

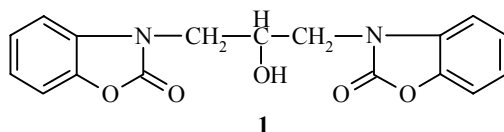
STRUCTURE OF 1,3-BIS-(3-BENZOXAZOLONYL)-2-HYDROXYPROPANE

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We have studied the ^1H NMR spectrum of the synthesized 1,3-bis(3-benzoxazolonyl)-2-hydroxypropane. The nature of the splitting of the signals in the spectrum and computer modeling indicate that free rotation is possible about all non-ring bonds in the compounds.

Keywords: 1,3-bis(3-benzoxazolonyl)-2-hydroxypropane, computer modelling.

We previously reported about synthesis of 1,3-bis(3-benzoxazolonyl)-2-hydroxypropane (**1**) by reaction of benzoxazolinone and its sodium or potassium salt with epichlorohydrin. The structure of compound **1** is consistent with IR and mass spectroscopy data [1].



In this paper, we present the results of a study of the structure of alcohol **1** by ^1H NMR spectroscopy. The ^1H NMR spectrum of compound **1** in CD_3OD is three groups of signals. The methine proton appears as a septet at 4.38 ppm with relative distribution of intensities for the components as 1:2:3:4:3:2:1. The two doublets of doublets at 3.96 ppm and 3.90 ppm belong to methylene protons and are characterized by a geminal constant of 14.69 Hz and two vicinal constants for coupling with the methine proton of 4.02 Hz and 8.06 Hz.

The well resolved spectrum of the ABCD type from the aromatic portion of the molecule is located in the 7.05-7.25 ppm region. High resolution of signals from aromatic protons is also seen for all their coupling constants for interaction with each other, with the characteristic values: *ortho*, 7.69 Hz; *meta*, 1.40 Hz; and *para*, 0.49 Hz. The probable assignment of the signals for the aromatic protons is 7-H, 7.21; 5-H, 7.15; 6-H, 7.08; and 4-H, 7.18.

In computer modelling of the compound, the barriers to free rotation for both the central bond $\text{CH}_2\text{-CH}_2$ and for the $\text{CH}_2\text{-N}$ bond were ~ 5 kcal/mole. In other words, at room temperature free rotation is possible about all four non-ring single C-C and C-N bonds of the molecule.

We dealt with the question of stabilization of some selected configuration as a result of possible intramolecular hydrogen bonding between the central hydroxyl group and one of the carbonyl groups by means of computer modeling in the PM3 semiempirical method. For an isolated molecule, the distance from the

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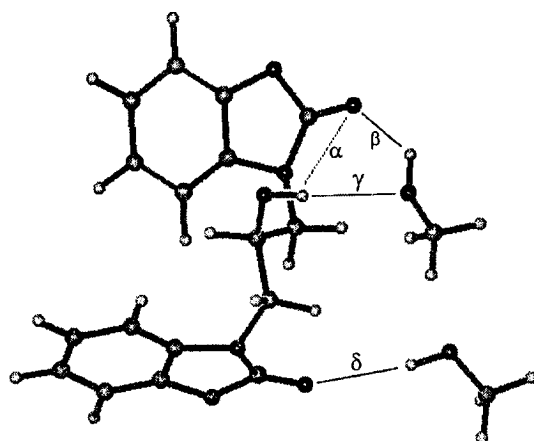


Fig. 1 General view of molecule 1.

hydroxyl proton to the carbonyl oxygen may be quite stabilized for values of 2.5-3.1 Å (depending on the configuration of the remaining portion of the molecule). The energy of the intramolecular hydrogen bond (the α -bond in Fig. 1) in this case is ~6.8 kcal/mole. However, modeling of compound **1** together with two methanol molecules, bonded to the main molecule by hydrogen bonds, showed a specific intermolecular interaction (the bonds β , γ , and δ). The energies of these hydrogen bonds were respectively 9.9, 11.3, and 10.6 kcal/moles. In this case, a certain cyclic structure appears that is stabilized by the two hydrogen bonds β and γ , competing with the intramolecular α bond but with total energy three times higher.

Thus possible preferred formation of an intramolecular hydrogen bond α and its effect on the stereochemistry of molecule **1** in CD₃OD solution has not been confirmed. The appearance of a quasicyclic structure with two hydrogen bonds with either one or the other carbonyl are alternately equally probable, and such an interaction insignificantly hinders rotation about the linear bonds of the molecule.

EXPERIMENTAL

The ¹H NMR spectrum were taken on a UNITYplus spectrometer with working frequency 400 MHz in CD₃OD (internal standard, HMDS).

REFERENCES

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