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F-Praktikum NMR

Lab Report

Nuclear Magnetic Resonance

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Contents

1. Introduction

The Nuclear Magnetic Resonance is a physical phenomenon wich allows to look into a nucleus. This works, because the atomic nuclei absorbs and emits electromagnetic radiation of a cerain resonance frequency when it is brought into a magnetic field. As other physical phenomena it is used in medicine for diagnosis (magnetic resonance imaging) or research (magnetic resonance microscopy). It is also used in chemistry or in physics as it is done in this experiment.

The aim of this experimt is to do something with high frequency circuits and to determine two time constants: the spin-spin and spin-lattice relaxation time.

2. Theory

2.1. Nuclear Zeemann effect

If one brings a nucleus in a magnetic field there is a separation of the energy levels which is already known from levels of the electrons, and know as the Zeemann effect. The Hamiltonian $\mathcal{H}$ can be written as

$$\mathcal{H}_\mathrm{Z} = -\vec{\mu}\vec{B} = -\gamma\vec{I}\vec{B} \tag{2.1}$$

with the gyromagnetic ratio

$$\gamma = g\frac{\mu_\mathrm{N}}{\hbar}, \tag{2.2}$$

the magnetic field $\vec{B}$, the nuclear magneton μ_N, the Landé factor g and the nuclear spin $\vec{I}$.

If the magnetic field is homogeneous the eigen values of the Hamiltonian can be calculated with

$$\langle\mathcal{H}_\mathrm{Z}\rangle = E_\mathrm{Z} = -\gamma B_z\hbar m. \tag{2.3}$$

If $I = 1/2$ the spin state splits up in two. The frequency for the transition is

$$\omega_L = \gamma B_z. \tag{2.4}$$

2.2. Free induction decay

The equation of motion for the magnetization is

$$\frac{\mathrm{d}\vec{M}}{\mathrm{d}t} = \gamma\vec{M} \times B_z\vec{e}_z. \tag{2.5}$$

Therefor the magnetization of the nucleus is rotating about the magnetic field. Written as a scalar this corresponds to the angular velocity $-\omega_L$.

In addition to the static magnetic field a small magnetic field B_{RF} is added. This field is an alternating field with a frequency in the range of several MHz. It is perpendicular to the static field:

$$\vec{B} = \begin{pmatrix} B_{\mathrm{RF}}(t) \\ 0 \\ B_z \end{pmatrix}. \tag{2.6}$$

Finally it is possible to change the angle of the magnetization of the nucleus by applying B_{RF} for a certain time t:

$$\alpha = \gamma t B_{\mathrm{RF}} \tag{2.7}$$

If the vector of the nuclear magnetization is turned in the x-y-layer, a alternating voltage U_{ind} is induced in the coil with a frequency of γB_z. This voltage decays after same time. This is called *free induction decay.*

2.3. Spin echo

For the NMR displacements angles of 90° and 180° are very interesting. If the angle is 90°, the magnetization lays in the x-y-layer. Since the static magnetic field is not homogeneous the precession of the nuclear spins are not the same. If one applies a high frequency pulse after a time τ to turn the magnetization by 180°, it is possible to refocus the spins after another period of time τ. Since there are losses the *echo* will not induce the the same voltage. The magnetization of the echo can be calculated with

$$M_{\text{Echo}} = M_{\text{Sat}} \cdot \mathrm{e}^{-\frac{2\tau}{T_2}}. \tag{2.8}$$

The decay of the nuclear magnetization is called *spin-spin relaxation.*

2.4. Spin-lattice relaxation

There are also losses due to the spin-lattice interaction. T_1 is the time constant of this *spin-lattice relaxation.* The effect is caused by the magnetization, wich tries to realign with the static field. With a 90°-pulse the saturation magnetization will be destroyed. After some time the magnetization reaches the saturation again. This is described by:

$$M_{\text{echo}} \sim M_{\text{sat}} \left(1 - \mathrm{e}^{-\frac{\Delta t}{T_1}}\right). \tag{2.9}$$

and can be examined by using another spin-echo-sequence.

3. Experimental Procedure

3.1. Preparation

The first step of this experiment was the preparation of a high frequency LC circuit. Therefore a coil had to be designed. Since the length of the coil and the number of turns are very low, the simple formula to calculate the inductance L could not be used. To determine the right number of turns different coils were tested with a network analyzer. Then the iron powder was inserted in the coil and the two capacitors in the circuit had to be adjusted to reach a resonance frequency of $45.5\,\mathrm{MHz}$. This frequency is needed for the NMR in the iron powder.

