Erdfeld-NMR Remote

Physikalisches Fortgeschrittenenpraktikum an der Universität Konstanz

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Abstract

TEXT

Alle Autoren haben zu jedem Abschnitt wesentliche Beiträge geleistet. Die Autoren bestätigen, dass sie die Ausarbeitung selbstständig verfasst haben und alle genutzten Quellen angegeben wurden.

Contents

1 Einleitung

2 Aufbau und Durchführung des Versuchs

3 Noisemeasurement

The first step in the EFNMR Remote experiment is to measure the external noise. The external noise depends on the location where the setup is placed, the orientation of the probe and by surrounding metal objects e.g. a metal desk. To detect this external noise, a measurement without an NMR signal is provided. The time domain noise signal is shwon in figure **??**. It is clearly visible that the noise is centered around $0\,\mu$V. To gain knowledge about the noise level, the computer calculates the root-mean-square (RMS). This means that it calculates the square of each data point, than sum up all squared values up, calculates the average and than applies a square root. With this method the noise level can be calulated. In this case it is $7.5\,\mu$V. This is an ecceptable noise value, because any value below $10\,\mu$V is good enough to provide good NMR data.

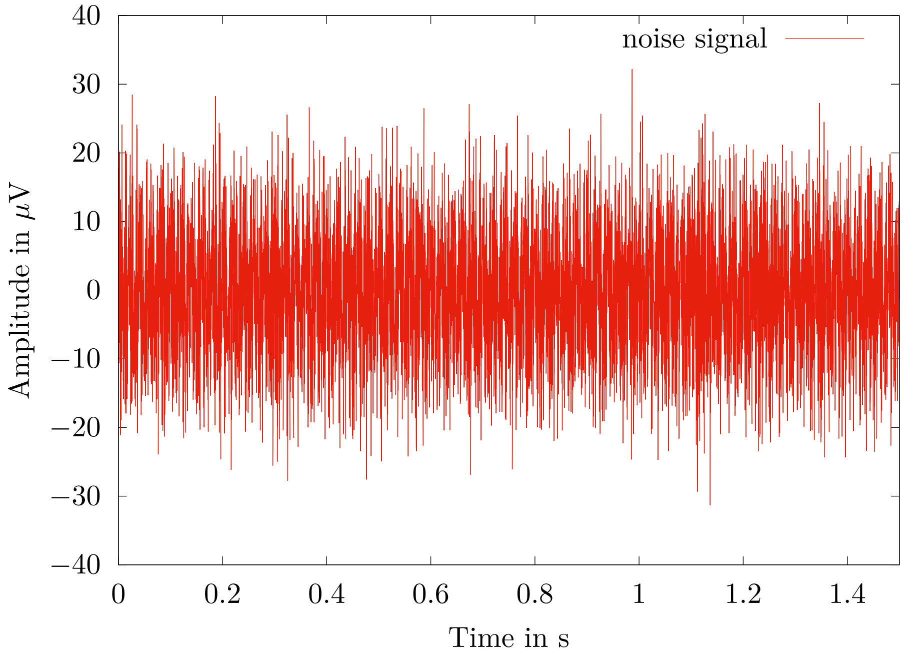

Figure 3.1: Noise signal taken by the B_1 coil. The noise value of this noise is $7.5\,\mu$V.

Figure **??** shows the frequency domain noise. This means that the time domain is fourier transformed into the frequency domain. This method is one of the basic principles we use in this experiment to make research about the properties of the measured signals. The frequency domain noise shows very specific sharp peaks every 50 Hz. To be more specific the peaks in the middle of every hundred Hz steps are way higher than those at 1400 Hz, 1500 Hz and so on. This results of the frequency in the power grid which is 50 Hz in

Germany. wieso sind die geraden kleiner??? Steckdose erklärt eig nur peak bei 50hz und nicht die alle 50 hz Despite all sharp peaks there is also a slight increase of the amplitude around 1850(1000) $\frac{\mu V}{Hz}$ visible. This is explicable by the resonance frequency of instrument and its sensitvity around the lamorfrequency (1841.4 Hz for water in Germany in July 2020). Nearby the lamorfrequency all our following measurements will be done that is why the instrument sensitvity is sharpend around this value.

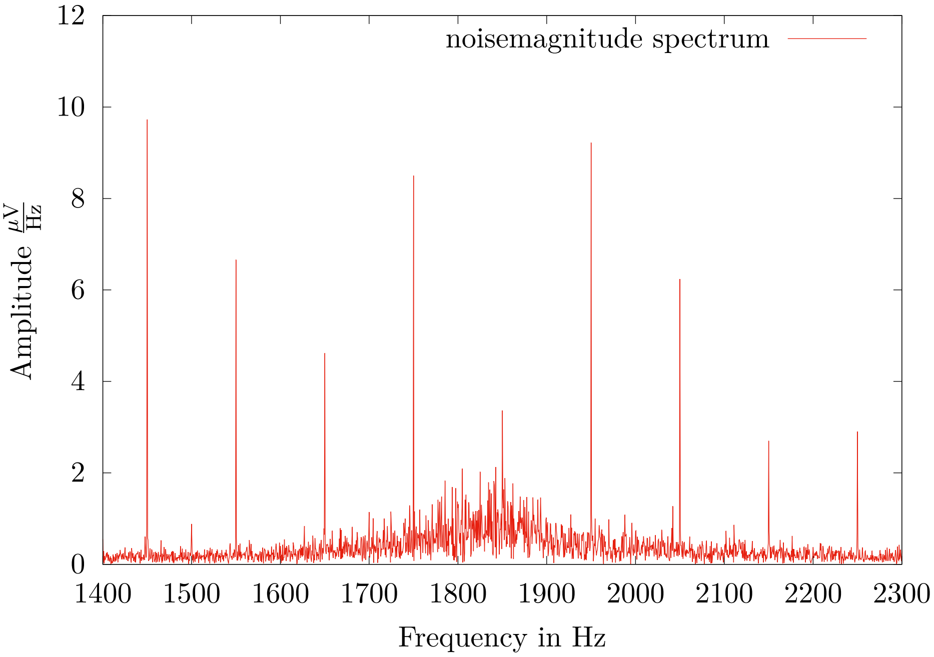

Figure 3.2: Fourie tranformed noise signal of the previous figure **??**. Strong peaks every 50 Hz correspond to the frequency of the power grid in Germany. The slight increase of the amplitude around 1850(1000) $\frac{\mu V}{Hz}$ is explicable by the resonance frequency of instrument and its sensitvity around the lamorfrequency (1841.4 Hz for water in Germany in July 2020).

