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Polyhedron

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Bulky metal aryloxides, arylamides and sulphur and phosphorus analogues—III. Aluminium compounds derived from 2,4,6-TRI-t-butylaniline; X-ray structure of [AlMe{ μ -NHC₆H₂Bu^t_{2-4,6}-C(Me)₂CH₂-2}]₂ ☆

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Abstract

Treatment of AlMe₃ with ArNH₂ (Ar = C₆H₂Bu^t_{3-2,4,6}) in C₆H₁₄ at 60°C afforded [AlMe₂(μ -NHAr)]₂ (**1**). Heating the amide **1** either at 200°C for 3 h or (NMR) in C₆D₆ at 80°C for 3 weeks in a sealed tube led to methane elimination and formation of the cyclometallated compound [AlMe{ μ -NHC₆H₂Bu^t_{2-4,6}-C(Me)₂CH₂-2}]₂ (**2**); unexpectedly, the hydrogen atom abstracted from the N HAr ligand of **1** originated from the 2-t-butyl group rather than the NH. Deuterolysis of **2** yielded D₂NC₆H₂Bu^t_{2-4,6}-C(Me)₂CH₂D-2. AlCl₃ and ArNH₂ gave the 1 : 1 adduct AlCl₃(NH₂Ar) (**3**), which upon heating either at 150°C and 10⁻⁴ torr or (NMR) in C₆D₆, at 80°C for 24 h gave isobutene and the retro-Friedel Crafts product AlCl₃(NH₂C₆H₃Bu^t_{2,4}) (**4**). An excess of LiNHAr did not react with AlMe₃, but with AlCl₃ furnished Al(NHAr)₂Cl (**5**). The X-ray structure of the cyclo-metallated compound **2** showed it to be a pentacyclic centrosymmetric dimer, containing a central (AlN)₂ square [\angle Al□N > 1.965(6) Å], with the adjacent six-membered unsaturated alumino-heterocycles in a twisted boat conformation, one below and the other above the (AlN)₂ plane and the two anilido ligands in a *transoid* arrangement. The preparation of the metal anilides M(NHAr) [M = Li (**6**), SiMe₃ (**7**) or SnMe₃ (**8**)] is described.



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