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## Phosphorus, Sulfur, and Silicon and the Related Elements

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### A Cyclotriphosphazane-Trisulfide from the Sulfuration of a 1,2,4,3-Triazaphosphole

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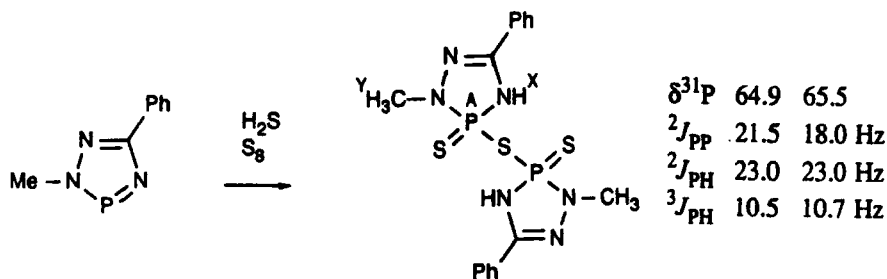
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## A Cyclotriphosphazane-trisulfide from the Sulfuration of a 1,2,4,3-Triazaphosphole

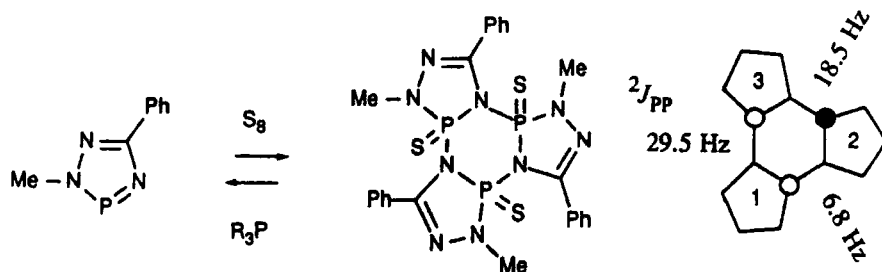
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The two-coordinate phosphorus of azaphospholes is typically inert to chalcogen oxidation. In general it reacts with sulfur only if a protic reagent as a third component is added simultaneously<sup>1</sup>. Hence the 2-methyl-5-phenyl-1,2,4,3-triazaphosphole adds sulfur in the presence of HCl or H<sub>2</sub>S, resulting in an azaphospholine sesquisulfide in the latter case<sup>2</sup>. The compound is obtained as a 55:45 mixture of diastereomers. Their structure is illustrated by their <sup>31</sup>P- and <sup>1</sup>H-NMR spectra of AA'XX'Y<sub>3</sub>Y'<sub>3</sub> type.



While azaphospholes are said not to react with sulfur alone, we find that in hot pyridine a slow direct sulfuration of the triazaphosphole can be achieved. It leads to a trimeric product with a central cyclotriphosphazane-trisulfide ring<sup>2</sup>.

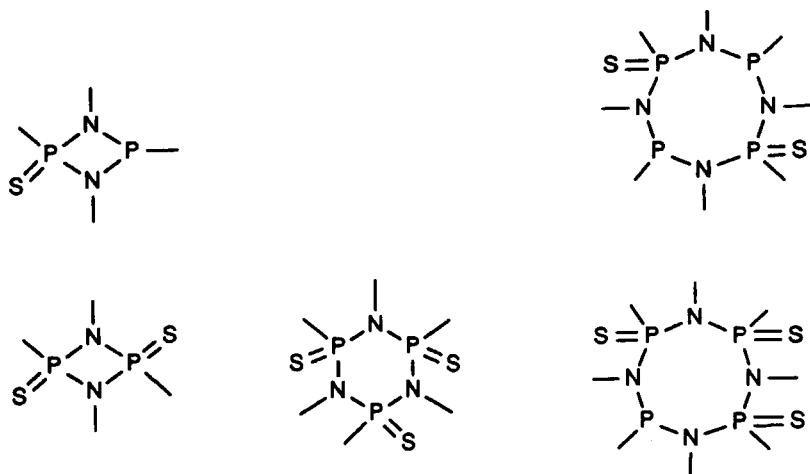


Its *cis*-substituted form would just give a single <sup>31</sup>P-NMR signal. The exclusively

observed ABC spin system therefore indicates the *trans*-substitution. A model suggests a boat-like conformation of the central six-membered ring with rather different S-P-N-P dihedral angles paralleled by the large differences in  $^2J_{PP}$  coupling.

On desulfuration by tributylphosphine the monomeric triazaphosphole reforms. This is reminiscent of the reversible tetramerization of a triazaphosphole by complex formation and decomplexation <sup>3</sup>.

While sulfides of cyclodiphosphazanes are common <sup>4</sup>, those of cyclotriphosphazanes <sup>5</sup> (and also of cyclotetraphosphazanes <sup>6</sup>) are rare.



In case of the azaphosphole derived cyclophosphazane sulfide the ring size is governed by the need of coplanar pairs of exocyclic bonds at adjacent P and N ring members.

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