4 Coil Analysis

Now knowing that we have a acceptable noise value under 10 μV, we can analyse the coil. In order to do so we explain the general approach of NMR signals first. To measure a NMR signal a pulse and collect measurement has to be done. Therefore the B_1 coil (transmit and collect coil) has to apply a pulse. This pulse changes the spins direction out of its thermal equilibrium (along z-axes, due to the earths magnetic field B_e) into a direction with a component in the transversal plain. Therefore the B_1 coil collects a signal, because its aligned orthogonal to B_e. The transmit and collect procedure is based on Faraday's law of induction. Figure **??** exemplary shows such a collect signal by the B_1 coil. Every following measurement in this paper is based on the procedure of pulse and collect.

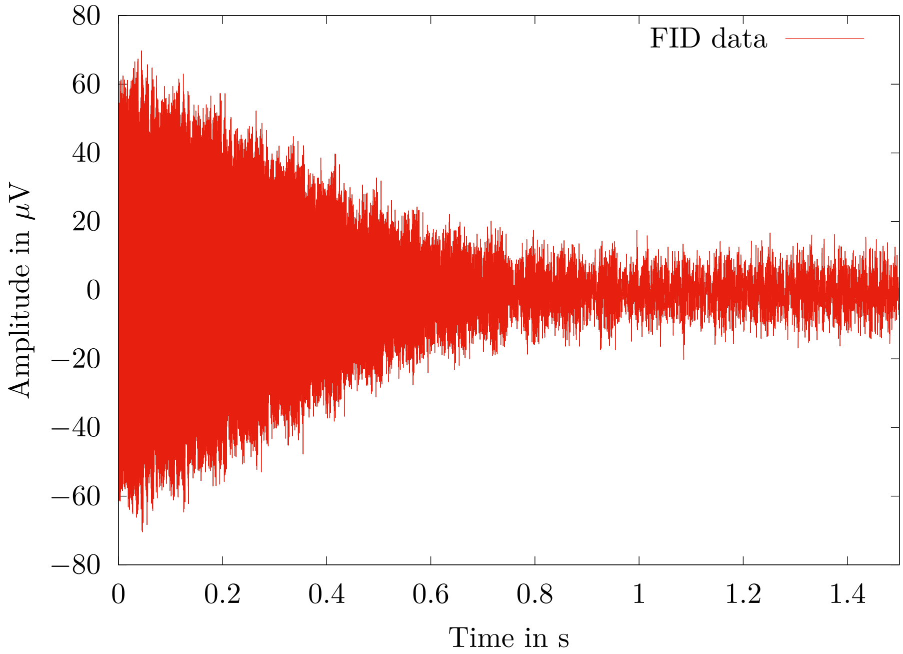

Figure 4.1: Example signal for a pulse and collect signal made by the B_1 coil. The example signal is taken from a FID signal.

Due to the fact that the B_1 coil is a tuned LCR circuit a resonance frequency exists, which can be caluclated by following formula:

$$\omega_{calc} = \frac{1}{\sqrt{L \cdot C}} \,. \tag{4.1}$$

To analyse the B_1 coil the resonance frequency versus the capacity is measured. Therefore the B_1 coil transmits a signal. Due to this signal the response of the coil can be measured. This signal is than fourie tranformed and the resonance frequency can be deduced from the frequency domain (maxmimum in the frequency domain). This procedure is repeated automatically by the computer programm "*Prospa*" for different capacities. By changing the capacity we can examen the best capacity in dependence of the lamor frequency. Figure **??** shows the measured and theoretically calculated resonance frequency (Equation **??**; $L = 0.417\,\mathrm{H}$) in dependence of the capacity. The horizontal line represents the lamor frequency of 1841.4 Hz for water in Germany in July 2020. To gain this value the vertical component of the earths magnetic field (43 248.8 nTQuelle Marc) is multiplied to the gyromagnetic ration 42.577 $\frac{\mathrm{MHz}}{\mathrm{T}}$. Quelle Marc. The vertical line represents the correct capacity we should use for our measurement, due to the resonance frequency of the lamor frequency. In this case the correct capacity is 13.8 nF. For the calculated resonance frequency the correct capacity would be 17.9 nF. It is not deniable that the measured curve is not parallel to the measured resonance frequency. This probably has its cause in the not fix inductance L. Due to heating of the coil L might change a little by increasing capacity and thus the calculated curve does not fit to the measured one.

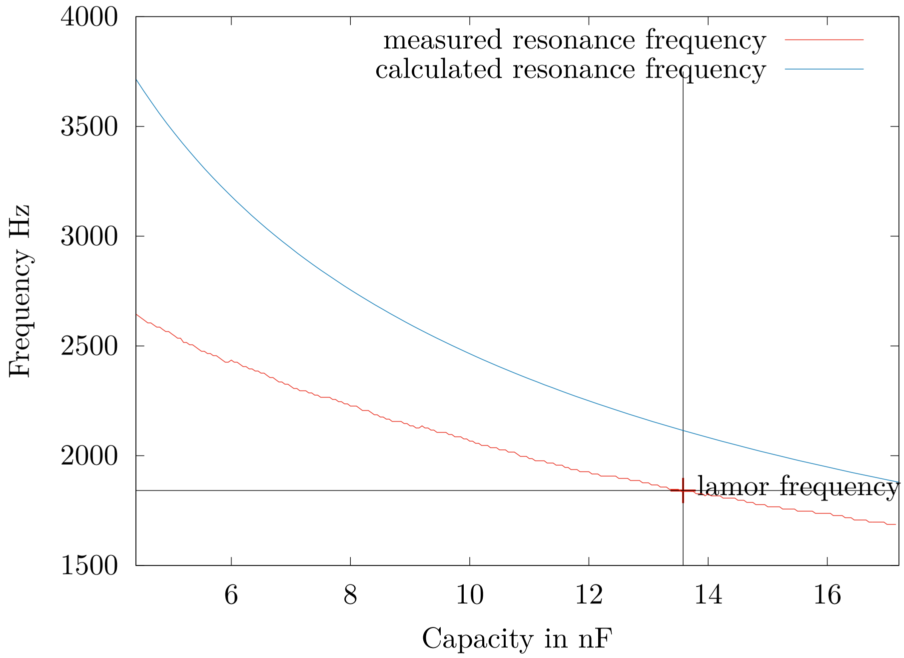

Figure 4.2: This figure shows the measured and calculated resonance frequencies for different capacities. The marked cross represents the lamor frequency of 1841.4 Hz for water in Germany in July 2020.

