique used in clinical diagnistics for describing the morphilogic and physiologic build-up of tissues and organs. For all of those applications some important parameters of particular physical compensation-processes, the so called *relaxation times* T_1 and T_2 need to be quantified. In the following experiment exactly those material-characteristic observables are determined for an ensemble of ^{57}Fe-nuclei. But at first some basic knowledge.

1.2 Nuclear Zeeman-Effect

Every quantum mechanic angular momentum - especially every spin - is correlated with a magnetic moment μ The proportionality factor is called the *gyromagnetic ratio* γ. So the intrinsic magnetic momentum matching to the nuclear spin $\vec{\mathcal{I}}$ considered in the experiment is given by:

$$\vec{\mu} = \gamma \vec{\mathcal{I}} \tag{1}$$

$$\gamma = g_I \frac{\mu_N}{\hbar} \overset{^{57}Fe}{=} 0.8661 \cdot 10^7 \text{ T}^{-1}\text{s}^{-1} \tag{2}$$

Where μ_N is the *nuclear magneton* and g_I is the Landé-factor which both are core-specific parameters. If those magnetic moments are placed in a static magnetic field $\vec{B} = B\vec{e}_z$ then the Hamiltonian of the system and its Eigenvalues to the Eigenstates of the spin-operator $|I, m_I\rangle$ are given by:

$$\mathcal{H} = -\vec{\mu}\vec{B} \overset{(1)}{=} -\gamma \mathcal{I}_z B \tag{3}$$

$$\langle \mathcal{H} \rangle = \langle I, m_I | \mathcal{H} | I, m_I \rangle = -\gamma B \langle I, m_I | \mathcal{I}_z | I, m_I \rangle = -\gamma B \hbar m_I \equiv E_{m_I} \tag{4}$$

Where the Eigenvalues of the Spin-operator for given spin-quantumnumber I and magnetic quantum number $m_I = -I, \ldots, I$ were used. So the outer magnetic field annuls the $2I+1$-fold degeneration of the energystates. The nuclear spin-quantum-number of ^{57}Fe is I $= 1/2$ so there are two additional energystates with a energydifference:

$$\Delta E = \hbar \gamma B = \hbar \omega_L \tag{5}$$

As equation (5) suggests, there may occur optical transitions between the terms which lead to an emission of photons with the angular frenquency w_L. One finds that this frequency is equivalent to the *Larmor-Frequency* that describes the precession of a magnetic moment around the z-axis caused by the torsional moment $\vec{M} = \vec{\mu} \times \vec{B}$ in a magnetic field. The classical description of this process leads to the same result as quantum mechanics do. If we consider the ensemble of N nuclei as canonical, then the number of spins in the state $|s, m_I\rangle$ in the thermodynamic equivilibrium is given by the Boltzman-statistics:

$$N(m_I) = N \cdot \frac{e^{-\frac{E_{m_I}}{k_B T}}}{Z} = N \cdot \frac{e^{\frac{\hbar \gamma B m_I}{k_B T}}}{Z} \tag{6}$$

Where $Z = $ const. is the canonical partition function and T is the absolute temperature of the environment. This implies that the spins prefer to be polarised not uniformely but in the direction of the B-field so this leads to a mean magnetic moment $\langle\vec{\mu}\rangle \neq 0$. This leads to an oberservable macroscopic magnetisation in the volume V:

$$\vec{M} = \frac{d\vec{\mu}}{dV} \cong \frac{N}{V}\langle\vec{\mu}\rangle \neq 0 \tag{7}$$

These changes in magnetisation are used to induce voltages that can be measured.

