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Preparation and Structural Characterization of 1-Phenyl-1-(Dicyclohexylamino)Cyclo-1-Phospha-3, 5-Dithia-2, 4, 6-Triazene; a Phosphathiazene With Different Substituents on Phosphorus

T. Mohan^a; C. J. Thomas^a; M. N. Sudheendra Rao^a; G. Aravamudan^a; A. Meetsma^b; J. C. van de Grampel^b

^a Department of Chemistry, IIT, Madras, India ^b Laboratory of Polymer Chemistry, University of Groningen, Groningen, The Netherlands

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PREPARATION AND STRUCTURAL CHARACTERIZATION OF 1-PHENYL-1-(DICYCLOHEXYLAMINO)CYCLO-1-PHOSPHA-3,5-DITHIA-2,4,6-TRIAZENE;
A PHOSPHATHIAZENE WITH DIFFERENT SUBSTITUENTS ON PHOSPHORUS

T. MOHAN, C.J. THOMAS, M.N. SUDHEENDRA RAO and G. ARAVAMUDAN
Department of Chemistry, IIT, Madras-600 036, India

A. MEETSMA and J.C. VAN DE GRAMPPEL
Laboratory of Polymer Chemistry, University of Groningen,
Nijenborgh 16, 9747 AG Groningen, The Netherlands

Abstract. The reaction of S_4N_4 with dicyclohexylaminophenyl-(2-aminopyridyl)phosphine in MeCN at room temperature leads to the formation of the novel compound $Ph(cycloHex_2N)PS_2N_3$. An X-Ray structure determination confirms that this compound consists of a six-membered PSN-ring with two different substituents at phosphorus.

The first representative of the class of 8- π electron six-membered $R_2PS_2N_3$ heterocycles ($R = Me_3SiNH$) was prepared in 1976 by Appel and *al.*¹ Since then, other examples were reported, viz. $R = Me$ ², Ph ²⁻⁴, OPh ², F ⁵, CF_3 ⁶ or C_2F_5 .⁶ Interestingly, all these compounds possess intense colours ranging from deep blue to dark purple. The compounds with R is phenyl^{2,3} or trimethylsilylamino⁷ are the only two species of which detailed structures have been established by X-ray studies.

Examples of such a ring skeleton possessing different substituents on phosphorus are not known so far. By using a new

synthetic route, we have prepared $\text{Ph}(\text{Hex}_2\text{N})\text{PS}_2\text{N}_3$ (**1**) as the first example of a cyclophosphadithiatriazene system having different substituents on phosphorus.

(Dicyclohexylamino)(phenyl)(2-pyridylamino)phosphine (DCA)⁸ and S_4N_4 were allowed to react in MeCN for 30h at room temperature.⁹ Hexane extracts of the reaction mixture yielded blue-violet needle-shaped crystals of **1** in 20% yield.

The molecular structure of **1** (Fig. 1) was elucidated by single crystal X-ray diffraction.⁹

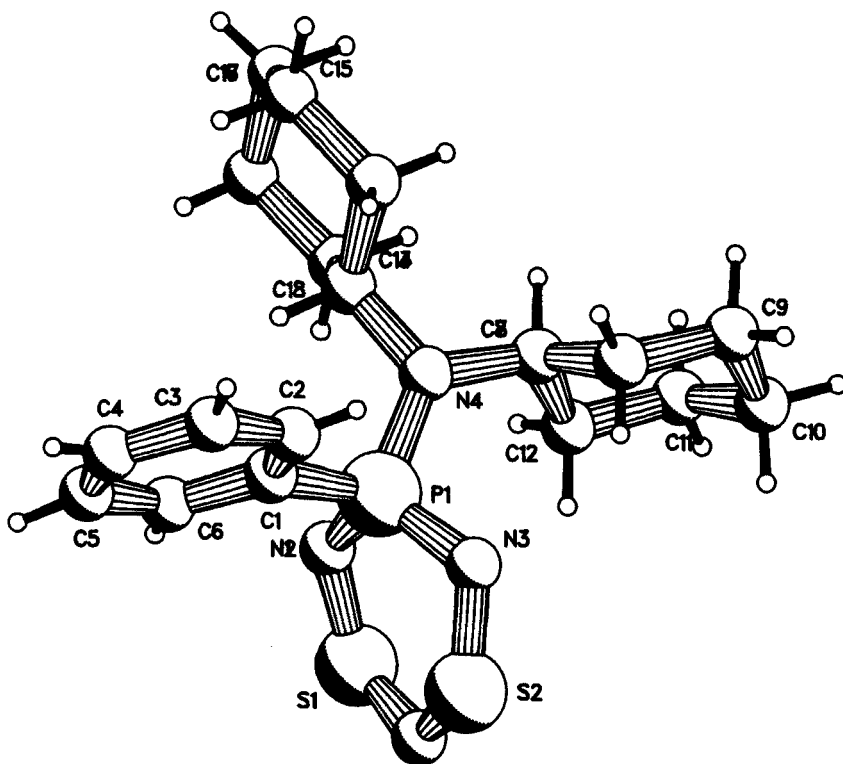


Fig. 1. Pluto drawing of the molecule of **1**.

The structural data of 1 can be compared with those of $(\text{Me}_3\text{SiNH})_2\text{PS}_2\text{N}_3$ (2) ⁷ and $\text{Ph}_2\text{PS}_2\text{N}_3$ (3). ³ This comparison reveals that the detailed conformation of the PSN ring differs in each case. Compounds 1 and 2 have more puckered ring skeletons whereas in 3 the ring is nearly planar. The S-N bond lengths in 1 vary from 1.574(4) to 1.606(4) Å; the mean value of the endocyclic P-N bonds equals 1.618(3) Å. Like in 2, the exocyclic P-N bond is shorter than a single bond, presumably as a result of $d_\pi(\text{P})\text{-}p_\pi(\text{N})$ bond formation.

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8. DCA was obtained by employing the procedure reported in T. Mohan, M.N.S. Rao and G. Aravamudan, Tetrahedron Letters, **30**, 4871 (1989).
9. Details will be published elsewhere.