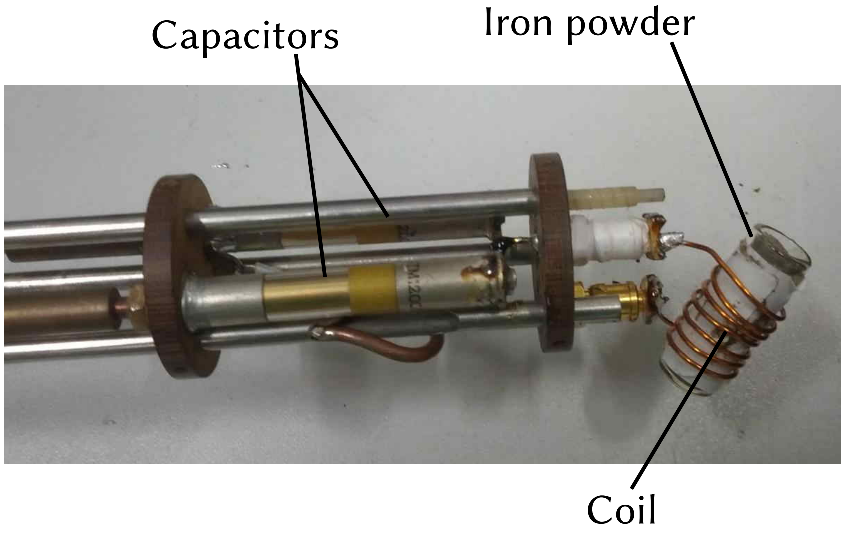

Figure 3.1: Preparation of the measurement.

The coil which was used in the experiment had in the end seven turns of copper wire and a diameter of ca. $0.8\,\mathrm{cm}$.

After this the LC circuit got mounted in a cryostat. This cryostat was not used to cool the specimen and circuit but to hold everything in position. For this experiment the zero field NMR was used. The used magnetic field was the internal magnetization of the iron powder. It can be calculated with

$$B = \frac{\omega}{\gamma} = \frac{2\pi f}{\gamma} \approx 33\,\mathrm{T} \tag{3.1}$$

and $\gamma/2\pi = 1.382\,\mathrm{MHz}\,\mathrm{T}^{-1}$ [3].

3.2. Examination of the resonance frequency

With the software *NTMR* three spin-echo spectra were taken at different frequencies (45.16 MHz, 45.5 MHz and 45.67 MHz). For each frequency the capacitors of the LC circuit had to be adjusted. The parameters of the software were chosen to achieve a good signal-to-noise ratio, they are listed in section A in the appendix. In figure 3.2 the three spectra can be compared.

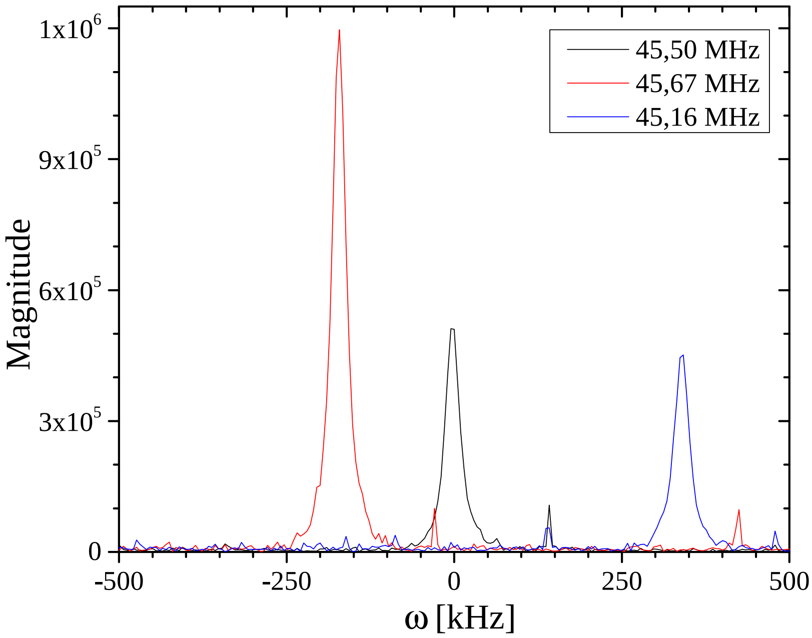

Figure 3.2: Spectra of the three measurements with different resonance frequencies of the LC circuit.

