Synthesis, Crystal Structure, and Spectroscopic Characterization of the Phosphorane Iminato Complex \([\text{Cl}_2\text{AlNPCl}_3]_2\), and of the Phosphane Imine Complex \([\text{Me}_3\text{SiNPCl}_3 \cdot \text{AlCl}_3]\)

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Z. Naturforsch. 57 b, 1237–1243 (2002); eingegangen am 23. August 2002

Phosphorane Iminato Complex of Aluminum, Phosphane Imine Complex of Aluminum,
Single Source Precursor

Reaction of \(\text{Me}_3\text{SiNPCl}_3\) with \(\text{AlCl}_3\) yields the donor-acceptor complex \([\text{Me}_3\text{SiNPCl}_3 \cdot \text{AlCl}_3]\) (1). Thermal treatment of 1 and simultaneously removing the by-product \(\text{Me}_3\text{SiCl}\) results in the formation of \([\text{Cl}_2\text{AlNPCl}_3]_2\) (2). Crystallization of 2 from \(\text{CH}_2\text{Cl}_2\) yields colorless crystals which have been characterized by single crystal X-ray diffraction analysis \((P\bar{1}, a = 719.1(2), b = 843.5(2), c = 857.4(2) \text{ pm}, \alpha = 64.23(1), \beta = 76.62(1), \gamma = 68.21(1)^\circ, Z = 1, R_1 = 0.0498)\). The molecule exhibits site symmetry \(C_1\) (point group approximately \(C_{2h}\)) and shows as a characteristic feature a planar, almost square \(\text{Al}_2\text{N}_2\) four-membered ring with Al-N distances of 187.5(3) and 187.9(3) pm. The phosphorus atoms of the \(\text{PCl}_3\)-groups attached to the nitrogen atoms are almost located in the same plane as defined by the \(\text{Al}_2\text{N}_2\)-ring. Both new compounds have been characterized by IR, NMR, and MS spectroscopy.