Vienna University of Technology

Faculty of Physics

Laboratory III

Laboratory Report

Electron Spin Resonance

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Contents

1 Resonanceabsorbtion of a passive HF-Osscilator

1.1 Fundamentals

To detect electron spin resonance (ESR) in DPPH, the DPPH sample is placed in an RF coil that is part of a resonant circuit with high quality factor . This circuit is excited by a variable-frequency RF oscillator operating between 15 and 130 MHz.

When the resonance condition for ESR is met at a frequency ν_0, the DPPH sample absorbs energy, which loads the resonant circuit. As a result, the AC resistance increases, and the voltage across the RF coil decreases.

To detect this change, a passive resonant circuit is used for comparison. Its coil is placed coaxially opposite the empty RF coil. The resonance frequency of the passive circuit is given by:

$$\nu_0 = \frac{1}{2\pi \cdot \sqrt{L_2 C_2}} \tag{1}$$

Where L_2 and C_2 are the inductance and capacitance of the oscillation circuit. The resonance frequency of the circuit can be adjusted by changing the capacitance C_2.

When the active circuit is driven at its resonance frequency ν_0, it is dampened, and the voltage U_1 across the RF coil decreases. The ESR signal is detected by measuring the rectified voltage U_1, which corresponds to the current I_1 through a measurement resistor $R_1 = 56\ \text{k}\Omega$:

$$U_1 = 56\ \text{k}\Omega \cdot I_1 \tag{2}$$

[1]

1.2 Setup

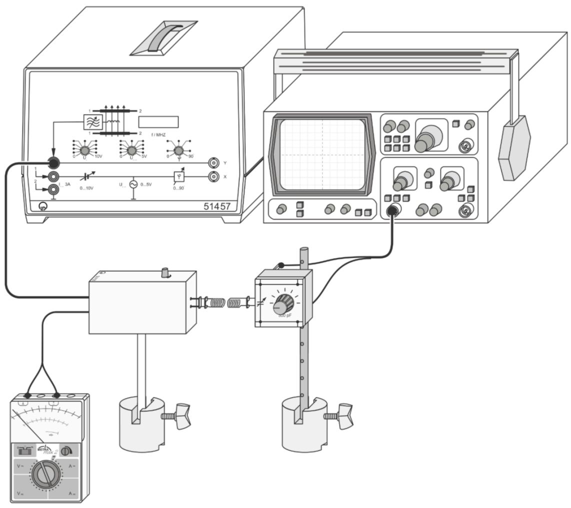

Figure 1: Experimental setup with the ESR base unit and an inductively coupled passive resonant circuit. [1]

- Connect the ESR base unit to the ESR operating unit via a 6-pin cable and set the rotary potentiometer on the top left to maximum sensitivity.
- Plug in the 13-30 MHz plug-in coil (large).
- Due to a bad connection of the measurement cable of I_1 the current and voltage of the active circuit could not be measured.

- Position the coil of the passive resonant circuit coaxially opposite of the active coil and connect via a BNC/4 mm measurement cable to channel I of the dual-channel oscilloscope.

[1]

1.3 Procedure

- Set the variable capacitor of the passive resonant circuit to position Skt. = 3/6.
- Adjust the minimum frequency on the ESR base unit.
- At the operating frequency, measure and record:
  - the frequency,
  - the voltage U_2 of the "passive" coil on the oscilloscope,
- Increase the frequency in steps and repeat the measurement.
- Perform additional measurement series with Skt. = 2/6 and 1/6.
- A measurement without the passive circuit could also not be done due to the bad connection of the active circuit cable.

[1]

1.4 Measurement values

The active coil voltage $U_1(\nu)$ could not be recorded, because the current measurement branch of the apparatus was broken during the time of the experiment. As a result, no data for U_1 were obtained, and only the passive coil voltage $U_2(\nu)$ is available for analysis. This won't be hindering the determination of the resonance frequencies of the LC-Oscillator, since it can be read out of the U_1 dataset in isolation.

ν / MHz	U_2 / V	ν / MHz	U_2 / V
11.5	1	15.5	2.35
12	1.01	16	2.2
12.5	1.15	16.5	2
13	1.2	17	1.8
13.5	1.4	17.5	1.25
14	1.6	18	1
14.5	1.8	18.5	0.8
15	2.2	19	0.7

Table 1: Measured voltage U_2 at fixed frequencies ν at scale division 3/6

ν / MHz	U_2 / V	ν / MHz	U_2 / V
11.5	0.8	18	1.5
12.5	0.85	18.5	1.6
13.5	0.9	19	1.7
14.5	0.97	19.5	1.6
15.5	1.05	20	1.5
16.5	1.2	20.5	1.35
17	1.3	21	0.95
17.5	1.4	21.5	0.8