5 Optimization and Characterisation of FID in water sample

On of the main goals of this experiment is to measure a good FID of the water sample. In order to do so we first have to optimize our FID signal of the water probe.
First the inhomogenity of the magnetic field has to be cancelled. The process to make the magnetic field more homogenious is to *autoshimm* the components of the gradient coil. The computer programm does this automatically. So it deshimms the system step by step and checks if the output maximizes or minimizes. By checking many different

combinations it finds the best shimming values for the gradient coil. In our case they are:

$$
\begin{aligned}
x &= 10.11\,\mathrm{mA} \\
y &= 20.88\,\mathrm{mA} \\
z &= -20.07\,\mathrm{mA}\ .
\end{aligned}
$$

That means with those shimming values the magnetic field in the setup is homogenious. The secon optimization step is to change the B_1 pulse duration. The longer the pulse duration is, the bigger is the angle of the flipping spins and thus the signal will get stronger (only counts for flipping angles till 90°). The best signal is obtained for an flipping angle of 90°, because with this angle the spins only have a component in the transversal plane and therefore the signal is maximized. If the pulse duration is too long, than the flipping angle is bigger than 90° and the spins get a horizontal component again and the signal will decrease again. When a flipping angle of 180° is reached the signal will be at its minimum. Afterwards the signal will raise again, because the horizontal component will increase again. Figure **??** shows this issue. The maxmimum at a pulse duration of 1.35 ms is clearly visible. This means that after apllying a B_1 pulse with a duration of 1.35 ms the spins are in the transversal plane and therefore the best signal is obtained.

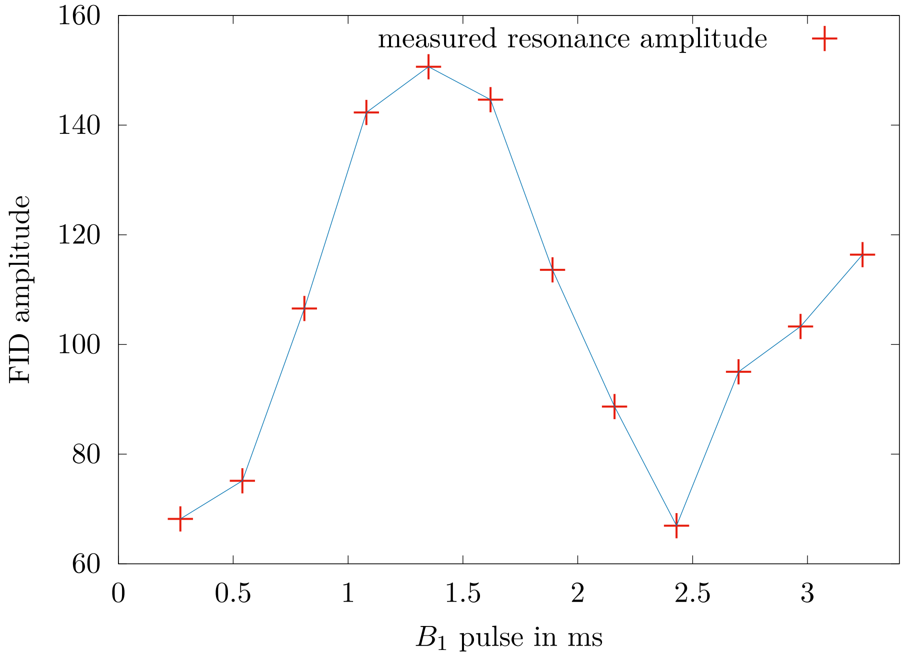

Figure 5.1: This figure shows which impact the B_1 pulse duration has to the ampitude of the FID. It is clearly visible that the duration has a maximum at 1.35 ms which is the duration for a 90° pulse.

Figure **??** exemplarily shows the correlation of the B_1 pulse duration and the signal which the coil detects. It is clearly visible that the amplitude is better for the pulse duration of 1.35 ms than for the pulse duration of 0.27 ms. The signal that was taken for the pulse duration of 0.27 ms is at the minimum of the figure **??** and therefore it is correct that the amplitude of the spectrum with the pulse duration of 1.35 ms is higher.

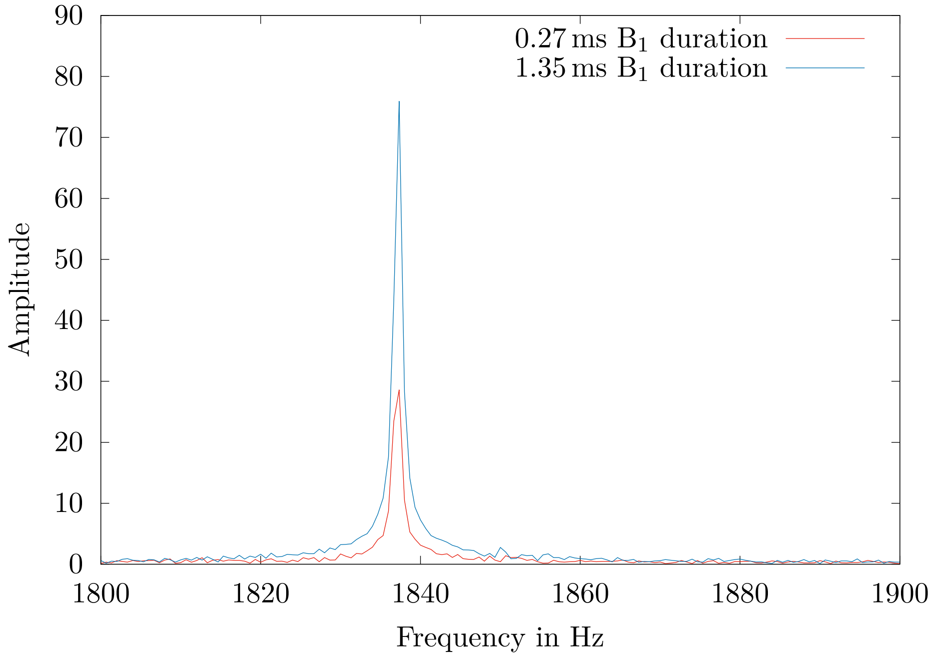

Figure 5.2: Example spectrum for two different B_1 pulse durations. The peak which is higher corresponds to the 1.35 ms duration pulse and represents the 90° pulse. This peak is high, because at this duration most of the spins are in the transversal plane and therfore the amplitude is maximal.

Now that the B_1 pulse duration is also optimized, we can have a closer look at the capacity of the LCR circuit of the B_1 coil again. First it is necessary to know that the B_1 pulse is applied by a *rectangular* function and the fourie transformation of a *rectangular* function is a *sinc* function. Therefore the fourie ransformed spectrum of the B_1 pulse signal is a *sinc* function. When we measure the signal shortly (acquisition delay: 2 ms) after the 90° pulse there shoud be a *sinc* function visible and indeed this is what we obtained (figure **??**). In figure **??** there is also a really sharp peak visible. This is referd to the hydrogen signal. The hydrogen signal is independed of the applied capacity, but the B_1 pulse is, because the capacity changes the properties of the LCR-circuit of the B_1 coil. The best capacity is adjusted when the hydrogen signal is in the middle of the *sinc* function, because then the LC- circuit is tuned to the lamor frequency of the hydrogen signal. This is also visible by the amplitude of the spectrum in figure **??**. The ampitude of the spectrum which was observed for a capacity of 13.8 nF is higher than for the ampitude of the spectrum which was observed for a capacity of 14.2 nF. As already explained before in the chapter **??** the capacity of 13.8 nF is indeed the best capacity in order to observe a maximized spectrum.

