MOLECULAR AND CRYSTAL STRUCTURE OF 2-METHOXY-3,4-DIMETHYL-5-PHENYL-1,3,2-OXAZAPHOSPHOLIDIN-2-ONE

K. N. Turdybekov, S. V. Lindeman, Yu. T. Struchkov, A. M. Gazaliev, O. A. Nurkenov, and M. Zh. Zhurinov

UDC 548.737

In the present paper we consider the results of an x-ray structural investigation of 2-methoxy-3,4-dimethyl-5-phenyl-1,3,2-oxazaphospholidin-2-one (I). The structure of the molecule of (I) with indications of bond lengths and torsion angles in the five-membered ring is shown in Fig. 1.* The conformation of the molecule is characterized by a departure of the pharmacophoric groups – the O1 and N1 atoms – in different directions from the plane of the benzene ring by –0.94 and 1.02 Å, respectively (in the molecule of pseudoephedrine (II) [1] the corresponding deviations are –0.95 and 1.17 Å). The torsion angle τ (C6C1C7O1) = 48.2(3)° coincides with that found in (II). The torsion angle ω (O1C7C8N1) = 34.8(2)° is appreciably less than in (II) and in pseudoephedrine hydrochloride (III) [1] (52 and 55°, respectively) because of the closure of the five-membered phosphorus-containing ring. The methyl group at the nitrogen atom in the molecule of (I) has an orientation [χ(C7C8N1C10) = –179.7°], close to that of this group in (II) and (III) (–162 and –171°, respectively).

The conformation of the five-membered ring is a slightly distorted envelope, the N1P1O1C7 atoms being coplanar to within +0.03 Å (the N1P1O1C7 torsion angle is 4.8°), and the C8 atom departing from their plane by 0.5 Å. The methyl group C8 is present in the equatorial orientation.

The conformation of the phosphorus atom is distorted tetrahedral with valence angles of 96.8-120.7°, which is characteristic also for other cyclic derivatives of tetracoordinated phosphorus [2].

The parameters and the intensities of 1525 reflections were measured on a Hilger–Watts diffractometer (λMo Kα, graphite monochromator, $\theta/2\theta$ scanning, $2\theta \leq 60°$). The crystals of

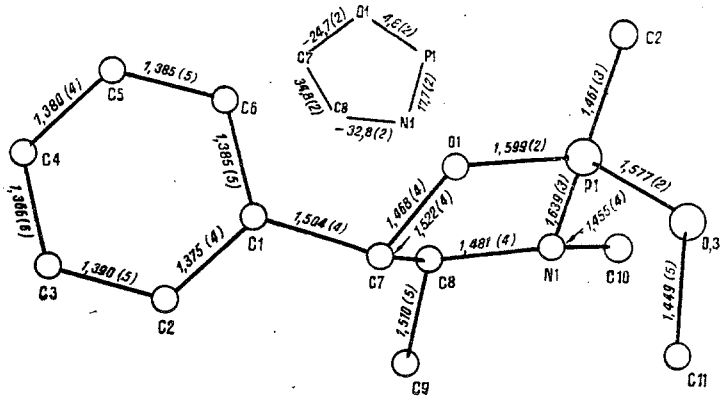

Fig. 1. Structure of the molecule of (I). The bond lengths and torsion angles in the 5-membered ring are given.

*The numbering of the atoms in (I) is based on the numbering of the atoms in the pseudoephedrine molecule.

Institute of Organic Synthesis and Coal Chemistry, Kazakh SSR Academy of Sciences, Karaganda. A. N. Nesmeyanov Institute of Organometallic Compounds, Academy of Sciences of the USSR, Moscow. Translated from Khimiya Prirodnykh Soedinenii, No. 2, pp. 291-293, March-April, 1989. Original article submitted May 26, 1988; revision submitted September 29, 1988.

 0009-3130/89/2502-0256$12.50

TABLE 1. Coordinates of the Atoms (×10⁴; for H, ×10³)

Atom	x	y	z	Atom	x	y	z
P1	7962 (1)	9996	6051 (1)	H2	916 (3)	766 (3)	1106 (2)
O1	8333 (2)	10460 (3)	7672 (2)	H3	835 (3)	848 (4)	1284 (2)
O2	6926 (3)	11379 (4)	5025 (2)	H4	645 (3)	1055 (4)	1248 (2)
O3	9549 (2)	10020 (4)	5899 (2)	H5	499 (4)	1206 (4)	1006 (4)
N1	7422 (3)	7754 (2)	6057 (2)	H6	589 (4)	1090 (4)	833 (3)
C1	7654 (3)	9336 (4)	9554 (3)	H7	923 (3)	817 (3)	887 (2)
C2	8390 (3)	8566 (5)	10895 (3)	H8	595 (3)	792 (2)	712 (2)
C3	7902 (4)	9032 (6)	11945 (3)	H9.1	714 (3)	532 (5)	867 (3)
C4	6685 (4)	10271 (3)	11656 (3)	H9.2	654 (4)	447 (6)	696 (3)
C5	5942 (4)	11070 (5)	10318 (3)	H9.3	834 (4)	482 (6)	798 (3)
C6	6424 (4)	10594 (5)	9270 (3)	H10.1	683 (3)	551 (4)	491 (3)
C7	8170 (3)	8744 (5)	8425 (3)	H10.2	554 (4)	706 (5)	448 (3)
C8	7049 (3)	7413 (4)	7288 (3)	H10.3	682 (3)	732 (5)	398 (3)
C9	7256 (4)	5329 (5)	7747 (3)	H11.1	1138 (4)	937 (6)	778 (3)
C10	6567 (4)	6697 (6)	4745 (3)	H11.2	1138 (4)	859 (7)	621 (3)
C11	10813 (4)	8736 (7)	6729 (4)	H11.3	1052 (5)	741 (6)	671 (4)

(I) are monoclinic, a = 9.4738(7), b = 6.9421(3), c = 10.3740(8) Å, β = 114.928(6)°, d_{calc} = 1.295 g/cm³, z = 2 ($C_{11}H_{16}O_3NP$), sp. gr. $P2_1$. The calculations were performed with 1467 independent reflections having $I \geq 2\sigma$. The structure was interpreted by the heavy-atom method, the system of coordinates being selected on the basis of the known configuration of pseudoephedrine [1]. Refinement was made by the block-diagonal MLS in the anisotropic approximation for the nonhydrogen atoms. The H atoms of the methyl and methoxy groups were revealed in a difference synthesis. The positions of the other hydrogen atoms were calculated. All the H atoms were included in a refinement in the isotropic approximation. The final divergence factors were R = 0.037 and R_w = 0.031. All the calculations were performed on an Eclipse S/200 computer by means of the INEXT programs [3]. The coordinates of the atoms are given in Table 1.

LITERATURE CITED

1. M. Mathew and G. J. Palenik, Acta. Cryst., B33, 1016 (1977).
2. L. S. Khaikin and L. V. Vilkov, Usp. Khim., 41, No. 12, 2224 (1972).
3. R. G. Gerr, A. I. Yanovskii, and Yu. T. Struchkov, Kristallografiya, 28, 1029 (1983).