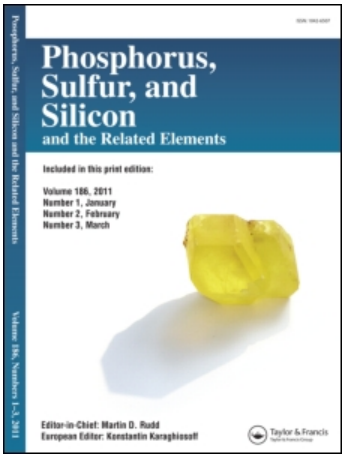

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Spirocyclic (0Xa)Azaphosphasilanes

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SPIROCYCLIC (OXA)AZAPHOSPHASILANES

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Abstract 12,14-Diamino-13-phenoxy-13-thioxo-6-oxa-12,14-diaza-13λ⁵-phospha-5,7-disiladispiro[4.1.4.3]-tetradecane **1 b** and 3-Phenoxy-3-thioxo-1,2,4,5-tetra aza-3λ⁵-phospha-6-silaspiro[5.5]undecane **2 a** are pre pared from thiophosphoryldihydrazide and cyclic dichlorosilanes or the corresponding dichlorodi-siloxanes. 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, β = 104.46(2)°, 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 dichlorodi-siloxanes, 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), ^{1}H-, ^{13}C- and ^{31}P-NMR spectra. The ^{1}H-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 NH_2-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).

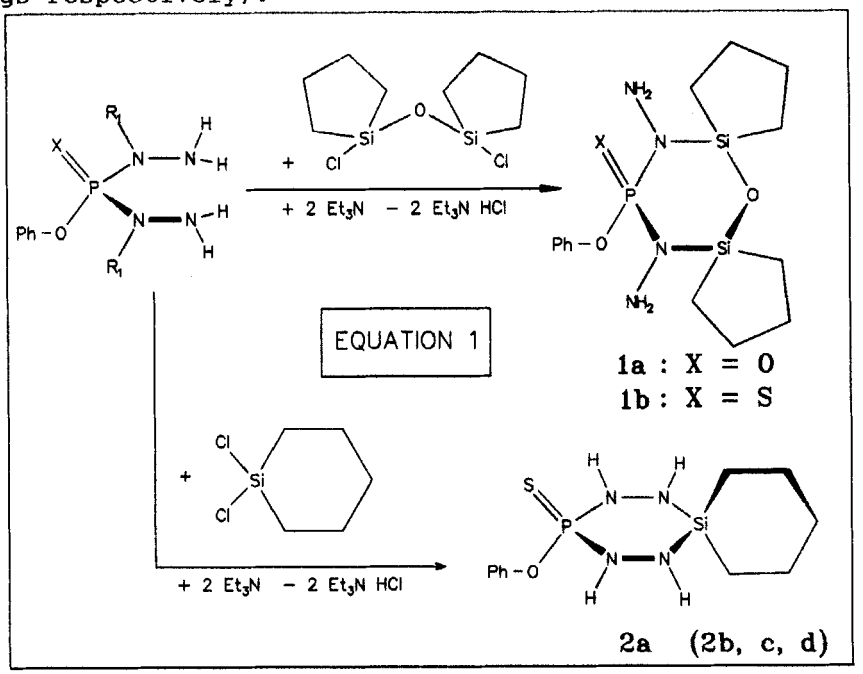
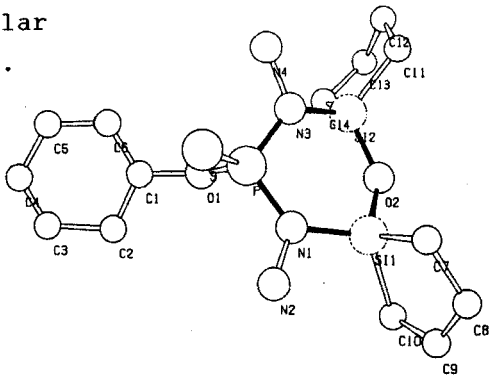


FIGURE 1 Molecular Structure of 1 b .



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