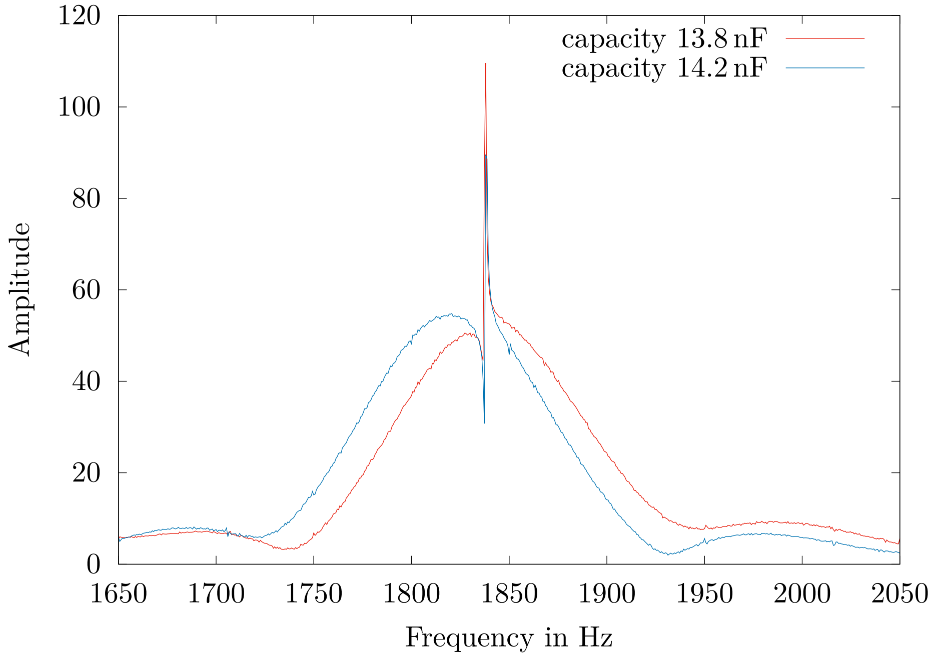

Figure 5.3: This figure shows the impact of the capacity in the LCR circuit of the B_1 coil. The *sinc* function comes from the fourie transformed B_1 pulse, which is rectangular. The peak at 1837.27(5) Hz is the peak from the hydrogen signal.

Now that the FID signal is optimized best we can start to characterize it. Therefore we measure a FID with a acquisition delay of 25 ms, because after this delay there are no effects from the rectangular applied B_1 pulse anymore (no *sinc* function in the spectrum). The figure **??** shows the observed spectrum and two different fit possibilities.
One option to fit a peak in a spectrum is by applying a *voigt*-profile ($V(x;\sigma,\gamma)$). This function is a convolution of the *Cauchy-Lorentz*- and *Gaussian*-distribution and is described by following formula:

$$V(x;\sigma,\gamma) = (G \star L)(x) = \int G(\tau)L(x-\tau)d\tau \tag{5.1}$$

$$G(x;\sigma) = \frac{exp\left(\frac{-x^2}{2\sigma^2}\right)}{\sigma\sqrt{2\pi}} \tag{5.2}$$

$$L(x;\gamma) = \frac{\gamma}{\pi\left(x^2+\gamma^2\right)} \ . \tag{5.3}$$

σ represents the standard deviation, γ is half of the peak width at half height from the *Lorentz*-distribution and x is the shift from the line center. In figure **??** the *voigt*-profile

(green) is fitted to the measured spectrum (red). The problem about this fit is that is not as sharp as the measured data. This might be, due to the fact that the measured spectrum does not have many data points especially around the maximum. Therefore the peak is really sharp and a correct fit with the *voigt*-profile is rather difficult. Therefore a second fit function has been apllied. This time only the *Gaussian*-distribution. This fit function is better to calculate the width of the peak, due to the fact that it easier to fit to this narrow peak. The full width of the peak at have maximum (FWHM) is calculated by the applied *Gaussian*-fit and is 1.180(40000) Hz.
The amplitude of the peak is 73.85 according to the *Gaussian*-fit and is in comparison to the amplitude of the noise measurement (magnitude 1) in figure **??** high. That means that the peak must come from the hydrogen signal is barely disturbed by any noise.
The disadvantage of the *Gaussian*-fit is that area under the curve does not equal the measured one, especially around 1836 Hz and 1839 Hz. Therefore the discussion about the integral under the measured curve will just be qualitative and will be done in the chapter **??**.
real and imaginary signal -> explain it
signal to noise ratio: what to do? -> magnitude, which unit is the amplitude, tutor will send us an email, try back fouriertransform (only keep real values)

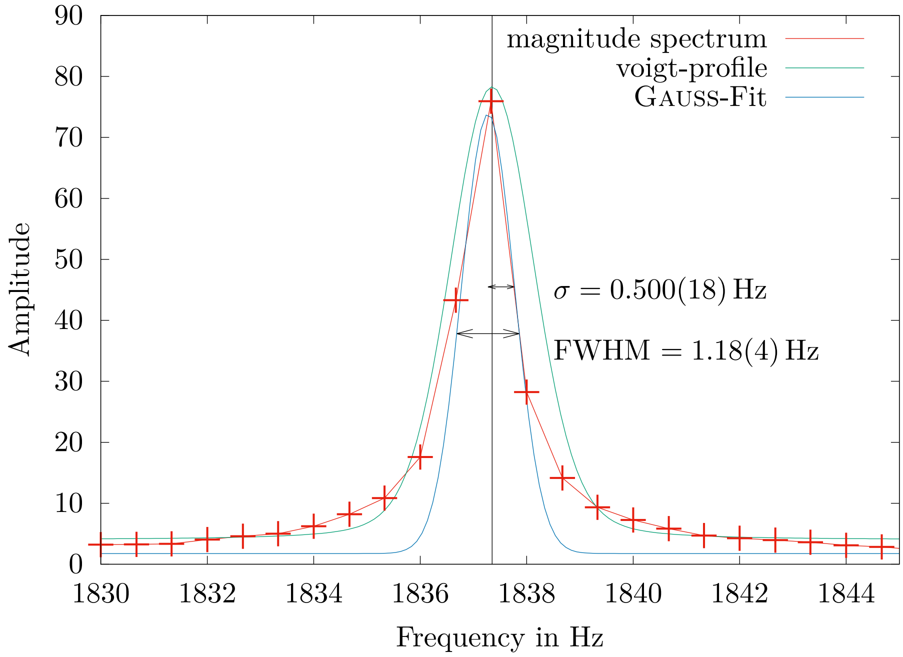

Figure 5.4: This figure shows the measured hydrogen signal after an acquisition delay of 25 ms and two possible ways to fit the peak. Due to a very short frequency range the peak looks very wide. Indeed it is actually very sharp. To fit the peak a *voigt*-profile and *Gaussian*-fit is used.