The maximum peak is found at a frequency of 45.67 MHz, but the peaks seam to be very similar.

3.3. Spin-spin and spin-lattice relaxation time

For measuring the spin relaxation time a frequency of 45.5 MHz was used, because it is the resonance frequency of the 57-Fe nucleus.

Since induced voltage and M_{Echo} are proportional, the spin-spin relaxation time constant can be determined with (2.8) and measurements with varying τ. In figure 3.3 one can see the result of these measurements with a fit. It returned a value of $T_2 = (12\,100 \pm 30)\,\mu\text{s}$. The used function for the fit was

$$y = y_0 + A \cdot \mathrm{e}^{R_0 \cdot x}. \tag{3.2}$$

In table 3.1 the parameters of the fit are listed.

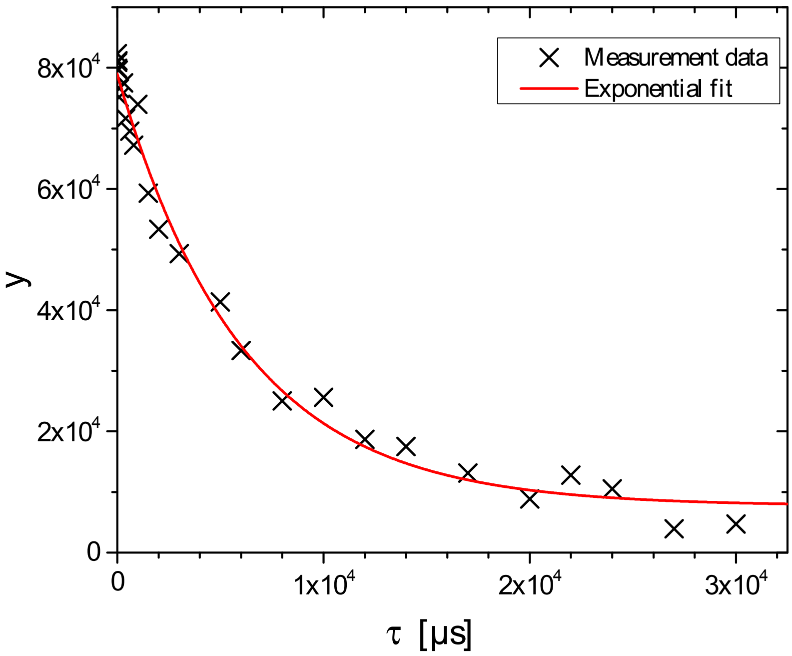

Figure 3.3: Measured behavior of the spin-echo amplitude.

Table 3.1: Raw parameters of the fit for T_2.

parameter	value	error
y_0	7703.883 84	1551.197 93
A	71 186.915 92	1663.788 23
R_0	-1.65×10^{-4}	1.26×10^{-5}

The fit for T_1 was done with the equation

$$y = A1 \cdot \mathrm{e}^{-x/t1} + y_0 \tag{3.3}$$

the parameters of the fit can be found in table 3.2. An additional fit for a constant function helped to find the saturation amplitude an to improve the exponential fit.

Comparing with (2.9) the time constant has the value $T_1 = (6900 \pm 900)\,\mu\mathrm{s}$. In figure 3.4 the measured data and the fit are shown.