1.3 Hahn-spinecho

If the spins are just inside of a static magnetic field along the z-axis then they precess around this axis with an angular frequency of $-\omega_L$. In the next step an alterning magnetic field $\vec{B}_{RF} = B_{RF}\sin(\omega t)\vec{e}_x$ is applied to the spins additionally. If we consider *resonance* of the Larmor-precession and the radio-frequency field, i.e. $w = w_L$, then we can rotate the magnetisation around the x-axis by an angle of $\alpha = \gamma B_{RF} t$ where t is the time the RF-field is applied. With this effect we can now apply different *magnetic resonance sequences* to investigate the interaction of the spins with each other and the external magnetic fields.
The first sequence is a really simple one: the 90°-pulse where the spins are flipped in the xy-plane, i.e. $\vec{M} \perp B\vec{e}_z$. Because they now rotate around the z-axis in the laboratory system the periodic change of the magnetic field within the coil that is part of the HF-circuit leads to the induction of an alterning voltage with frequency ω_L. Because the spin-moments interact with each other their angular frequency differs from spin to spin. This Dephasing leads to the decrease of the mean magnetisation in xy-plane and so the measured voltage fades away. This effec is called *Free Induction Decay*. By applying an 180°-pulse after a time τ the spins rephase and the voltage increases again (*Hahn-Spinecho*). Because of irreversible effects during de- and refocussing the polarisation decreases and the mean magnetisation in xy-plane and the measured voltage have a lower amplitude. The correlation between the dephasing time τ and the mean magnetisation in xy-plane can be described by an exponentially decaying function:

$$U_{ind}(\tau) \propto M_{xy}(\tau) = M_{xy}(\tau = 0) \cdot e^{-\frac{2\tau}{T_2}} \tag{8}$$

The decay of the reversible nuclear magnetisation is called *spin-spin-relaxation* and the corresponding time constant is the *spin-spin-relaxationtime* T_2. In the experiment it will be measured by variing τ and measuring the alterning voltage U_{ind}. Figure (1) depicts the Hahn-Spinecho.

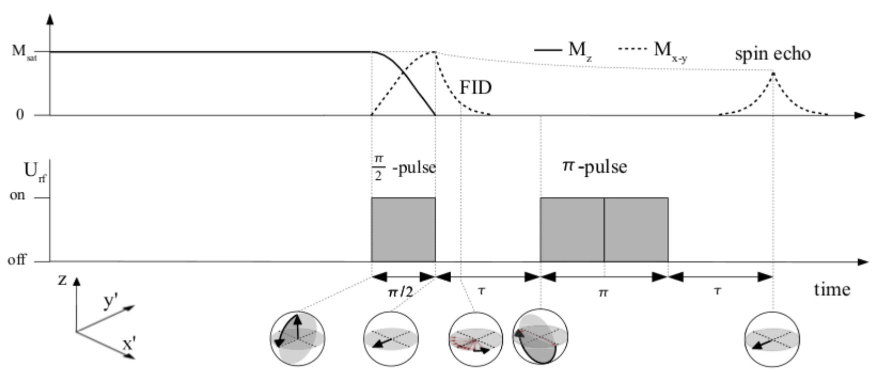


Figure 1: Hahn-Spinecho sequence for measuring T_2 relaxation time

1.4 Spin-Latice Relaxation

Because the energy of an ensemble of spins is minimal when they are polarised longitudinally along the magnetic field $B\vec{e}_z$ they are into they will relax to this equivilibrium state when it is perturbed, e.g. by a 90°-pulse. After applying this pulse and waiting for a time Δt the z-component of the mean magnetic moment $\langle \mu_z \rangle$ increases. After *Deltat* a Hahn-Spin-echo sequence mentioned in section (1.3) is applied. Because the measure signal of this sequence is proportional to the initial magnetisation we will induce an echo-voltage in the HF-coil that increases by waiting longer after the 90°-pulse such that more spins are relaxed back to the equivilibrium state. The dependency between Δt and the measure signal can be described by:

$$U_{ind}(\Delta t) \propto M_{xy}(\Delta t) = M_{sat} \cdot [1 - e^{\frac{\Delta t}{T_1}}] \tag{9}$$

The relaxation of the longitudinal component of the magnetisation is called *Spin-latice relaxation.* The occuring time constant T_1 is the *Spin-latice-relaxation time* and corresponds to the time one has to wait between the pulses such that the magnetisation is approximately 63.21 % of the saturation value. This parameter will be measured by variing Δt and holding the time-constant of the Hahn-sequence τ constant. Figure (2) depicts the sequence.