6 Longitudinal relaxation measurements T1

To measure the longitudinal spin lattice relaxation exist two possibilities. The first we want to have a closer look at is the measurement via τ_p (polarizing pulse duration). Therefore the computer program *Prospa* applies a polarizing pulse orthogonal to the earths magentic field. Due to this polarizing pulse the spins align in the transversal plain and form a bulk magnetisation. By time the magnetisation becomes stronger, therefore the signal becomes stronger. This relation is visualized in the figure **??**.

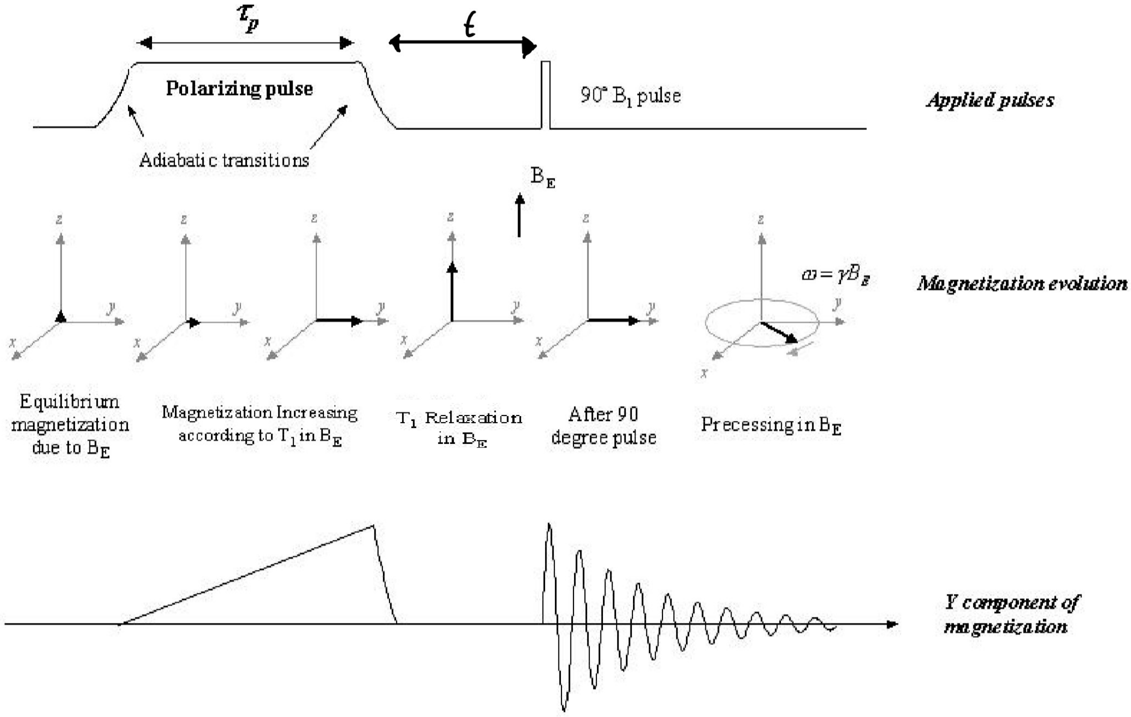

Figure 6.1: Sketch to show how T_1 can be measured. One way is by changing the polarizing pulse duration τ_p and the other way is by variy the time between the polarizing pulse and the 90° pulse. [?]

Due to the increasing magnetisation it is possible to calculate the $T_{1,p}$ relaxation. In order to do so the magnetisation time is increased step by step from 500 ms to 4500 ms in step sizes of 500 ms and in each configuration the signal maximum is calculated of the fourie transformed spectrum. Figure **??** shows the attenuation of the signals normalized to the maximum peak E_0. The reason of that is that by applying a fit function as followed:

$$S(x) = S_0 \cdot [1 - exp(\frac{-x}{T_{1,p}})] \ , \tag{6.1}$$

it is possible to calculate the relaxation time $T_{1,p}$. The exponential decay is a result of loss of phase coherence between the spins and will be used for every measurement of spin relaxation. In this case $T_{1,p}$ is 2912.8800(48) ms.

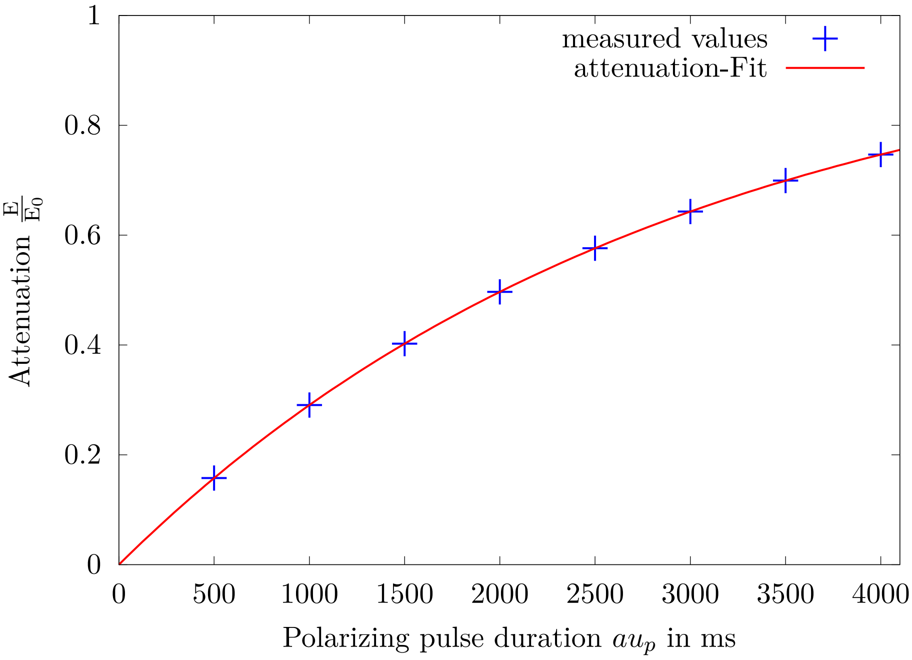

Figure 6.2: $T_{1,p}$ measurement by variy τ_p and see how the attenuation $\frac{E}{E_0}$ evolves. The provided exponential fit results in a value for $T_{1,p}$ of 2912.8800(48) ms.

