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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-$NHC_6H_2Bu^t{}_2$-4,6-$C(Me)_2CH_2$-2}]$_2$ ☆

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Abstract

Treatment of $AlMe_3$ with $ArNH_2$ (Ar = $C_6H_2Bu^t{}_3{}^t$-2,4,6) in C_6H_{14} at 60°C afforded [$AlMe_2$(μ-NHAr)]$_2$ (**1**). Heating the amide **1** either at 200°C for 3 h or (NMR) in C_6D_6 at 80°C for 3 weeks in a sealed tube led to methane elimination and formation of the cyclometallated compound [**AlMe**{μ-$NHC_6H_2Bu_2^t$-4,6-$C(Me)_2CH_2$-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 $D_2NC_6H_2Bu_2^t$-4,6-$C(Me)_2CH_2D$-2. $AlCl_3$ and $ArNH_2$ gave the 1 : 1 adduct $AlCl_3(NH_2Ar)$ (**3**), which upon heating either at 150°C and 10^{-4} torr or (NMR) in C_6D_6, at 80°C for 24 h gave isobutene and the retro-Friedel Crafts product $AlCl_3(NH_2C_6H_3Bu_2^t$-2,4) (**4**). An excess of LiNHAr did not react with $AlMe_3$, but with $AlCl_3$ 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 [<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**), $SiMe_3$ (**7**) or $SnMe_3$ (**8**)] is described.

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* For Part II, see ref. 13.

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