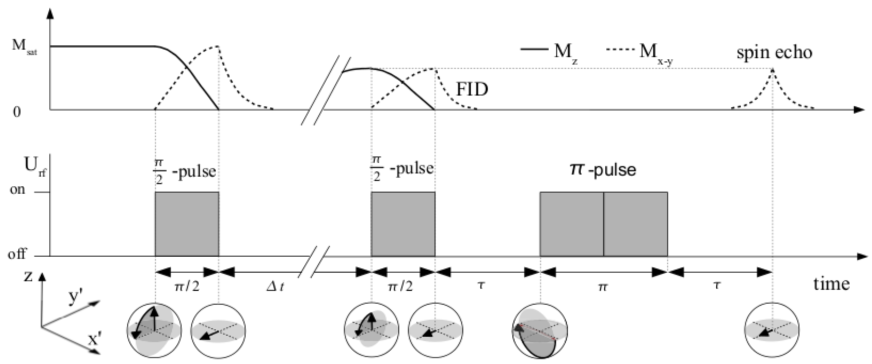


Figure 2: MR-sequence for measuring the spin-latice relaxation time T_1

2 Experimental procedure

The followoing task had to be done:
2.1: Preparation of a radio frequency resonant circuit with the insertion of an iron powde probe in a cryostate
2.2: Find the nuclear spin resonance by varying the NMR-pulse frequency while recording a spectrum of the ^{57}Fe nuclear spin ensemble and calculation of the local magnetic field
2.3: Optimization of the pulse sequence by recording a rotation angle curve
2.4: Find the spin-spin- and spin-lattice relaxation constants T_2 and T_1.

2.1 Preparation of a high frequency resonant circuit

First one has to prepare a copper coil with a diameter big enough to hold an iron powder assay. After the coil is wrapped one has to sold it onto the contacts of a stick, which provides a mechanism to tune the measured frequency to find the resonance frequency. While solding one has to be careful to make sure, that one do not take to much tin, because the resisteaces of tin and copper are different. In the same way the soldered point should connect the contact and the coil directly, otherwise this could influence the results while tuning to the resonance frequency.

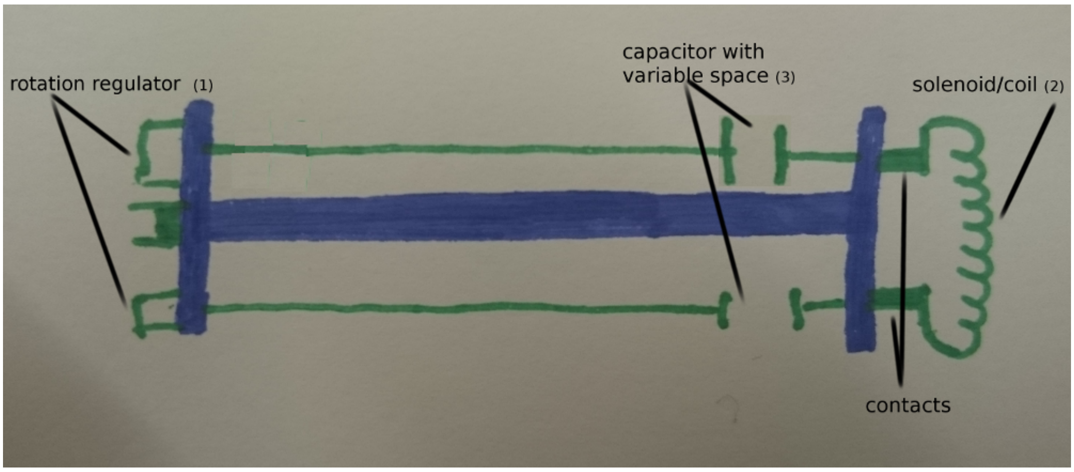
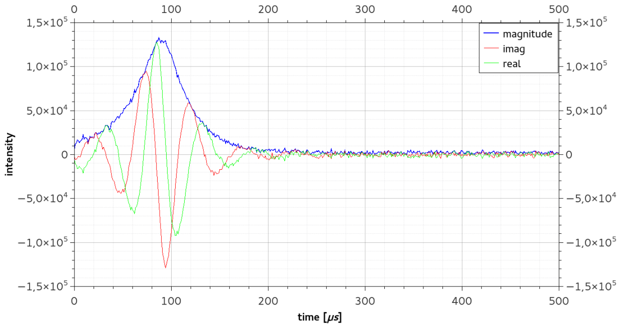