The second option is to calculate the spin lattice relaxation via the earths magneitc field B_e. In this case the index will be e for the spin lattice relaxation. The procedure in this case is to change the time t (pre-90 minimum delay) between the polarizing pulse ends and the 90° pulse beginns. This relation is also visualized in the figure **??**. The pre-90 minimum delay is chosen as 0 ms and the pre-90 delay step size 500 ms. For every configuration the signal maximum is calculated again of the fourie transformed spectrum. Figure **??** shows the attenuation of the signals normalized to the maximum peak E_0. This time the $T_{1,e}$ can be calculated by following fit function:

$$S(x) = S_0 \cdot exp(\frac{-x}{T_{1,e}}) \ . \tag{6.2}$$

For our case $T_{1,e}$ is 2753.0500(12) ms.
In both ways the uncertainty of the T_1 values are really small. This is the result of really good align values to the fit function. Nevertheless $T_{1,p}$ and $T_{1,e}$ are not consistent even though the uncertainty is considered. This might be, due to the fact that those to measurements are based on two different methods and there can always be differences in the setup genauer anschauen. Even though they are not consistent, the values for $T_{1,p}$

and $T_{1,e}$ have the same magnitude.

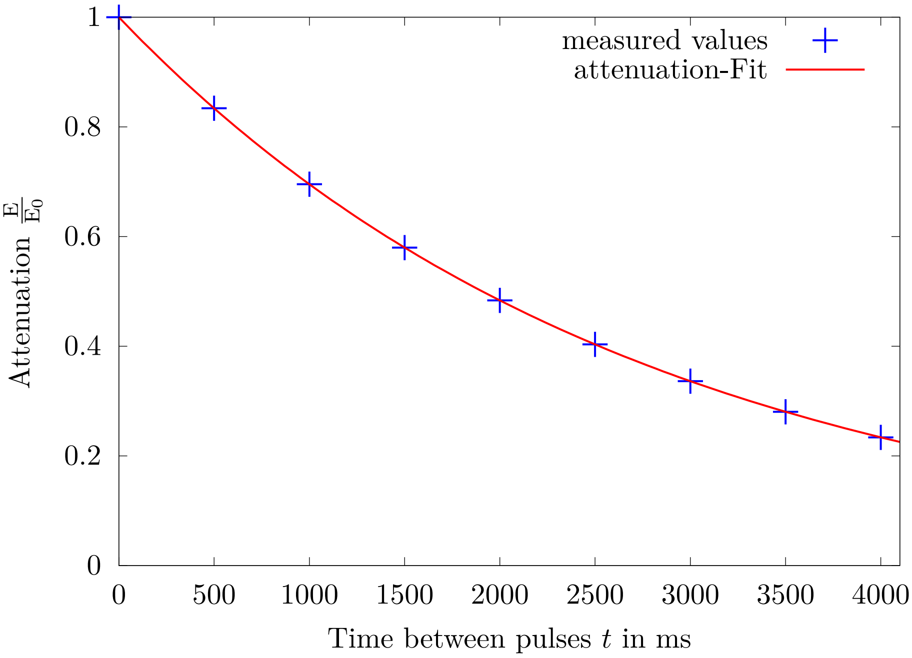

Figure 6.3: $T_{1,e}$ measurement by variy t and see how the attenuation $\frac{E}{E_0}$ evolves. The provided exponential fit results in a value for $T_{1,e}$ of 2753.0500(12) ms.

7 Hahn echo

The other relaxionmeasurement is the T_2 measurement. In order to understand that, we first have to explain the *Hahn* echo and the principle of multiple echo sequences.
The principle of the *Hahn* echo is that an 90° pulse is applied and after a certain time τ a 180° pulse. The reason behind this method is that after the 90° pulse the spins are oriented in the transversal plane and start to precise around the earths magnetic field vector (z-axis). Due to spin-spin interaction (inhomogenious magnetic field accrues) the spins also interact with each other and therefore some spins have a higher lamor frequency and some have a smaller one. If an 180° pulse is applied after a certain time the spins will flip in the transversal plane and the slow precessing spins will be before the fast precessing spins again and when the fast precessing overtake the slow ones again the B_1 coil will de detect a signal again. The reason why the B_1 coil does not detect something while the slow and fast precessing spins are at different position is that they erase each other. If the spin-spin interaction is too weak than it also helps to deshimm the system along the

x-direction. This also makes the homogenious magnetic field inhomogenious and thus the spins will get different lamor frequencies according to there position.
Figure **??** exemplarily shows the *Hahn* echo for a shimming value of 4.95 mA along the x-axis (original value 10.11 mA). It is also possible to change the time between the 90° and 180° pulse. This would shift the peak to higher times in the timescale and due to loss effects the amplitude will shrink a little bit.

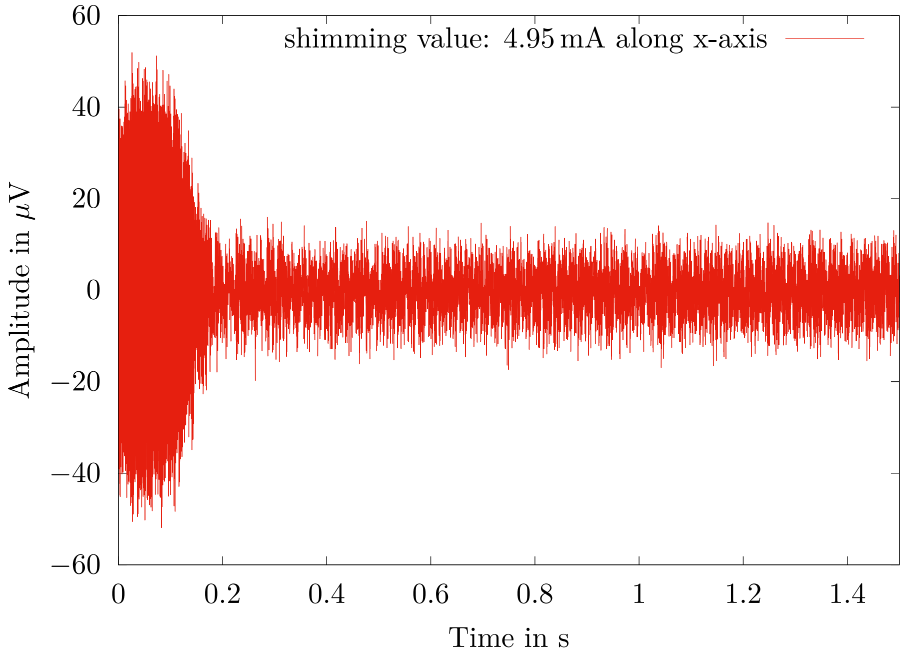

Figure 7.1: Example of a single *Hahn* echo for an echo time of 0 ms. The maximum of the echo is clearly visible. Due to relaxation after the maximum the signal after about 0.2 s is noise.