Table 2: Measured voltage U_2 at fixed frequencies ν at scale division 2/6

ν / MHz	U_2 / V	ν / MHz	U_2 / V
18	0.75	24.5	1.15
19	0.8	25	1.1
20	0.85	25.5	1.05
21	0.9	26	1
22	0.95	26.5	0.95
23	1	27	0.9
23.5	1.05	27.5	0.8

Table 3: Measured voltage U_2 at fixed frequencies ν at scale division 1/6

1.5 Data analysis

After the plotting of the Voltage data, peaks are identified for simplicity only using the maximum Voltage value of each dataset. This might introduce slight errors, since the actual peak could be situated slighty shifted on either side of the measured peak. To get a more accurate peak information peak finding methods could be used.

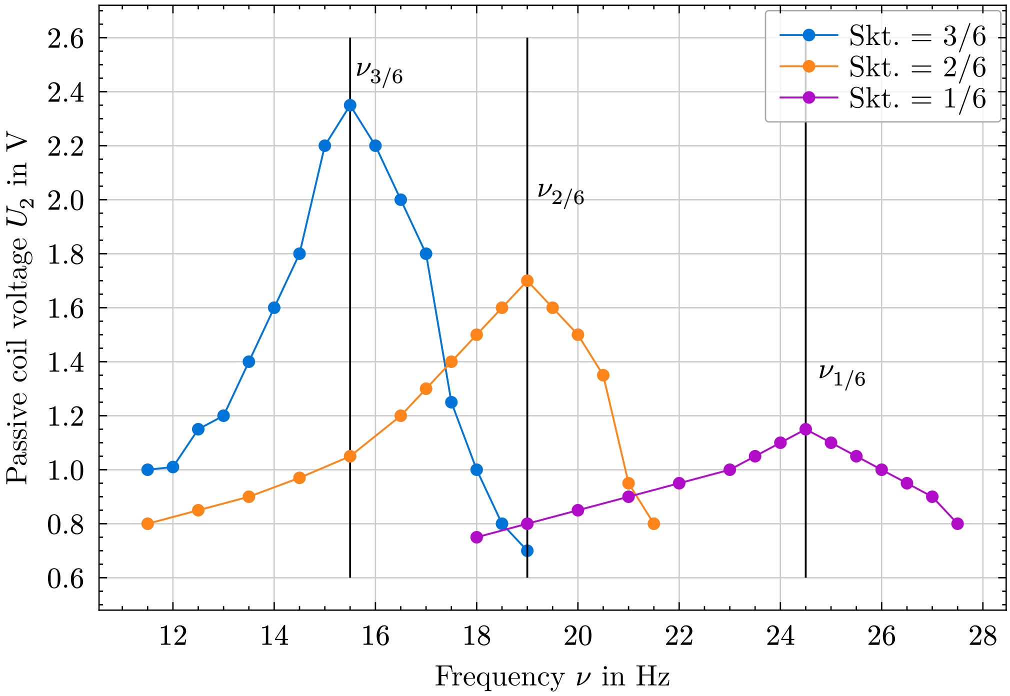

Figure 2: Passive coil voltage $U(\nu)$ vs frequency ν, with resonance frequencies $\nu_{3/6}, \nu_{2/6}$ and $\nu_{1/6}$ indicated by vertical lines. (see: Table 1 - Table 3)

The measured $U_2(\nu)$ curve exhibits three clear peaks at approximately 16 Hz, 19 Hz, and 25 Hz, which should align with the expected LC resonance modes $\nu_{3/6}, \nu_{2/6}$ and $\nu_{1/6}$. Each maximum marks a frequency at which energy oscillates most efficiently between the coil's inductance and its capacitance. Between these resonances, the voltage falls off, reflecting the reduced impedance mismatch away from the natural oscillation frequencies.

2 Electronspinresonance on DPPH

2.1 Fundamentals

Electron spin resonance (ESR) detects transitions between spin states of unpaired electrons in a magnetic field B_0. The energy levels split due to the Zeeman effect, and when electromagnetic radiation of the right frequency ν is applied, resonant absorption occurs.

This resonance condition is:

$$h\nu = g\mu_B B_0 \tag{3}$$

Where h is the planks constant, ν is the radiation frequency, g is the lande g-factor, μ_B the Bohr magneton and B_0 the static magnetic field B_0.