Figure 3: basic sketch of a probe with a coil on it

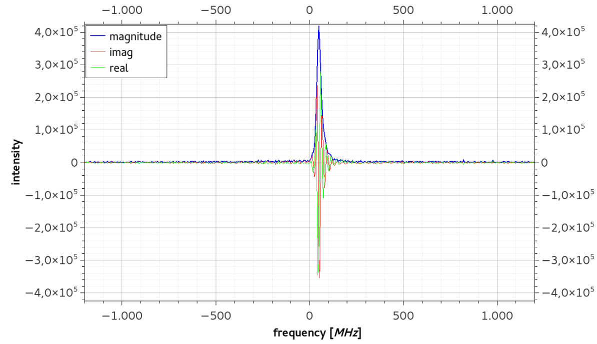
The rotation regulator (3.1) varies the space between the capacitor's plates (3.3) so one can tune the frequency of the solenoid (3.2).

2.2 Finding of the nuclear spin resonance by varying the pulse frequency

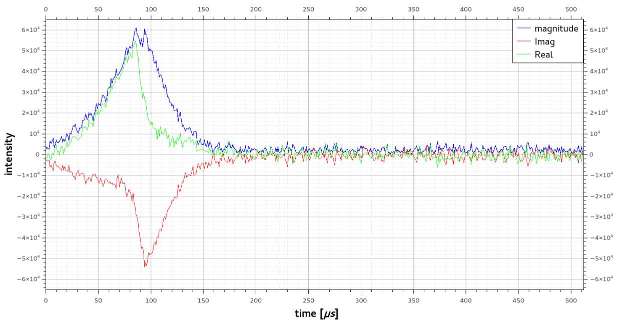
After the preparation of the solenoid and the insertion of the probe into the cryostat one has to mention what the different paramters, the measurement software has, do with the curve. The different parameters one could vary are pw, τ, rd and ad. pw makes the magnitude look more gaussian and gives it a bigger value by make the imaginary and real parts more oscillating the higher it is. Varying τ shows less oscillations and a lower magnitude the higher τ is. At least one can also vary the parameter rd. The higher it is, the more oscillations can be found. One varies this parameters to better fit the magnitude to a gaussian. This means, one searches parameters with a low amount of oscillations, which also have not a big intensity. After the best parameters ($pw = 2\mu$s, $\tau = 60\mu$s, $rd = 15\mu$s, $ad = 10\mu$s) are found, one looks at the behavior of the curve at the resonance frequency, a little bit below and a bit above as shown in the figures 4(a) - 4(f).



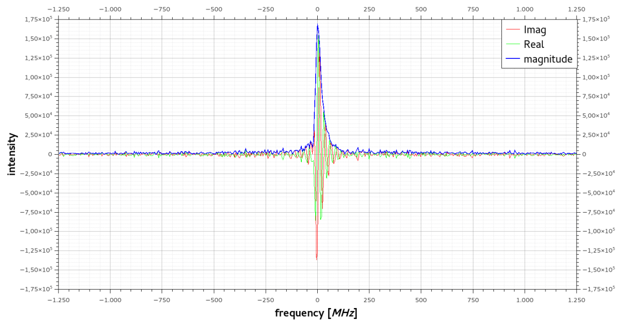
(a) intensity below resonance frequency in time domain



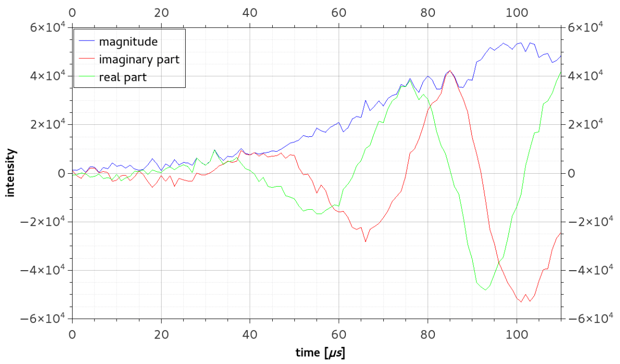
(b) intensity below resonance frequency in fourier space