It is also possible to fourie transform the signal from figure **??**. Figure **??** shows this for two different shimming values. The amplitude of the spectrum with the shimming value of 0 mA along the x-axis is clearly smaller than the amplitude of the spectrum with shimming value of 4.95 mA along the x-axis. This effect comes from the more inhomogenious magnetic field of the spectrum with the shimming value of 0 mA along the x-axis. A more inhomogenious magnetic field also means that the spins have more different lamor frequencies and thus the total intensity will shrink. The area under the spectrum should be independent from the inhomogeneity, because in total the magnetisation has to be the same. Only the distribution is different. This effect is also really good visible in the figure **??**. This time it is not possible to find a good fitting function. Therefore this discussion is more qualitativ as mentioned before in chapter **??**. The reason why there is no good fitting function is that there are a lot of random peaks in the spectrum and the more peaks there are the more difficult it is to find a good fitting function. Another thing which makes it rather hard is that the frequency steps are not very small and thus there are not many datapoints to make a good fit. This was also a problem in chapter **??** as mentioned before.

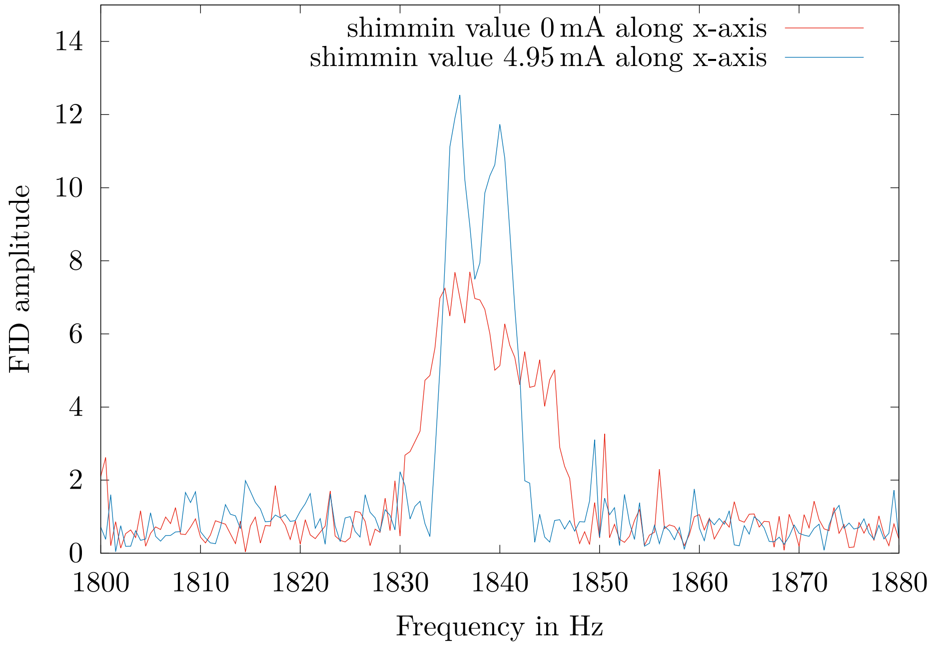

Figure 7.2: Spectrum of a single *Hahn* echo applied by different shimming values. Due to more deshimming of the red curve, the apmlitude is lower. Nevertheless the area under the spectrum is the same, due to the same magnetisation.

For the following chapter it is specifcally important to know which relaxation time we observe. Due to the inhomogenious magnetic field there exist different relaxation times of the transversal relaxation time T_2. The transversal relaxation time T_2^* describes the relaxation in consideration of the inhomogenious magentic field. Therefore the formula has following shape:

$$\frac{1}{T_2^*} = \frac{1}{T_2} + \gamma \Delta B_0 \ . \tag{7.1}$$

In this equation γ is the gyromagnetic ratio of the probe and ΔB_0 is the difference of the magnetic field to its equilibrium. Knowing that we now know that every time we deshimm the system we observe T_2^* and not T_2.

8 Multiple echo sequences

Beside the *Hahn* echo it is also possible to apply multiple *Hahn* echos in one experimental measurement. This method is called *Call-Purcell-Meiboom-Gill*-method (CPMG). Therefore the 180° pulse is applied every 2τ and thus there accur many maximums in the signal every 2τ. The reason to use the CPMG method is that it is possible to measure the amplitude of two consecutiv maxima more often and therefore the measurement of T_2 is more precise. More about this will be discussed in the next chapter.
To make the CPMG signals smoother in the time domain we do not use rectangular functions for the pulses, but smoothen them at the edge by a sine-bell-square function. This is possible, due to the fact that it does not change the physical properties of our measurements, but will make them smoother.
A main advantage of CMPG is that errors in the refocusing pulse can be vanished (minimize term of inhomogenious magnetic field), by changing the phase between the B_1 excitation and the refocusing pulses. The program *Prospa* profides a function called "Constant 180 pulse phase". This function keeps all the phases of the refocusing pulses equal. The second function *Prospa* provides is "Alternating 180 pulse phase". This function compensates echo errors by alternating the refocusing pulses by 180°. In figure **??** it is visible what a change in the 180 pulse phase does to the signal. Unfortunately we only saved the signal vor 180 pulse phases of 270° and 90°. For those two values the signal does not change. That is also the reason why there is only one signal visible. The other one is just directly behind the other one and therefore not visible. If we would have saved a pulse phase of 180° the signal should change to na zu was sollte es sich ändern?? schwächer?, was macht pulse phase eig genau.

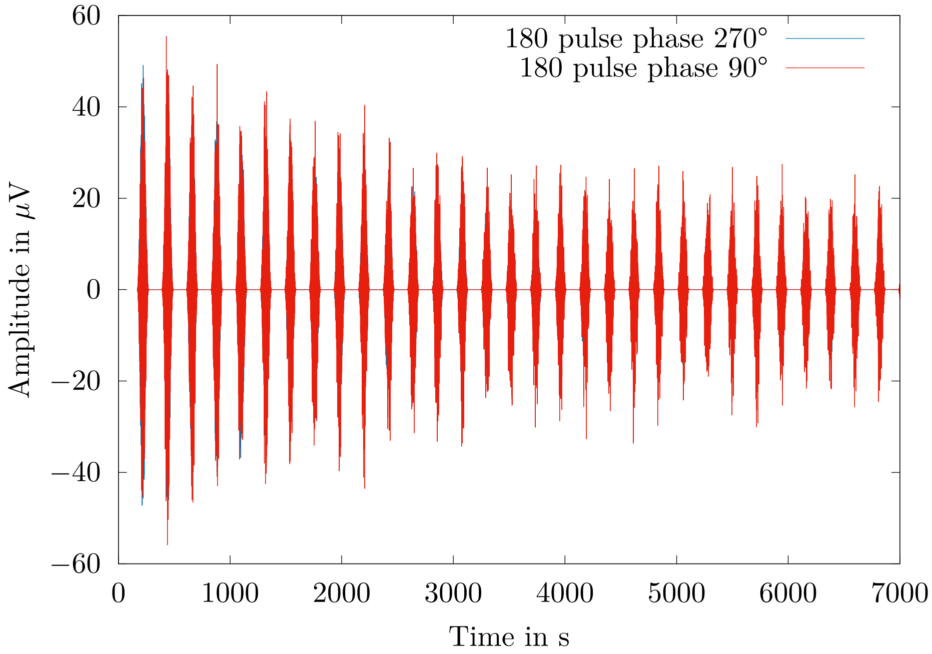

Figure 8.1: This figure shows the impact of the 180 pulse phase. Unfortunately we only saved data for a 180 pulse phase of 270° and 90° and for those values it is correct that the signal does not change, but a signal for a 180 pulse phase of 180° would have shown a different signal.

