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Reactions of Bifunctional Sulfur Compounds with Dihydrazido Phosphoric Acid Derivatives—Five- and Six-Membered Rings with Four N-Atoms

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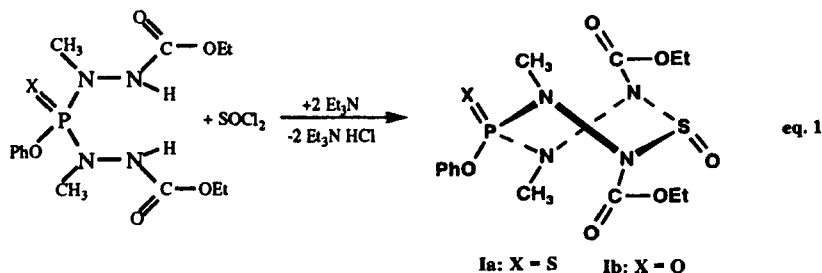
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REACTIONS OF BIFUNCTIONAL SULFUR COMPOUNDS WITH DIHYDRAZIDO PHOSPHORIC ACID DERIVATIVES -- FIVE- AND SIX- MEMBERED RINGS WITH FOUR N-ATOMS

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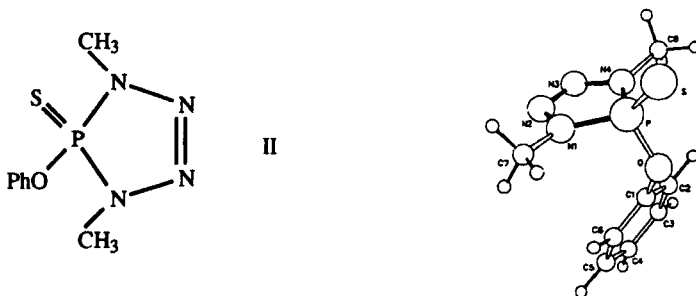
Abstract Thionylchloride and sulfurdichloride react with N-substituted phosphoric acid or thiophosphoric acid dihydrazides in the presence of triethylamine to yield six-membered rings containing sulfur if the substituents at N adjacent to the sulfur are electron-withdrawing groups such as $-C(=O)Et$. These groups lacking redox reactions are observed, resulting in the formation of a five-membered ring with an NN double bond that contains no sulfur. The X-ray structure of this " λ^5 -phosphatetrazolin" has been investigated: the ring is planar, PN 1.665(6), 1.680(6), N-N 1.372(9), 1.385(10), N=N 1.275(10)Å.

As reported earlier by other authors and by our group, stable sulfur hydrazine heterocycles can be obtained by substituting the hydrazine N-atoms with electron withdrawing groups such as $-C(=O)-OR$ or $-C_2F_5$ ¹⁻⁵. X-Ray structure and NMR investigations in our laboratory reveal that the corresponding six-membered rings I containing phosphorus(V) (eq.1) have twist-boat conformations in the solid and in solution due to the steric demand of the substituents at N⁵⁻⁷.



Without these electron withdrawing groups, the corresponding phosphoric acid dihydrazido derivatives react with thionylchloride, sulfurylchloride, or sulfur dichloride to form elemental sulfur and oxidation products, one of which could be isolated and identified by X-ray structure analysis to be the λ^5 -phosphatetrazolin derivative **II**.

Molecular structure of **II**:



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