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22 June 1965

Topchiev Institute of Petrochemical Synthesis,
AS USSR, Moscow

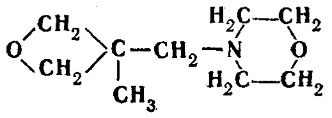
UDC 547.867.4 + 543.422

PMR SPECTRA OF SOME MORPHOLINE DERIVATIVES

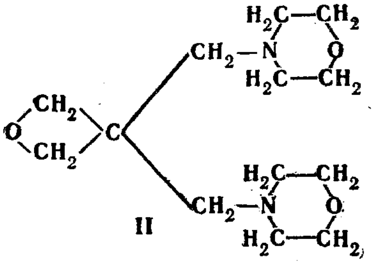
A. S. Zvontsova, S. Z. Kaplan, and N. M. Grad

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 2, No. 4, p. 631-632, 1966

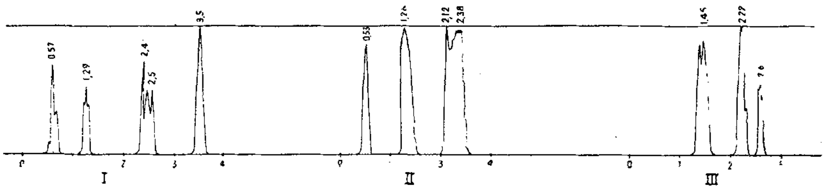
Figure 1 shows experimental PMR spectra* for two morpholine derivatives which we recently synthesized, 3, methyl-3-morpholinomethyloxacyclobutane, bp 244° C (I) [1], and 3, 3-bis(morpholinomethyl) oxacyclobutane, bp 72° C (II) [2].



I



II



PMR spectra of morpholine derivatives (for explanation see text).

For comparison the PMR spectrum of morpholine (III) itself, determined under the same conditions, is given. Compound II was dissolved in CCl_4. The measurements were made with a JNM-3 instrument, frequency 40 mcps, resolution 5×10^{-8}. With increase in the magnetic field, the ppm values increase, too. The proton resonance chemical shifts are measured relative to water, used as an external standard.

According to the literature, the proton spectrum of morpholine [3, 4] consists of two very intense signals from protons of the CH_2 groups, amounting to 2.3 and 1.1 ppm. The first of these signals belongs to the methylene group α to the nitrogen atom, and the second to the β methylene group. Our proton resonance chemical shift values for the α and β methylene groups of the morpholine ring are quite close to those for all three compounds considered (table).

According to [4], it is correct that the PMR spectrum of morpholine (III) should also contain a signal from the NH group, value 4.3 ppm. However, our morpholine PMR spectrum had a third signal due to a proton, and equal to only 2.6 ppm.

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*For which we thank V. B. Lebedev

Values of Chemical Shifts of Signals Due to Protons, Derived from PMR Spectra of Morpholine and Two of its Derivatives (I and II)

Name of proton-containing group	Value of the chemical shift, ppm		
	I	II	III
CH_2 α to N	2.4	2.12	2.22
CH_2 β to N	1.29	1.26	1.45
NH	—	—	2.6
Oxocyclobutane CH_2	0.57	0.53	—
CH_3	3.5	—	—
CH_2 connected to oxacyclobutane and the N atom of morpholine	2.5	2.38	—

It would be interesting to explain the cause of the multiplicity of the signal from the oxacyclobutane CH_2 groups in compound I. Possibly it is due to the dissymmetry of the groups joined to them. However, only further research will answer this question.

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14 August 1965

Higher Naval Engineering School, Leningrad

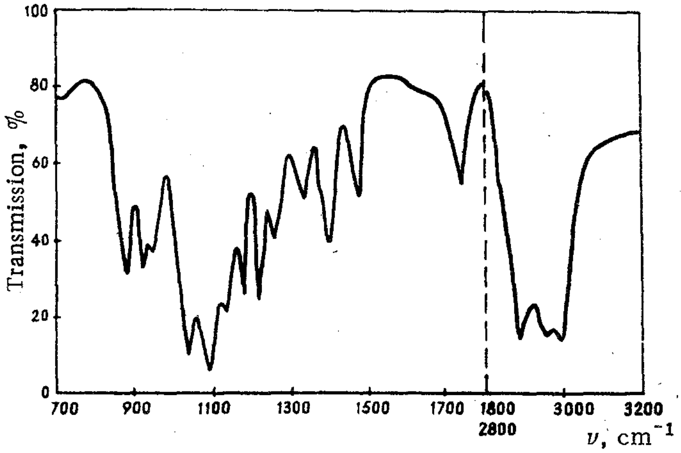
UDC 547.712 + 547.722 + 542.951.4

BISTETRAHYDROFURFURYL ETHER

L. M. Prutkov, I. K. Sanin, and I. V. Kamenskii

Khimiya Geterotsiklicheskikh Soedinenii, Vol. 2, No. 4, p. 632-633, 1966

Bistetrahydrofurfuryl ether I, hitherto not described in the literature, has been isolated in the course of a study of the thermal condensation of tetrahydrofurfurylhydroxysilanes.



IR spectrum of bistetrahydrofurfuryl ether.