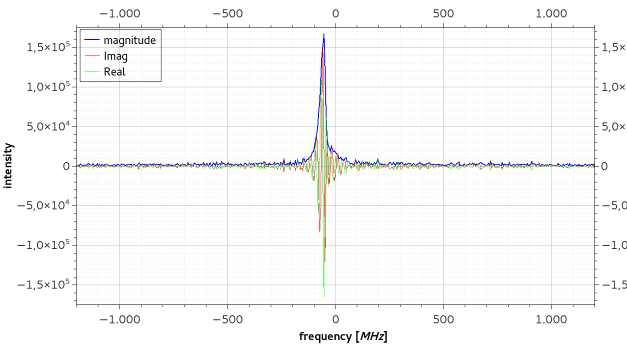
(c) intensity at resonance frequency in time domain



(d) intensity at resonance frequency in fourier space



(e) intensity below resonance frequency in time domain



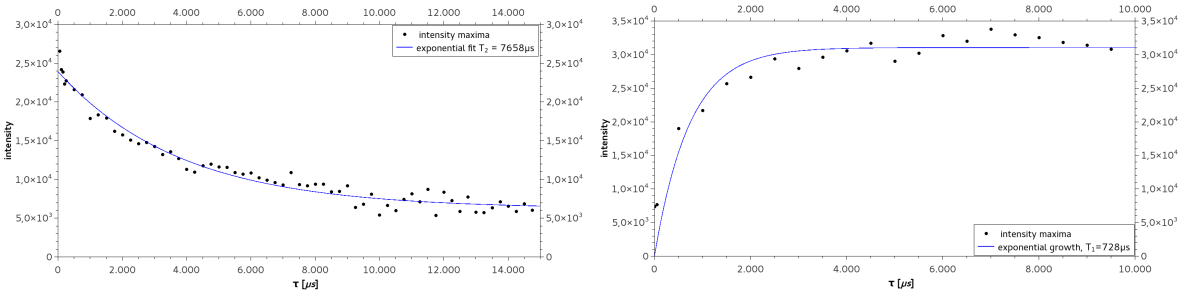
(f) intensity below resonance frequency in fourier space

Figure 4: Different spectra below, at and above the resonance frequency

One can see that the number of oscillations in 4(e) and 4(a) is bigger than in 4(c). In figure 4(d) one recognizes a good gaussian peak. With that information the frequency is found at $\omega_L = 45.47\text{MHz}$. Using the γ-factor defined in (2) and the formula (5) one can calculate the magnetic field as $B_z = \omega_L/\gamma = 0.525$ T. This is a typical value for a magnetic flux density occuring in the bond of a solid state body.

2.3 Optimization of the pulse sequence by recording a rotation angle curve

Now one can configure the time intevalls how long the measurement device should wait until it takes a measurement. In both cases, the spin-spin- and the spin-lattice-relaxation one get different times for τ aus one can see in the diagrams. After this measurements the computer takes all magnitude maxima as data points one can analyze. One can recognize that 5(b) looks like an exponential growth and 5(a) looks similar to an exponential decay. To determine the relaxation constants one uses this information to make fits.



(a) another test (b) some test

Figure 5: rotation angle curves for spin-spin- (a) and spin-lattice-relaxation (b)

2.4 Determination of the relaxation constants T_1 and T_2

The following fit functions are used;

$$y(\tau) = A \cdot (1 - e^{-\tau/T_1}) \tag{10}$$

$$y(\tau) = y_0 + Ae^{-2\tau/T_2} \tag{11}$$

With a tool called Qtiplot one makes a fit with these funktions a gets the blue line in 5(b) and 5(a). The parameters for 11 are: $A = 17720$, $y_0 = 6220$ and the spin-spin-relaxation constant $T_2 = 7628\mu\text{s}$ The parameters for the spin-lattice-relaxation are:

$$A = 31050, \ T_1 = 728\mu\text{s}$$

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3 Data Analysis

4 Discussion and conclusions

References

[01] S. Diez. *Advanced practical course - Experiment MMC*, Dresden, 2015

[02] `https://en.wikipedia.org/wiki/Histogram` [November 20, 2015]

[03] `https://en.wikipedia.org/wiki/Kinesin` [November 22, 2015]

[04] S. Ferbrugge et al. *Novel ways to determine kinesin-1's run length and randomness using fluorescence microscopy*. Amsterdam. October 2009

[05] S. Klumpp, R. Lipowsky. *Cooperative cargo transport by several molecular motors*. Potsdam. November 2005