From this, the magnetic field B_0 can be calculated using:

$$B_0 = \frac{h\nu}{g\mu_B} \quad \rightarrow \quad g = \frac{\nu}{B_0} \cdot \frac{h}{\mu_B} \tag{4}$$

The magnetic field B of the Helmholtz coils can be calculated from the current I through each coil using the following formula:

$$B_0 = \mu_0 \cdot \left(\frac{4}{5}\right)^{\frac{3}{2}} \cdot \frac{n}{r} \cdot I \tag{5}$$

where $\mu_0 = 4\pi \cdot 10^{-7} \frac{\mathrm{Vs}}{\mathrm{Am}}$ is the vacuum permability, n the number of turns per coil and r the coil radius.

For $n = 320$ and $r = 6.8$ cm this yields:

$$B_0 = 4.23 \frac{\mathrm{mT}}{\mathrm{A}} \cdot I \tag{6}$$

[1]

2.2 Setup

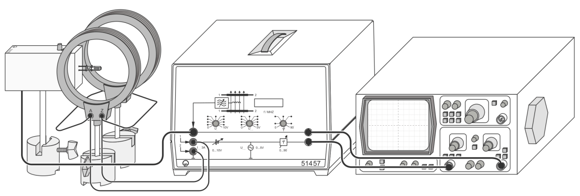

Figure 3: Experimental setup for electron spin resonance [1]

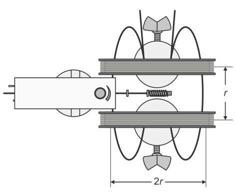

Figure 4: Arrangement of the Helmholtz coils and the ESR base unit, viewed from above. [1]

- Place the Helmholtz coils parallel to each other at a center distance of 6.8 cm (equal to the mean radius r).
- Connect both Helmholtz coils in series with the ammeter to the ESR operating unit.
- Connect the ESR base unit to the ESR operating unit via a 6-pin cable.

- Connect output Y of the ESR operating unit via a BNC cable to channel I of the dual-channel oscilloscope, and output X to channel II.

[1]

2.3 Procedure

2,2-diphenyl-1-picrylhydrazyl (DPPH) is used as a stable free radical sample. The magnetic field is generated by Helmholtz coils and modulated at 50 Hz. A high-Q RF resonant circuit detects the absorption via a drop in voltage when resonance occurs.

Determination of the Resonance Magnetic Field B_0

- Insert the 30-75 MHz plug-in coil (medium) and place the DPPH sample in the coil.
- Switch on the ESR base unit and position it so that the plug-in coil with DPPH sample is in the center of the Helmholtz-coil pair (see Figure 4).
- Set the resonance frequency $\nu = 30$ MHz.
- Set the modulation amplitude U_{mod} to the second scale division.
- Set the phase shift to 0°.
- Operate the oscilloscope in dual-channel mode:
  - Dual on
  - Time base $2\frac{\text{ms}}{\text{cm}}$
  - Amplitude I and II $0.5\frac{\text{V}}{\text{mm}}$ AC
- Slowly increase the DC voltage U_0 to the Helmholtz coils until the resonance signals are equidistant.
- Switch the oscilloscope to XY mode and adjust the phase shift so that the two resonance peaks coincide.
- Vary U_0 until the resonance signal is symmetric, keeping the modulation voltage as low as possible. (see Figure 5)

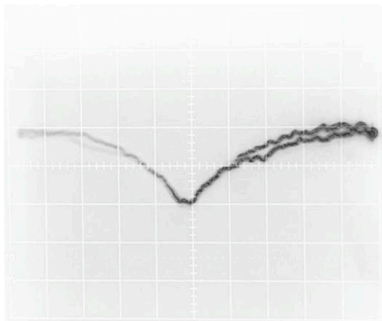

Figure 5: Symmetric and equidistant resonance signal

- Measure the DC current $2I_0$ through the Helmholtz-coil pair and record it together with the resonance frequency ν.
- Increase ν by 5 MHz and adjust U_0 to reestablish resonance.
- Again measure and record the current $2I_0$.
- Continue raising ν in 5 MHz steps (switch to the 75-130 MHz coil (small) at 75 MHz) and repeat the measurements.

[1]

Determination of the full width at half maximum (FWHM) δB_0

- Operate the oscilloscope in XY mode:
  - Amplitude II $0.05\frac{\text{V}}{\text{mm}}$ AC

- Reestablish the resonance condition for $\nu = 50$ MHz (medium plug-in coil).
- Vary the modulation voltage U_{mod} until the resonance trace spans the full screen width (10 cm) in the X-direction.
- Switch the ammeter to AC mode and measure the effective current $2I_{\text{mod}}$ corresponding to U_{mod}.
- Increase the X-deflection, read off the width ΔU of the resonance peak at half its height, and record it.

