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LABORATORY III

Laboratory Report

Electron Spin Resonance

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1 Resonanceabsorbption of a passive HF-Oscillator

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:

$$\nu_0 = \frac{1}{2\pi \cdot L_2 C_2} \quad (1)$$

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 $R_1 = 56 \text{ k}\Omega$:

$$U_1 = 56 \text{ k}\Omega \cdot I_1 \quad (2)$$

1.2 Setup

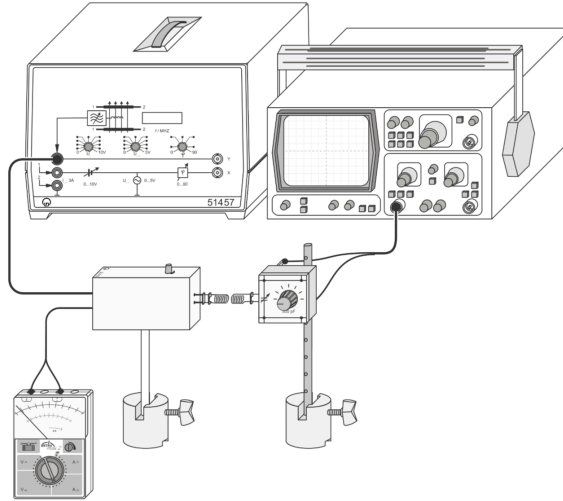


Figure 1: Experimental setup with the ESR base unit and an inductively coupled passive resonant circuit. [1]

- 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.

1.3 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.

1.4 Measurement values

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: Voltage U_2 at Skt. = 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: Voltage U_2 at Skt. = 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: Voltage U_2 at Skt. = 1/6

1.5 Data analysis

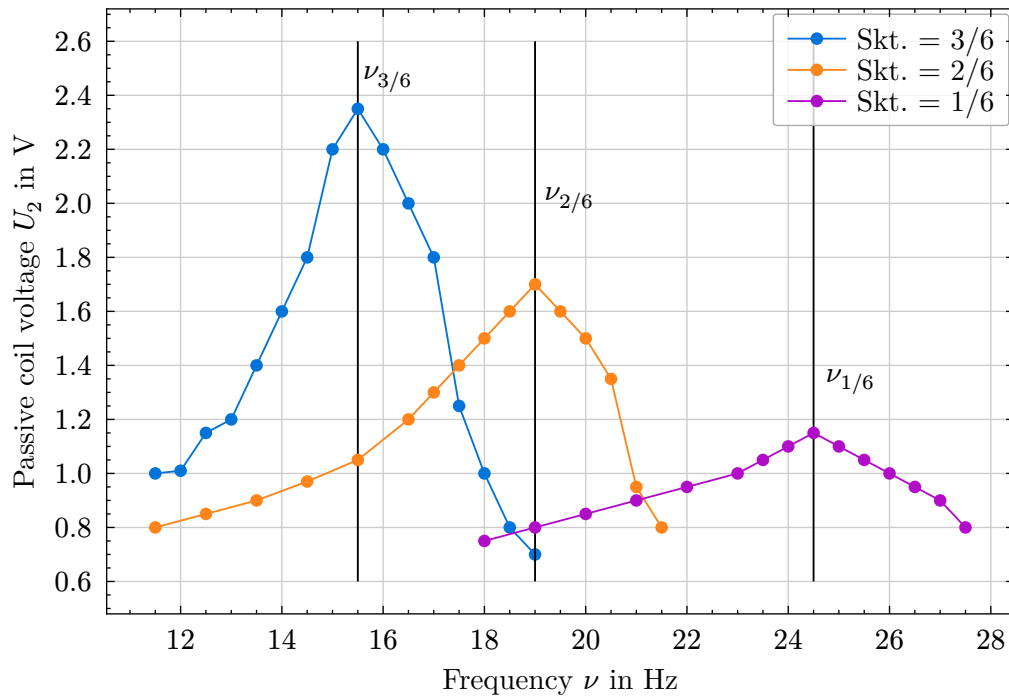


Figure 2: The resonance frequencies ν_n can be determined by determining voltage peaks in the passive coil voltage $U_2(\nu)$. The measurement apparatus for the current through the active coil was broken. Thus, the active voltage $U_1(\nu)$ couldn't be determined. It would usually correspond to a damped oscillation thus creating corresponding local minima in U_1 at the same resonance frequencies