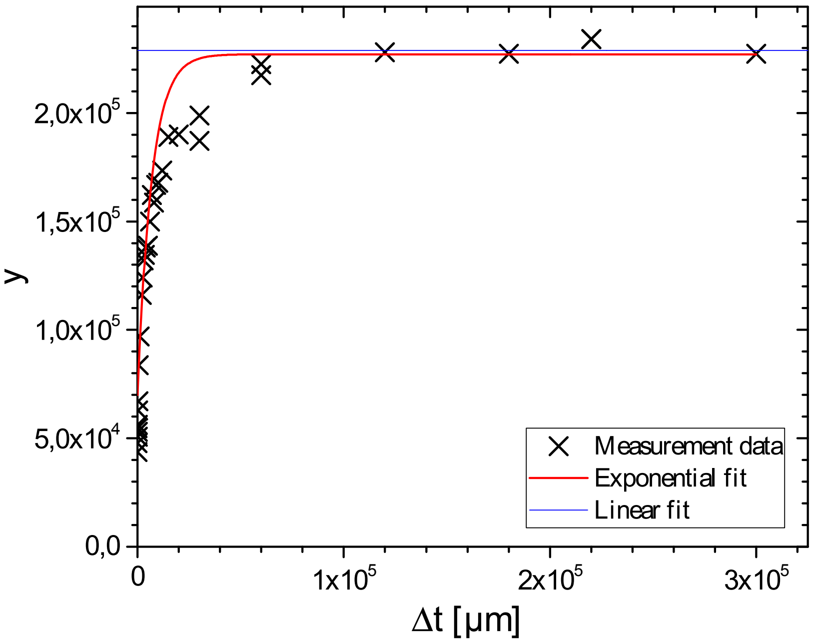

Figure 3.4: Measured behavior of the spin-echo amplitude.

Table 3.2: Raw parameters of the fits for T_1.

(a) Exponential fit.

parameter	value	error
y_0	227 083.4303	5603.6844
A_1	−157 942.907 98	7608.275 35
t_1	6860.169 54	897.682 07

(b) Linear fit.

parameter	value	error
y-axis intercept	228 901.430 08	1817.999 79
slope	0	–

4. Discussion

Zero field NMR is possible due to the very high internal field of the 57-Fe nucleus, which is much higher than the fields which can be achieved when using conventional magnets. The spin-spin relaxation time T_2 is 7/4 times higher than the spin-lattice relaxation time T_1.

There were some problems which lead to inaccuracy of the results, which could not be calculated: The LC circuit could not be exactly adjusted since the parameters changed when the coil was moved. This was observed after the measurements. There was also a problem with the examination of the resonance frequency of the nucleus, which was maybe result of a wrong usage of *NTMR*. This shows in the spectra which seem to be the same except for size and position.

References

[1] Instruction: *Fortgeschrittenen-Praktikum – Nuclear Magnetic Resonance (NMR)*, TU Dresden, 2013.

[2] Wikipedia: *Nuclear magnetic resonance*, `https://en.wikipedia.org/wiki/Nuclear_magnetic_resonance`, 23. January 2016.

[3] Wikipedia: *Gyromagnetic ratio of an atomic nucleus*, `https://en.wikipedia.org/wiki/Gyromagnetic_ratio#Gyromagnetic_ratio_for_a_nucleus`, 28. January 2016.

A. Parameters for NTMR

parameter	value
pw	1
tau	60
rd	15
ad	10
Acq Time	204.8
Last Delay	30×10^3
C13pw90	12
tunblank	1

B. Raw data from Measurements for time constants

B.1. T_1

$\Delta t/\mu s$	amplitude
30	54 676.261 183
40	43 682.404 513
50	47 703.885 890
100	50 753.242 941
150	52 922.642 489
200	56 293.473 663
300	62 844.620 398
400	67 140.349 068
600	83 723.423 067
1000	97 017.892 886
2000	116 124.242 762
2500	124 163.511 713
3000	132 078.182 002
3500	134 642.002 455
4000	137 911.581 649
5000	138 767.495 722
6000	149 992.827 565
7000	162 202.883 353
8000	158 676.999 474
9000	166 820.295 846
10 000	167 846.481 905
12 000	173 411.293 822
15 000	188 998.486 258
20 000	190 152.377 069
30 000	198 888.130 800
60 000	217 462.181 238
30 000	187 195.787 356
60 000	222 547.480 338
120 000	228 008.901 554
180 000	227 366.007 074
220 000	234 061.801 072
300 000	227 362.589 381

B.2. T_2

$\tau/\mu s$	amplitude
5	83 838.267 665
10	88 580.499 254
20	88 777.038 952
30	86 075.638 760
50	78 321.442 326
80	80 851.874 839
100	81 687.482 083
200	78 232.887 183
300	75 603.154 815
500	73 672.013 743
800	67 309.897 474
1000	67 400.246 743
2000	56 958.231 231
3000	46 416.041 290
5000	39 621.242 547
8000	30 204.418 236
10 000	22 029.613 251
20 000	9134.450 503
30 000	7106.261 183
40 000	3024.651 054