Z6 - Continuous Wave NMR

Physics Laboratory II
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By means of a Continuous Wave NMR spectrometer the student will measure spectra of various solid or liquid state samples, including e.g. $Co(NH_3)_6(BF_4)_3$ or its derivatives, at varying temperatures. From the thermal dependence of the spectral line shapes, information on the movement of protons or fluorine nuclei will be derived, and therefore on the rotational dynamics of the molecular groups incorporating those elements.

Preparatory questions

- Applications of Nuclear Magnetic Resonance (NMR).
- What is needed to observe the NMR phenomenon? [1, 2]
- Pulsed method versus the continuous wave method. [1, 2]
- In what kind of materials can NMR be observed? The condition for the NMR phenomenon to occur. [1, 2]
- Nuclear energy levels. Energy level transitions. [1, 2]
- Definition of magnetic field and the basic information concerning the subject. [1, 2]
- Definition of magnetization. The motion formula of the magnetization vector in external magnetic field. [1, 2]
- Longitudinal and transverse relaxation. T_1 and T_2 relaxation times. [1, 2]
- Definition of chemical shift and local field. [2]
- Basic information on phase transitions in solid state. [3]
- The Arrhenius formula. Activation energy. [3]

Attention: some of the problems are not directly related to the laboratory class, however, the knowledge of them, even perfunctory, is useful to obtain complete insight into NMR as a research method. Specifically,

- Spin echo definition. [1, 2]
- Magnetic Resonance Imaging and contrast. [1, 2]

Computational assignments

1. From the condition for resonance, calculate the magnetic field in which resonance for protons will be observed. Assume $\nu = 20$ MHz. [1, 2]
2. Calculate the maximum value of the local field in the water molecule. [6]

Apparatus and materials

The experimental setup consists of (see Fig. 2)

- An electromagnet,
- An electronic panel that includes: a magnetometer, temperature controller, spectrometer and electromagnet power supply
- A computer to control the experiment and analyze the data.

Experiment

- Determine the electromagnet characteristics (the dependence of magnetic field on the current).
- Prepare the sample(s).
- Perform measurements of NMR spectra at various temperatures in the vicinity of the phase transition.
- By means of the spectrometer software determine the second moment of the spectral lines and produce a plot of its thermal dependence. [4, 5, 6]
- The students may also perform measurements of various substances proposed by themselves.

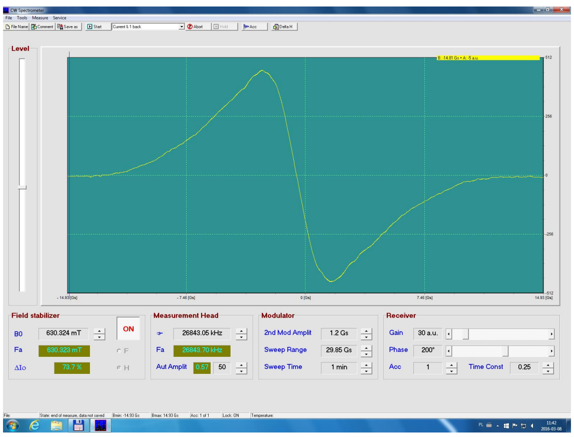

Figure 1: A typical registered resonance spectrum visible at the computer screen.

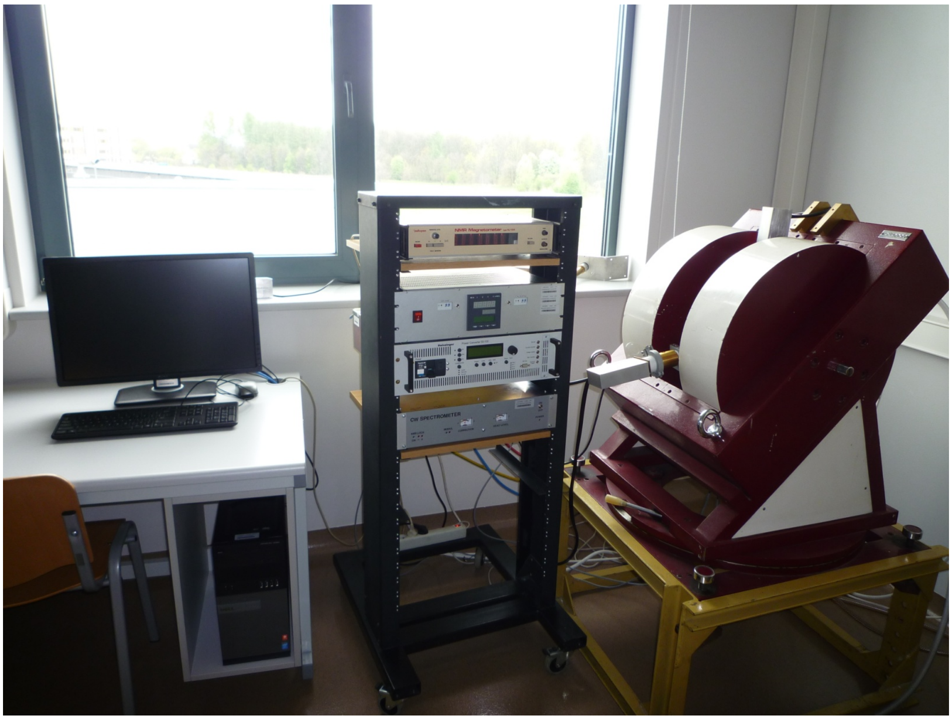

Figure 2: Experimental setup of the continuous wave spectrometer. From the right: electromagnet; electronic panel including (from the top): magnetometer, temperature controller, the spectrometer and electromagnet power supply with good current stabilization; the computer controlling the experiment.

Data analysis

- Determine the peak position characteristic for the given sample for each measurement. Pay close attention to the fact that the peaks are always overlapped with the background, and in some of the cases the measured peaks from the sample itself overlap with each other. Due to this, in some cases the fitting functions must be a composition of a Gaussian function (or Lorentzian function) describing the peak position with a second order polynomial which describes the background.
- Estimate the spin-spin relaxation times at each temperature from the FWHM (Full Width Half Maximum) of the resonance lines.
- Plot the relaxation times vs. temperature to determine the transition temperature.

References

[1] *Handbook of Analytical techniques*, edited by Helmut Günzberg and Alex Williams, Wiley-VCH, 2001

[2] *Handbook of Instrumental Techniqeus of Analytical Chemistry*, edited by Frank A. Settle, Prentice Hall PTR, 1997

[3] *Solid State Physics*, Charles Kittel, John Wiley & sons, 2005

[4] G.R. Murray, J.S. Waugh, J. Chem. Phys. 29, 207 (1958)

[5] N. Kummer, J.L. Ragle, N. Weiden and A. Weiss, Z. Naturforsch, 34a, 333-339 (1979)

[6] Full instruction materials available at the Laboratory website after logging in.