DECREASING THE SIGNAL OF THE SOLVENT IN NMR SPECTRA

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The signals of the solvent not infrequently mask the signals of the substance under investigation, thereby decreasing the informativeness of an NMR spectrum. It is possible to decrease the signals of the solvent if use is made of the difference in the relaxation times T_1 of the solvent and of the substance. For this purpose, before recording the signals the magnetization M of the nuclei of the solvent must be changed to its opposite and the time be awaited when, in the process of relaxation, the projection of the magnetization vector M_z, which is proportional to the NMR signal, returns to zero. In pulsed spectrometers this is achieved by using a sequence of 180° and 90° pulses following after time τ [1].

We have succeeded in decreasing the signals of the solvent in a nonpulse HA-100D spectrometer by using adiabatically rapid passage (ABP) through resonance to invert the vector of M [2]. For a given high-frequency field, it is impossible simultaneously to satisfy the conditions for ABP and a weak field (for the undistorted recording of the signals). Consequently, we made use of a two-frequency method. Field H_1 was used in the usual way to observe the signals and field H_2 to invert the nuclear moments. The scanning of the spectrum was effected by using the magnetic field. The direction of scanning was selected in such a way that the nuclei first came into the resonance condition at frequency ν_2 (inversion) and then, after time τ, at frequency ν_1 (observation). The time τ is determined by the difference in frequencies ν_1 and ν_2 and the rate of passage of the spectrum V ($\tau = \Delta\nu_{1,2}/V$). For many solvents, the ABP condition is satisfied at the usual rates of recording spectra (2-4 Hz/sec).

Figure 1* shows the spectra of a solution in $CDCl_3$ with added ethanol of the biologically active substances visnadin (I) and dihydrosamidin (II) (1:1 mixture) [3, 4]. Atmospheric oxygen had been removed from the solution by passage of ultrapure nitrogen through the solution in the tube. The tube was closed with a stopper (in a set with the tube). The temperature of the sample was somewhat below room temperature.

The lowest spectrum was obtained under the ordinary conditions for recording spectra on a HA-100D spectrometer (attenuation of the high-frequency field 20 dB, rate of scanning of the spectrum 2 Hz/sec). The other spectra were obtained by the method of preliminary inversion. In recording these spectra, no changes whatever took place in the working conditions of the spectrometer, apart from the switching in of the additional sonic-frequency generator. The low-frequency voltage in the modulation coil was 10 mV. In the spectra given that were obtained by the method described above, the signals of the solvent have been reduced more than 10-fold. The method of preliminary inversion can also be used for studying relaxation in molecules if the time T_1 is greater than 1 sec.

LITERATURE CITED

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*Figure 1 is displayed on the following page.

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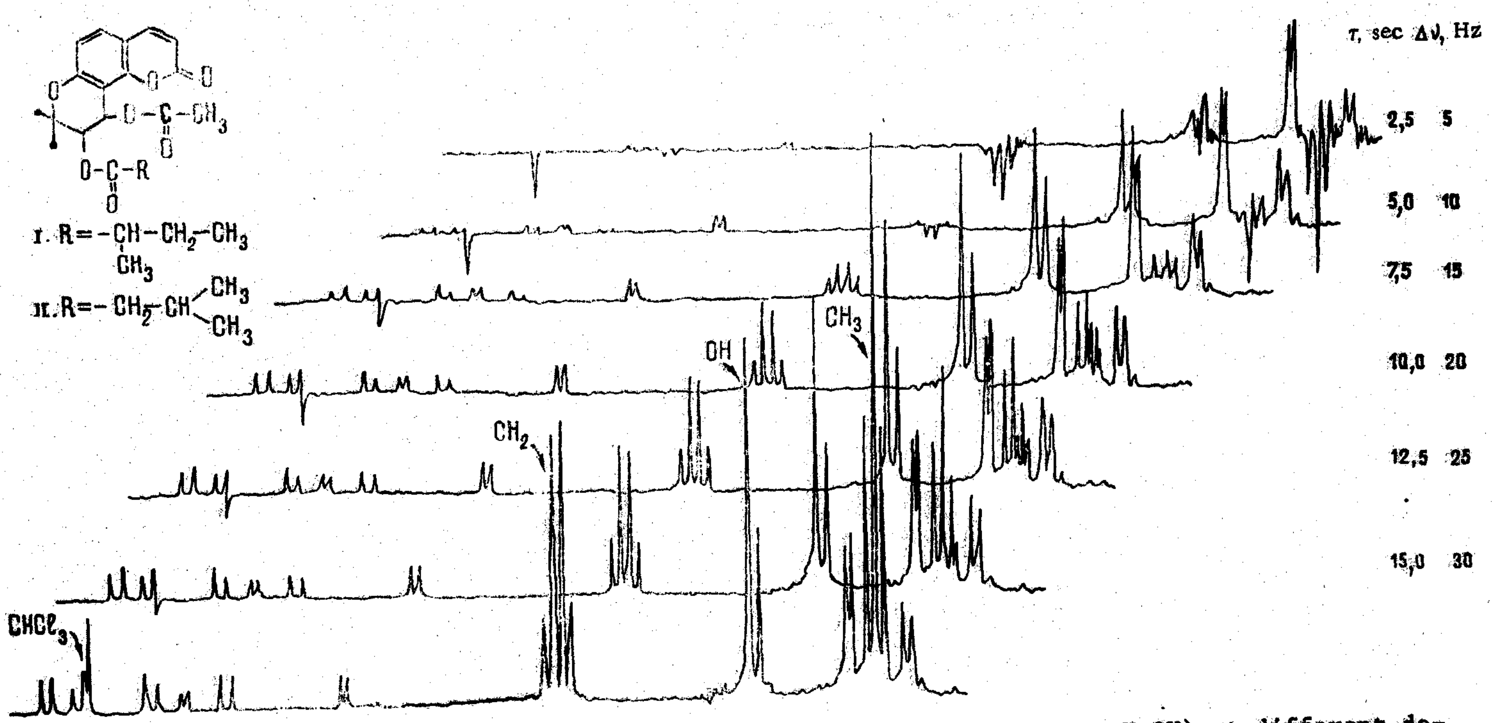

Fig. 1. NMR spectra of visnadin and dihydrosamidin (1:1 mixture, $CDCl_3 + C_2H_5OH$) at different degrees of detuning of the frequencies ν_1, ν_2. At the bottom, the usual spectrum, at the top the spectra of previous inversion. The values of τ and $\Delta\nu_{1,2}$ are given on the right.