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LETTERS TO THE EDITOR

Spirophosphorane in the Reaction of Hexamethyltriamidophosphite with Bis(salicylal)-1,2-diaminopropane

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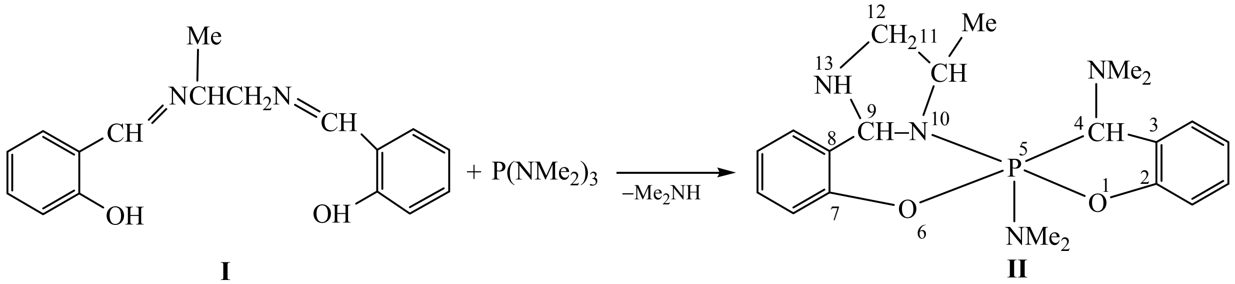
In order to synthesize a new type of the tetradentate ligands we previously performed the phosphorylation of bis(salicylal)diaminoethane with alkylenechlorophosphites. Unexpectedly, the reaction products were found to be the framwork structures containing the hexacoordinated phosphorus atom [1–3]. In continuation of these studies we examined the reaction of bis(salicylal)-1,2-diaminopropane **I** with hexamethyltriamidophosphite. The cascade reaction involves the intramolecular transformations that result in the spirocyclic compound **II**. Since the molecule of this compound contains several chiral centers, and according to the ^{1}H and ^{31}P NMR spectra it is formed as a diastereomers mixture in the ratio of 50:50%, we can conclude that the process is stereoselective. One of the diastereomers was isolated in a pure form and its structure was established by the X-ray diffraction analysis.



I **II**

Compound **II** crystallizes in a space group *P*21/*c*. The unit cell contains two independent molecules, which differ slightly from each other. The conformation of the five-membered phosphorus ring is a *P-envelope*, of the six-membered ring, *sofa*. The fragment $O^6C^7C^8C^9$ is planar. The N^{10} and P^5 atoms deviate from the fragment on one side, but by different distances. The conformation of the nitrogen-containing $N^{10}C^{11}C^{12}N^{13}C^9$ fragment attached to the six-membered heterocycle is a *semichair*. The crystal packing of the molecules is stabilized by the classical hydrogen bonds of the N–H···N type, which lead to the formation of the molecular chains along the crystallographic 0*b* axis.

4,5-Bis(dimethylamino)-11-methyldibenzo[*b*,*g*]-imidazolidino[2,1-*i*]-10,13-diaza-5-phosphaspiro-[5,6]deca-2,7-diene (II). A mixture of 1.4 g of diimine **I** and 0.8 g of triamidophosphite in 15 ml of acetonitrile was refluxed for 1 h. Then solvent was evaporated, and the residue was dissolved in a methylene chloride–hexane mixture. The precipitate was recrystallized from benzene. Yield 0.96 g (48%), mp 119°C. IR spectrum, ν, cm^{-1}: 3239 (NH). ^{1}H NMR spectrum (CDCl$_3$), δ, ppm (*J*, Hz): 1.27 d (3H, *J* 6.3), 1.35 d (3H, *J* 6.2), 2.44 d (6H, *J* 2.5), 2.46 d (6H, *J* 2.8), 2.50 d (6H, *J* 10.3), 2.56 d (6H, *J* 10.0), 3.01–3.05 m (1H), 3.17–3.22 m (1H), 3.38–3.45 m (1H), 3.49–3.56 m (1H), 3.84–3.89 m (1H), 3.91–3.95 m (1H), 4.26 d (1H, *J* 5.41), 4.28 m (1H, *J* 7.06), 5.37 s (1H), 5.48 s (1H), 6.75–7.40 m (16H). ^{31}P NMR spectrum, δ_P, ppm: 40.30, 41.29. Found, %: C 62.75; H 7.62; N 14.50; P 7.77. $C_{20}H_{28}N_3O_2P$. Calculated, %: C 63.00; H 7.25; N 14.00; P 7.75.

The IR spectra were recorded on a Bruker Vector-22 spectrometer in the range of 400–3600 cm^{-1} for the dispersion of samples in mineral oil. The ^{1}H NMR spectra were taken on an Avance 600 spectrometer (600.13 MHz), the residual proton signals of $CDCl_3$ serving as internal reference. The ^{31}P NMR spectra were registered on a Bruker MSL-400 NMR Fourier spectrometer (100.62 MHz).

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