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From a Heterocycle Through a Hetero(spiro)cycle to a “Genuine Heterocycle”

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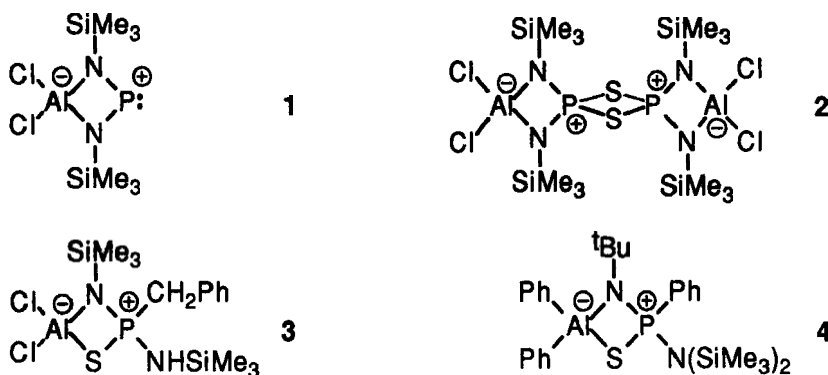
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FROM A HETEROCYCLE THROUGH A HETERO(SPIRO)CYCLE TO A "GENUINE HETEROCYCLE"

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Abstract A "Genuine Heterocycle" containing a PNAIS framework is obtained from a heterocyclic phosphonium species with a novel heterobis(spiro)tricyclic as an isolable intermediate.

Reaction of the zwitterionic heterocyclic phosphonium derivative **1**¹ with elemental sulfur in toluene yields the novel dimeric bis(spiro)tricyclic compound **2**.² If the mixture is allowed to stir at room temperature for two weeks, a complex reaction involving toluene gives the unexpected heterocycle **3**. Interestingly, toluene solutions of compound **2** in pure form are indefinitely stable. Spectroscopic³ and X-ray crystallographic investigations⁴ confirm **3** (Figure 1) as a new example of a "Genuine Heterocycle" (a ring system containing only one atom of each element in the heterocyclic framework).⁵ The PNAIS framework of **3** has been previously observed in **4** and the structural parameters are comparable.⁶ Although the mechanism of the reaction has not been assessed, the formation of **3** most likely involves the insertion of a P-N bond into the methyl C-H bond of a toluene molecule, cleavage of the Al-N bond and formation of an Al-S heterocyclic bond.



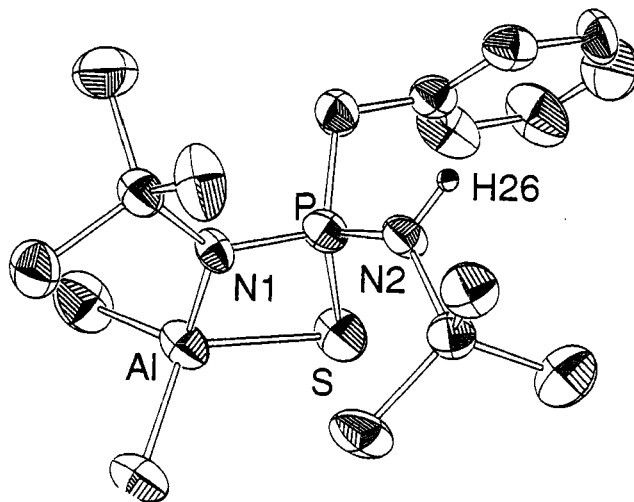


FIGURE 1 ORTEP view of **3**. Selected parameters: P-N1, 1.636(8)Å; N1-Al, 1.855(8)Å; Al-S, 2.272(5)Å; P-S, 2.045(4)Å; N1-P-S, 99.9(3)°; Al-N1-P, 98.4(4)°; N1-Al-S, 85.9(3)°; Al-S-P, 75.4(2)°.

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3. Spectroscopic data for **3**: Isolated yield 37%; NMR (CD₂Cl₂) ³¹P{¹H}: 55 ppm; ¹³C{¹H}: 130, 129.9, 129.5, 128.5 (d, J = 3.8Hz), 46.9, 2.2, 1.2 ppm; ¹H: 7.36 (m, 5H), 3.51 (m, 2H), 2.48 (s, 1H), 0.31 (s, 6H), 0.24 ppm (s, 6H); IR (Nujol, CsI, cm⁻¹): 3313m, 2722w, 1953w, 1602w, 1587w, 1493m, 1411w, 1258s, 1187w, 1130w, 1070sh, 1050s, 979s, 912s, 880sh, 846s, 829sh, 809s, 777s, 758s, 699s, 673m, 647m, 612w, 595m, 551s, 531s, 508s, 487s, 439w, 417m.
4. X-ray data for **3**: C₁₃H₂₆AlCl₂N₂PSSi₂ MW = 427.45, colourless plates, monoclinic, P2₁/n, Z = 4, a = 12.694(2)Å, b = 12.275(3)Å, c = 15.541(3)Å, β = 107.95(1)°, V = 2303.8(8)Å³, D_c = 1.232 g cm⁻³, F(000) = 896, reflections with I > 3σ_I = 1269, parameters = 199, R = 0.0406, R_w = 0.0551, GOF = 2.441.
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