

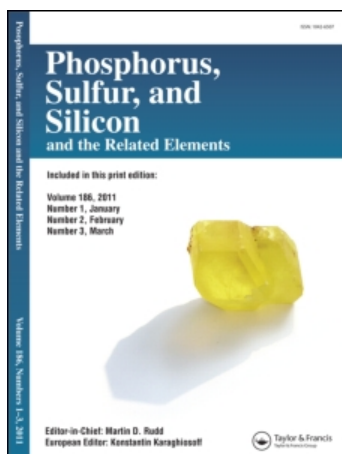
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Spirocyclic (0Xa)Azaphosphasilanes

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SPIROCYCLIC (OXA)AZAPHOSPHASILANES

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Abstract 12,14-Diamino-13-phenoxy-13-thioxo-6-oxa-12,14-diaza-13 λ^5 -phospha-5,7-disiladispiro[4.1.4.3]-tetradecane **1 b** and 3-Phenoxy-3-thioxo-1,2,4,5-tetraaza-3 λ^5 -phospha-6-silaspiro[5.5]undecane **2 a** are prepared from thiophosphoryldihydrazide and cyclic dichlorosilanes or the corresponding dichlorodisiloxanes. Constitutions of the new spirocyclic systems are confirmed by MS, NMR, elemental analysis and for **1 b** by x-ray structure analysis (monoclinic, $P2_1/c$, $a = 16.091(3)$, $b = 6.024(1)$, $c = 20.969(3)$ Å, $\beta = 104.46(2)^\circ$, $Z = 4$). The inorganic ring in **1 b** is essentially planar.

In the reaction of phenoxythiophosphoryldihydrazides with diorganodichlorosilanes or dichlorotetraorganodisiloxanes preferably sixmembered inorganic heterocycles are formed ^{1,2}. Using the corresponding cyclic compounds such as dichlorosilacyclopentane or the corresponding dichlorodisiloxanes, where the silicon is a member of an organic silacyclohexane, spirocyclic compounds of type **1** and **2** can be prepared according to equ.1. The constitutions of the new compounds are confirmed by MS (mol-peaks), ¹H-, ¹³C- and ³¹P-NMR spectra. The ¹H-NMR spectrum of **1 b** for instance reveals four distinct groups of signals: a multiplet for the phenyl protons at 7 - 7.4 ppm, a doublet for the exocyclic NH₂-protons at 3.6 ppm, ³J(PNNH) = 2 Hz, and two complicated multiplets at 0.3 - 0.9 and 1.5 - 1.8 ppm (intensity ratio 1:1) for methylene protons α or β to silicon in the silacyclopentane rings. The non-planar

inorganic rings in **2 a - d** interconvert rapidly at ambient temperature (Compounds **2 b-d** have N-methyl groups in α -positions to phosphorus and four- and sixmembered organic rings respectively).

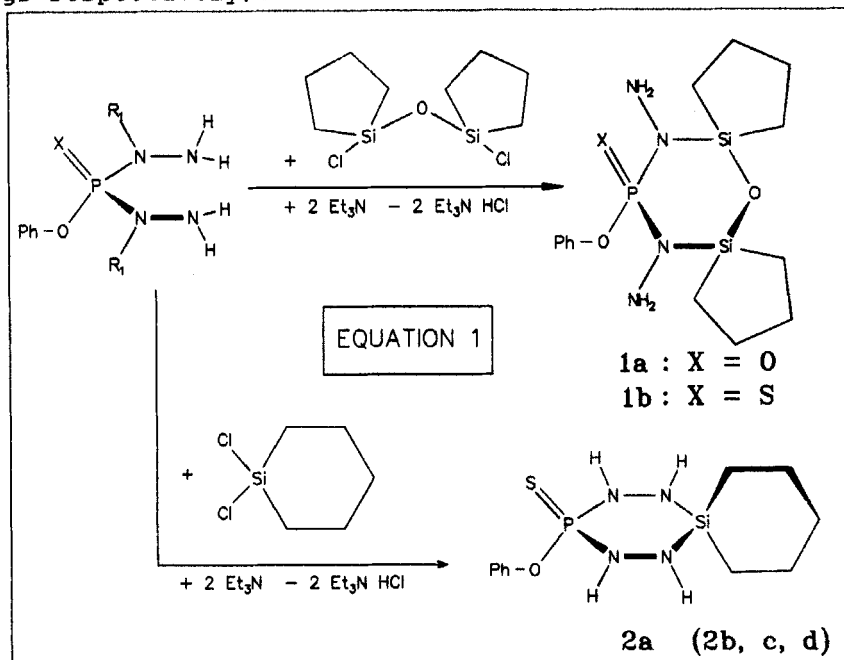
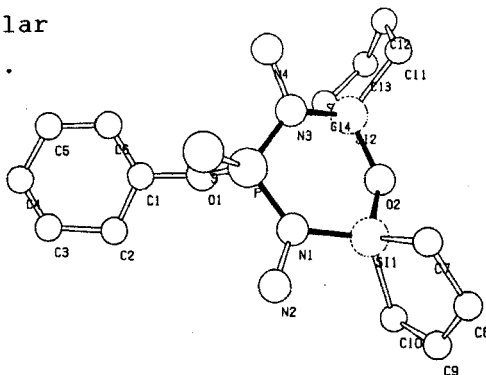


FIGURE 1 Molecular Structure of **1 b** .



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