1.6 Interpretation

2 Electrons spin resonance 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:

$$h\nu = g\mu_B B_0 \quad (3)$$

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:

$$B_0 = h\nu g\mu_B \quad (4)$$

For a free electron: $g = 2.0023$

Spin quantum number: $J = s = \frac{1}{2}$

ESR transition: $\Delta m = J = \pm 1$

The ESR line width ΔB_0 relates to level lifetime T via the uncertainty relation:

$$\Delta B_0 = \frac{1}{2}g\mu_B T \quad (5)$$

2.2 Setup

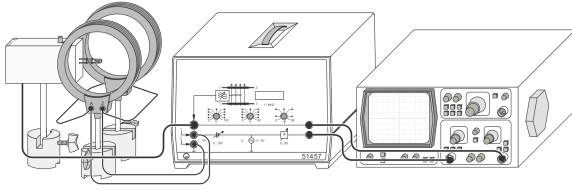


Figure 3: Experimental setup for electron spin resonance [1]

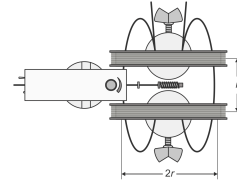


Figure 4: Arrangement of the Helmholtz coils and the ESR base unit, viewed from above. [1]

- 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.

2.3 Procedure

DPPH is used as a stable free radical sample with $g \approx 2$. 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 Fig. 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 (see Fig. 6).
- 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.
- 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.

Determination of the Half-Width 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.

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 $2I_0$ as a function of frequency ν of the magnetic field

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

2.5 Data analysis

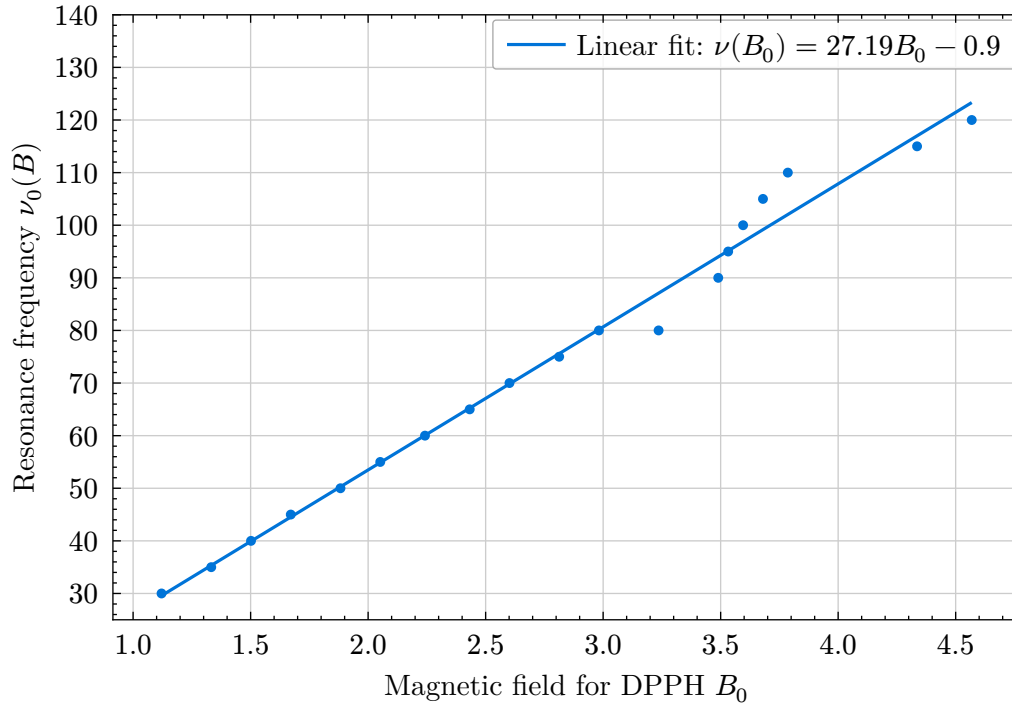


Figure 5: Resonance frequency as a function of the magnetic field for DPPH

Measured voltage half-width: $\delta U = 0.95$ V

First using

$$\delta I = \frac{\delta U}{U_{\text{mod}}} \cdot I_{\text{mod}} \quad (6)$$

the calculated current half-width yields: $\delta I = 0.078$ A

Further using:

$$\delta B_0 = 4.23 \text{ mT} \cdot \delta I \quad (7)$$

the half-width of the magnetic field yields: $\delta B_0 = 0.33$ mT

Laboratory Work III - Electron Spin Resonance

Slope: $27.19 \frac{\text{MHz}}{\text{mT}}$

In experiment determined g-factor:

$$g = 1.9426$$

g-factor from literature:

$$g = 2.0036$$

Despite a deviation from linearity in the higher frequency range when using the smallest coil, a linear relationship between resonance frequency and magnetic field strength is observed. Furthermore, the measured g-factor is in sufficient agreement with the literature value.

2.6 Error Estimation

Since the voltage values were read from a oscilloscope, the error of these values is ± 0.1 V. Since the Current was measured by an amperemeter, the error for the current is ± 0.01 A.

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Bibliography

- [1] H. Michor, "Elektronenspinresonanz," LD Didactic GmbH, 2025.