Letters to the Editor

INVESTIGATION OF NUCLEAR MAGNETIC RESONANCE IN ADSORBED GAS

A. A. GALKIN and I. V. MATYASH

Institute of Radiophysics and Electronics, Academy of Sciences, Ukr. S.S.R.

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COMPARATIVELY little work has been devoted to the study of physical properties of adsorbed gas. Indeed, only isothermal adsorption and heat capacity of thin films of gas have been studied in any degree of detail.[1-4] Therefore, it is of definite interest to carry out investigations of nuclear magnetic resonance (n.m.r.) in thin films of a substance, since from the form of the resonance curves it is possible to evaluate the interaction between the molecules of the adsorbed gas and to study the effect of the substratum.

In order to study the problems indicated above, n.m.r. investigations were undertaken in thin films of hydrogen, water, and methane adsorbed by activated charcoal. The investigations were carried out by means of the spin-echo method,[5] which enables us to measure in a comparatively simple way the longitudinal and the transverse relaxation times (T_1 and T_2), and also to evaluate the self-diffusion coefficients.

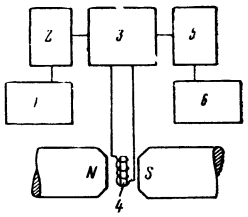

FIG. 1

The block diagram of the apparatus is shown in Fig. 1. The rectangular pulse generator 1 enabled us to obtain: a) two or three pulses with independent control of their length, repetition frequency and the time interval between them; b) a single pulse, followed by a series of equidistant pulses with independent control of the pulse length and the time interval between them. The pulse generator modulates the radiofrequency oscillator 2 stabilized by a quartz crystal and operating at a frequency of 14 Mcs. In order to avoid overloading the radiofrequency amplifier the latter is separated from the oscillator by the r-f bridge 3 one of whose arms contains the coil with the sample 4; the r-f amplifier 5 has a gain of $\sim 10^5$. The signal from the detector was applied to the input 6 of an IO-4 oscillograph.

A magnetic field of 3300 oe was produced by a permanent magnet with pole pieces 110 mm in diameter and with a 40 mm gap. The magnet was provided with coils which made it possible for the magnetic field to be varied by ± 50 oe.

This apparatus enabled us to measure the longitudinal and transverse relaxation times (T_1 and T_2) in the range $10^{-4} - 10$ sec.

For the determination of T_1 three pulses were applied to the sample and the relaxation time was determined by analyzing the intensity of the stimulated echo signal as a function of the time between the first and the third pulses.[5] A study of the intensity of the spin echo signal as a function of the time between two pulses enabled us to calculate the relaxation time T_2 and the self-diffusion coefficient. Moreover, the dependence of the intensity of the spin echo on the time elapsed from the application of a single orienting pulse and a series of equidistant pulses enabled us to determine T_2 directly.[5]

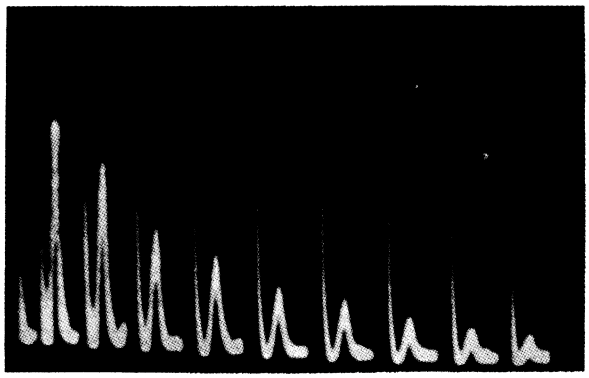

FIG. 2

As an illustration Fig. 2 shows a spin echo oscillogram from which the relaxation time T_2 was determined for hydrogen adsorbed on charcoal at a temperature of 77° K.

The table summarizes data on the measurement of the relaxation times T_1 and T_2 for hydrogen, methane, and water adsorbed on charcoal, and also an evaluation of the self-diffusion coefficient (D), and of the activation energy (Q) obtained by the n.m.r. method at different temperatures.

From the table it may be seen that at a temperature of 77° K the width of the n.m.r. line in hydrogen adsorbed on charcoal is 0.2 oe, while according

Substance	T, °K	$10^3 T_1$, sec	$10^3 T_2$, sec	ΔH, oe	$10^2 \cdot D$, cm²/sec	Q, joule/mole	Notes
	90	2	1.4	0.2	2.7		Monomolecular
	77	5	1.3	0.2	2.4	590	layer
H_2	20.4	10	0.1	2			
	20.4	20	8	0.03	$2 \cdot 10^{-2}$		Liquid film
	14_{liq}	200	200	10^{-3}			In free state
H_2	14_{sol}		2.3	0.1	0.1	1200	according to data
	11		0.23	1.0			of reference 6.
	290	20	1.9		4		Monomolecular
CH_4	90	8	2.1		2.5	760	layer
	77	7	2.6		2.1		
		1	0.7				10 Steam pressure
		2	0.7				20 over charcoal
H_2O	290	10	5				70 in mm Hg
		15	6				170
		3000	2000				In free state

to Bloom's data[6] hydrogen has an absorption line of 0.1 oe width only near its freezing point (liquid hydrogen gives a line width of 10^{-3} oe). It should be pointed out that at a temperature in the neighborhood of 25° K the n.m.r. line width in hydrogen adsorbed on charcoal increases to 2 oe. It is of definite interest to make an estimate of the coefficient of internal friction for water. According to n.m.r. data the viscosity of water in the adsorbed state increases by more than an order of magnitude.

Thus, an analysis of the results of this investigation shows that physical properties of adsorbed gases can be successfully studied by the n.m.r. method which significantly extends the range of possibilities in the study of molecular physics.

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OBSERVATION OF SPONTANEOUS COHERENT RADIATION OF A FERRITE IN A RESONATOR

A. P. ALEKSANDROV, Ya. I. KHANIN, and É. G. YASHCHIN

Radiophysics Institute, Gor'kiĭ State University

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In recent years there have been published a number of researches related to spontaneous coherent radiation of electron spin systems in the microwave range. In the majority of cases,[1-3] generation is accomplished by use of paramagnetics that possess, at liquid-helium temperatures, very long spin-lattice relaxation times (from milliseconds to minutes). Such a choice is not fortuitous, for the duration of the relaxation processes determines the time scale of the experiment. With long relaxation times, it is easy to excite the spin system by the method of adiabatic rapid passage or with the aid of 180° pulses. A similar method (a 45° pulse) has been successfully applied also to the excitation of organic free radicals[4] possessing relaxation times from 0.03 to 0.1 μsec.

For the overwhelming majority of ferrites, the relaxation times τ, estimated from the width of the ferromagnetic resonance line, are much smaller than the figures mentioned. This circumstance substantially complicates the technical application of the indicated method for excitation of ferrites. Furthermore there is a difficulty in principle, in that τ is of the order of magnitude of, or less than, the time constant of the apparatus. Therefore we used a somewhat different principle, consisting of this: that the resonance excitation of the