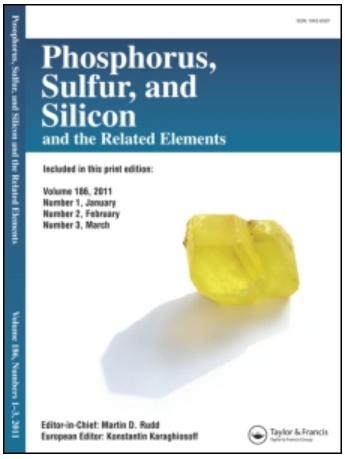

Phosphorus, Sulfur, and Silicon and the Related Elements

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The Synthesis and X-Ray Structural Studies of Bis(1,3,2-Dithiaphospholane)

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The Synthesis and X-Ray Structural Studies of Bis(1,3,2-Dithiaphospholane)

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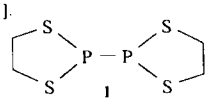
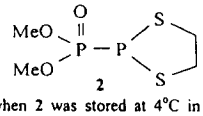
The title compound **1** [^{31}P NMR (CD_3CN): δ=36.7ppm] has been identified among the products of controlled hydrolysis of 2-dimethoxyphosphoryl-1,3,2-dithiaphospholane (**2**) [1].

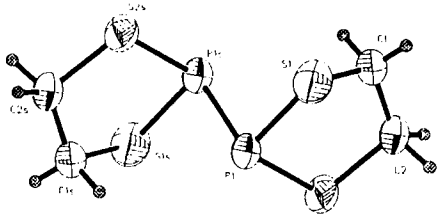


1



2

The crystalls of hypophosphite **1** were obtained when **2** was stored at 4°C in a wet acetonitrile solution. Compound **1** was found to crystallize in monoclinic system in a space group $P2_1/n$. The molecular geometry and conformation of crystalline form of **1** was studied by X-ray method on an Enraf-Nonius CAD4 diffractometer with graphite monochromatized CuKα radiation. The unit cell of **1** consists of two hypophosphite molecules. There is one dithiaphospholane ring in an asymmetric part of the unit cell; the second ring is symmetrical (symmetry: *-x, 1-y, -z*). The analysis of torsion angles and asymmetry parameters of a dithiaphospholane ring reveals that the heterocyclic ring adopts a deformed envelope conformation, with the C1 atom in the flap position. The dihedral angle 51.0(2)° was found between the least-squares plane passing through „basic" atoms of the envelope (S1, P1, S2, C2) and another one drawn through S1, C1, C2 atoms. The analysis of hydrogen contacts did not show any inter- or intramolecular contacts with distances shorter than 2.80 Å [2].



References

[1] L.V. Ermanson, N.N. Godovikov, V.S. Blagoveshchensky, Izv.Akad.Nauk SSSR. Ser.Khim. **1986,** 423–427.

[2] The details are available on request from Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge, CB2 1EZ, UK.