THE COMPOSITION OF VISNADIN

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Visnadin was obtained in the form of acicular cyrstals with mp 85-87°C (from petroleum ether; Kofler block); its IR spectrum was identical with that given by Kuznetsova [1]. In an examination of its NMR spectrum, a form of the signal from the proton at C_4' which is unusual for the spectra of acylcoumarins of the khellactone group can be seen (Fig. 1). The smaller splitting of this signal cannot be due to the spin–spin coupling of H_4' with the other protons of the molecule, since the components of the quartet have very different intensities, in spite of the large chemical shifts between the signals. This form of the quartet has permitted the assumption that the substance contains another coumarin as an impurity to which a doublet of lower intensity corresponds. The similar chromatographic behaviors of visnadin and dihydrosamidin [2] and the fact that they are both present in the raw material [2, 3] indicated that this impurity could be dihydrosamidin.

In actual fact, the addition of dihydrosamidin to visnadin in a ratio of 1 : 1 led to an increase in the intensity of the signal from the C_4' proton of dihydrosamidin, and all the other signals of these substances in the aromatic region of the spectrum coincided. A calculation of the intensities of the two doublets from the C_4' proton in the spectrum of visnadin showed that it contained 20% of dihydrosamidin. The further purification of the visnadin by recrystallization did not give a pure product. The amount of dihydrosamidin present shows that the acicular crystals of visnadin [2-6] probably consist of four molecules of visnadin and one molecule of dihydrosamidin which, apparently, form a crystal lattice. The difficulty of obtaining visnadin in the pure state is probably connected with this phenomenon.

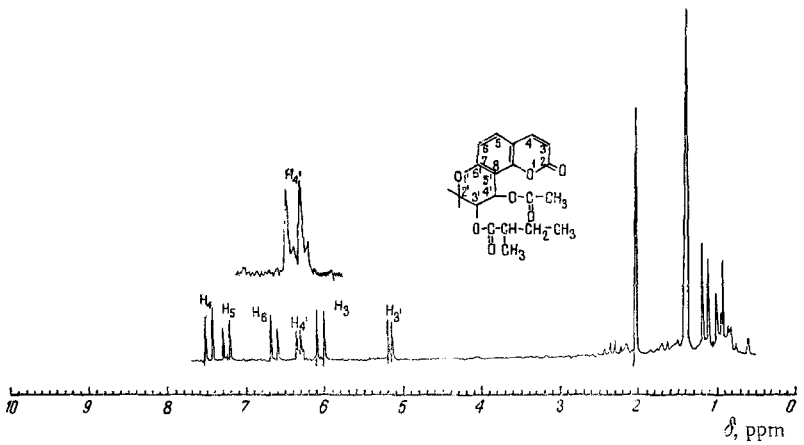

Fig. 1. NMR spectrum of visnadin (CCl_4, 100 MHz).

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