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MEASUREMENTS OF THE TRANSVERSE PROTON RELAXATION TIME IN AQUEOUS SOLUTIONS OF PARAMAGNETIC SALTS BY THE SPIN-ECHO METHOD

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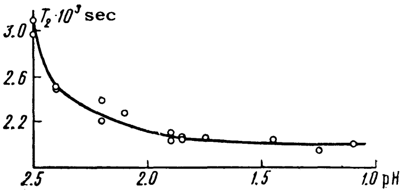
THE spin-echo effect[1,2] makes it possible to determine experimentally the absolute values of the longitudinal and transverse nuclear relaxation times T_1 and T_2. The advantage of the spin-echo method over other methods is particularly evident in measurements of nuclear relaxation times in low-viscosity liquids.[2]

To obtain the spin-echo effect, we constructed apparatus for the quantitative measurement of the transverse relaxation time T_2 of protons in water, in the presence of paramagnetic ions. The measurement was carried out at 12.2 Mcs in the field of a permanent magnet. A radio-frequency magnetic field with an amplitude of ~ 3.7 oersteds was applied to the sample in the form of two successive short rectangular pulses, with approximate durations of 16 and 32 microseconds, insuring 90° and 180° nutations respectively in the magnetic polarization of the water protons. The interval between the pulses could be varied from 0.3 to 2 milliseconds.

The solution to be studied, contained in a glass tube, was placed in a radio-frequency induction head of the "through-flow" type,[3] whose receiving coil picked up the induced nuclear echo signal, which was then amplified by a receiver with a 120 kcs bandwidth. The amplified output voltage from the receiver was applied to an oscillograph, and the echo signal on its screen was photographed. The non-uniformity of the permanent magnet field was estimated from the width of the echo signal. It proved to be equal to 1.2 oersted in the region where the sample (1 cm^3 in volume) was located.

In a field of the above non-uniformity, the effect of the self-diffusion of water molecules during the experimentally-determined time T_2 (about 10^{-3} sec) was negligible.

As a check on the operation of the apparatus, experiments were carried out on the hydrolysis of iron in nitric acid solution. In all the experiments the concentrations of Fe^{+++} and nitrate ions were constant and equal to 0.02 M and 0.6 M respectively. The results of the T_2 measurements for water protons in $Fe(NO_3)_3$ solutions are shown in the figure as a function of the acidity (pH). It is well known[4] that the proton relaxation effect in solutions of paramagnetic salts depends on the degree to which the ions are bound into a complex. The values of T_2 at higher pH are considerably larger than at high acidity. The gradual reduction in T_2 with increasing acidity indicates an increase in the effectiveness of the paramagnetic iron ions in shortening the transverse relaxation time of protons. This can be explained by the fact that when the pH is reduced the concentration of hydroxyl ions in the solution is decreased, leading to the dissociation of the iron hydro-complex. With increasing relative amounts of free iron ions in the solution, which are more effective in causing relaxation, the time T_2 must become shorter, as is well confirmed by the trend of the data shown in the figure.



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