9 Transversal relaxation measurements

The last chapter in the first part of this experiment is the transversal relaxation measurement. In order to do so there are two possible ways again.
The first one is by one single *Hahn* echo (spin echo). Therefore the ratio between the maximum of the signal after the 90° pulse and the maximum after the echo (maximum after 2τ) provides the transversal relaxation time T_2. Figure **??** shows measurements for this method by different echo time steps of $2 \cdot 400\,\text{ms}$. The exponential decay is clearly visible, due to the already explained loss ofphase coherence between the spins. Therefore the fit of the datapoints the following formula has been used:

$$M(x) = M_0 \cdot exp(\frac{-x}{T_2}) \ . \tag{9.1}$$

This formula shows a T_2 relaxation time of 2691(12) ms. Remember that the phase coherence loss because of the spin spin relaxation is irreversible and is always obtained when measuring T_2.

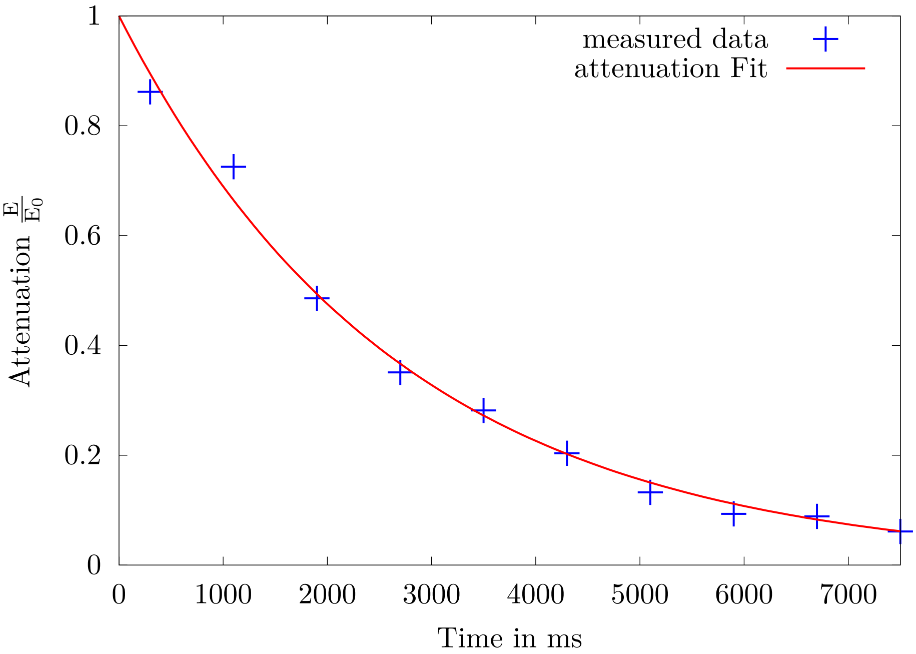

Figure 9.1: Attenuation $\frac{E}{E_0}$ for different echo times and exponential fit. The applied exponential fit results in a value for T_2 of 2691(12) ms.

One disadvantage of the T_2 measurement via one single *Hahn* echo is that the ratio of to back to back maxima is not that exact. Therefore the second option to measure T_2 is by using CPMG. Now that more maximums can be observed, the ratio of back to back maxima can be calculated more precisely. Therefore the result of T_2 is more exact using this method. Figure **??** shows measured data for 30 different echos. Due to the exponential decay the formula **??** has been used again to fit the measured data. This results a value for T_2 of 2317.760 00(62) ms.

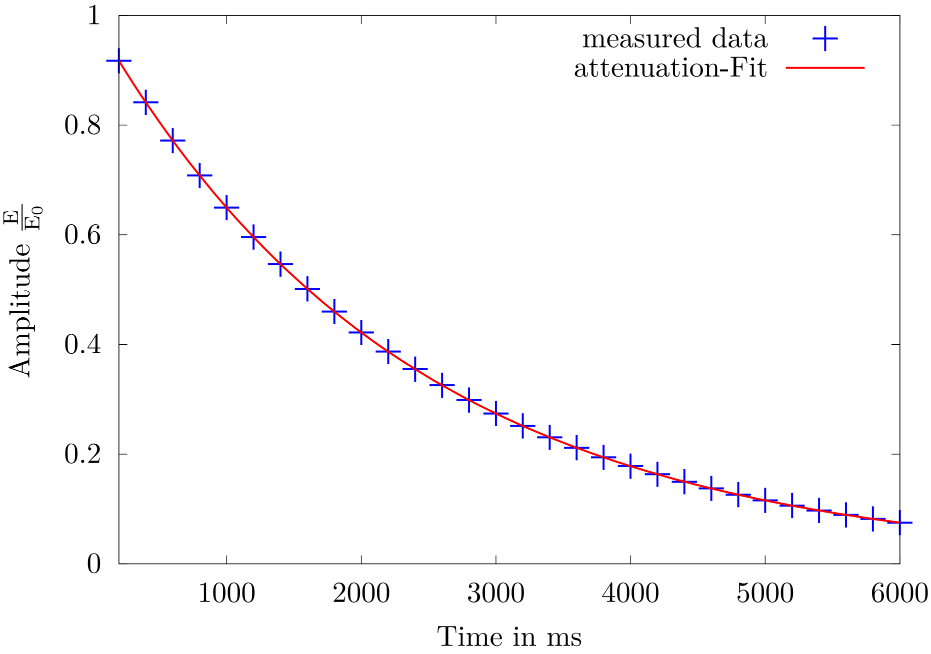

Figure 9.2: Attenuation $\frac{E}{E_0}$ for different echo maxima provided by the CPMG method. The applied exponential fit results in a value for T_2 of 2317.760 00(62) ms.

The difference of the two T_2 values might occur, due to deshimming the system for the CPMG method and therefore can always be some inaccurate pulse phases. Nevertheless note that the CPMG method is the more exact method to measure T_2, due to more back to back maxima. By measuring T_2 via the *Hahn* echo the inhomogeneity of the magnetic field is reversed, due to the 180° pulses.

10 Fehlerdiskussion und Fazit

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