[1]

2.4 Measurement values

ν / MHz	$2I_2$ / A	Plug-in coil	ν / MHz	$2I_2$ / A	Plug-in coil
30	0.53	middle	80	1.41	middle
35	0.63	middle	80	1.53	small
40	0.71	middle	90	1.65	small
45	0.79	middle	95	1.67	small
50	0.89	middle	100	1.7	small
55	0.97	middle	105	1.74	small
60	1.06	middle	110	1.79	small
65	1.15	middle	115	2.05	small
70	1.23	middle	120	2.16	small
75	1.33	middle			

Table 4: Current $2I_0$ at given frequency ν of the magnetic field

Using Equation 6 the corresponding magnetic field for every current can be calculated, noting that the current values are in $2I_2$ resulting in an additional factor of $\frac{1}{2}$. This results in the values shown in Table 5:

ν / MHz	B_0 / mT	ν / MHz	B_0 / mT
30	1.12	80	2.98
35	1.33	80	3.24
40	1.5	90	3.49
45	1.67	95	3.53
50	1.88	100	3.6
55	2.05	105	3.68
60	2.24	110	3.79
65	2.43	115	4.34
70	2.6	120	4.57
75	2.81		

Table 5: Magnetic field B_0 as a function of frequency ν of the magnetic field

Measured voltage FWHM: $\delta U = 0.95$ V

2.5 Data analysis

Using a simple linear-regression model a linear function can be fitted onto the values of the magnetic field, where the slope corresponds to $\frac{\Delta\nu}{\Delta B_0}$ is in the linear case is equal to $\frac{\nu}{B_0}$. Which will make it possible to use in Equation 4 to calculate the g-factor of the probe.

The uncertainty introduced through the linear fit is around $\pm 0.77 \frac{\text{Mhz}}{\text{mT}}$, uncertainty of I or B_0 measurement does not affect the g-factor, since we are purely using the fitted slope of $\nu(B_0)$. Also even tho the intercept is expected to be 0, there might be miss calibrations which result in a y-shift of the curve, resulting in a non-zero intercept. This y-shift should not have any influence on the value of the slope.

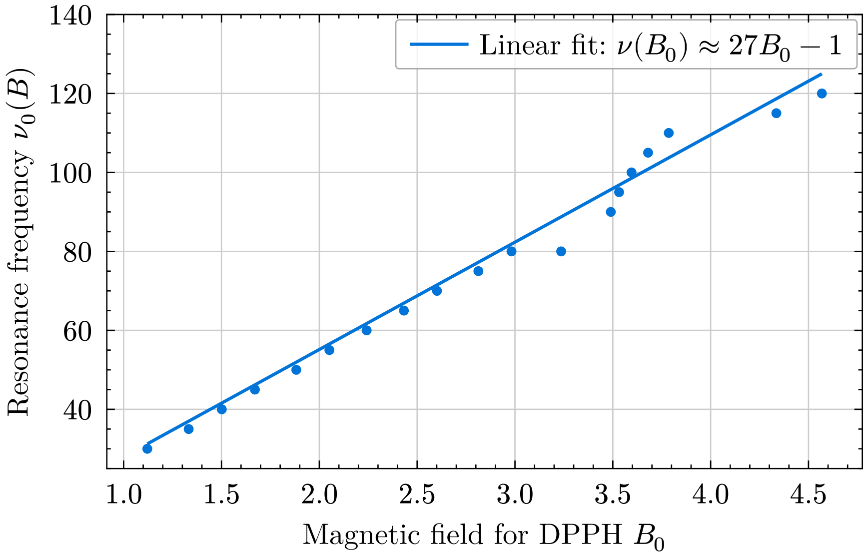

Figure 6: Resonance frequency as a function of the magnetic field for DPPH (see Table 5)

FWHM calculation:

Using the FWHM of the voltage U we can determine the FHWM of B_0 using the following equations:

$$\delta B_0 = 4.23 \text{ mT} \cdot \delta I = 4.23 \text{ mT} \cdot \frac{\delta U}{U_{\text{mod}}} \cdot I_{\text{mod}} \tag{7}$$

thus the FWHM of the magnetic field yields:

$\delta B_0 \approx 0.33$ mT, compared to common values of $\delta B_0(\text{DPPH}) = 0,15 - 0,81$ mT.

g-factor calculation:

In experiment determined g-factor using Equation 4 combined with the slope of the linear fit:

$g = 1.94 \pm 0.06$ compared to the literature value of $g = 2.0036$

The linear relation $\nu \propto B_0$ confirms Zeeman splitting of DPPH electron spins. The extracted $g = 1.94 \pm 0.06$ agrees well with the literature, and the measured field half-width $\delta B_0 \approx 0.33$mT reflects intrinsic line broadening due to spin–spin relaxation and field inhomogeneities and lies in the range of expected values.

List of Tables

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Bibliography

[1] H. Michor, “Elektronenspinresonanz,” LD Didactic GmbH, 2025.