Synthesis and Solid-State Structure of $(Li_4Am_3)^+\cdot\{Li[(\mu\text{-}Me)_2Al(Me)tBu]_2\}^-$ $\{Am = [PhNC(Ph)NPh]^-\}$: A Polymeric Species Incorporating a Lithium-Nitrogen Cluster Cation

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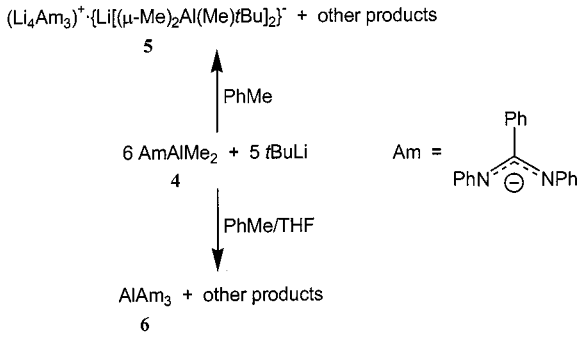
The sequential reaction of PhN(H)C(Ph)NPh (AmH) with $AlMe_3$ and tBuLi leads to the isolation of both the cluster $(Li_4Am_3)^+\cdot\{Li[(\mu\text{-}Me)_2Al(Me)tBu]_2\}^-$ **(5)** and the aluminium tris(amidinate) $AlAm_3$ **(6)**. In the solid state, **5** has a polymeric structure based on tetranuclear Li_4-cluster cations and lithium bis(aluminate) anions which associate by the formation of weak Li···MeAl bonds.

Introduction

The current interest being shown in the structures and reactivities of synthetically useful[1] lithium-containing heterobimetallic species[2] led us to synthesise lithium aluminate monomers $Me_2Al(\mu_2\text{-}OAr)_2Li$ (Ar = aryl), species which may play a role in conjugate additions to α,β-unsaturated ketones.[3] Moreover, it has recently been shown that tBuLi reacts with $Me_2AlN(Me)C(O)Ph$ to give a lithium aluminate which, in turn, yields a unique heterobimetallic mixed-anion ladder upon introduction of oxygen to the reaction mixture.[4] Whereas this chemistry utilised a reagent containing the $Me_2AlN(R)C(=X)$ fragment (X = O), the use of species in which X = N has also been fruitful. Accordingly, whereas the 1:1 lithium[5] and sodium[6] salts of 2-pyridylamine reagents [(2-Pyr)N(H)R] have been characterised, it has lately been reported that BuLi isomers react with the amidoalane $Me_2AlN(2\text{-}Pyr)Ph$ **(1)** to afford the hydride-containing compounds[7] $[Li(Me_2AltBu_2)_2]^-\{Li_8(H)[N(2\text{-}Pyr)Ph]_6\}^+$ **(2)** and $Li_7(H)[N(2\text{-}Pyr)Ph]_6$ **(3)** both of which incorporate cationic lithium-nitrogen clusters. In seeking to extend this new type of chemistry we have tested the reactivity of BuLi reagents towards Group 13 amidinates — species which have been extensively studied in both neutral[8] and cationic[9] contexts. Accordingly, we have employed $AmAlMe_2$ **(4)** $\{Am = [PhNC(Ph)NPh]^-\}$. Although simple lithiation[2b,10] has been reported for amidine reagents, as indeed have the syntheses of sodium[10c] and potassium[11] derivatives, we report here the isolation and characterisation of a more complex intermetallic amidinate compound. Reaction of tBuLi with **4** affords the tetralithium cluster $(Li_4Am_3)^+\cdot\{Li[(\mu\text{-}Me)_2Al(Me)tBu]_2\}^-$ **(5)** and also the aluminium tris(amidinate) $AlAm_3$ **(6)** (Scheme 1).



Scheme 1

Results and Discussion

Reaction of a solution of *N,N'*-diphenylbenzamidine[12] in toluene with $AlMe_3$ yields the aluminium amidinate $AmAlMe_2$ **(4)**, the treatment of which with tBuLi affords a solution from which crystals of **5** can be obtained (see Experimental Section). X-ray crystallography shows that in the solid state **5** is a mixed aggregate composed of an unusual lithium bis(aluminate) anion weakly bonded to a superlithiated cluster cation (Figure 1). The $\{Li[Me_2Al(Me)tBu]_2\}^-$ ion contains two Al centres arranged such that two methyl substituents on each are disposed tetrahedrally about Li(1). The resulting $Li[(\mu\text{-}C)_2Al]_2$ motif indicates some generality for the single previous report of this unit in a molecular context.[7] To the best of our knowledge the only close relative of this anion is present in the solvent-separated ion pair $[Li(DME)_3]^+\{Li[(\mu\text{-}OPh)_2AlEt_2]_2\}^-$.[13] Furthermore, the $\{Li[Me_2Al(Me)tBu]_2\}^-$ ion also constitutes part of a group of only a few characterised Group 1 metal-containing anions.[13,14] The mean M−(μ-C) distances (2.234 and 2.034 Å for M = Li and Al, respectively)

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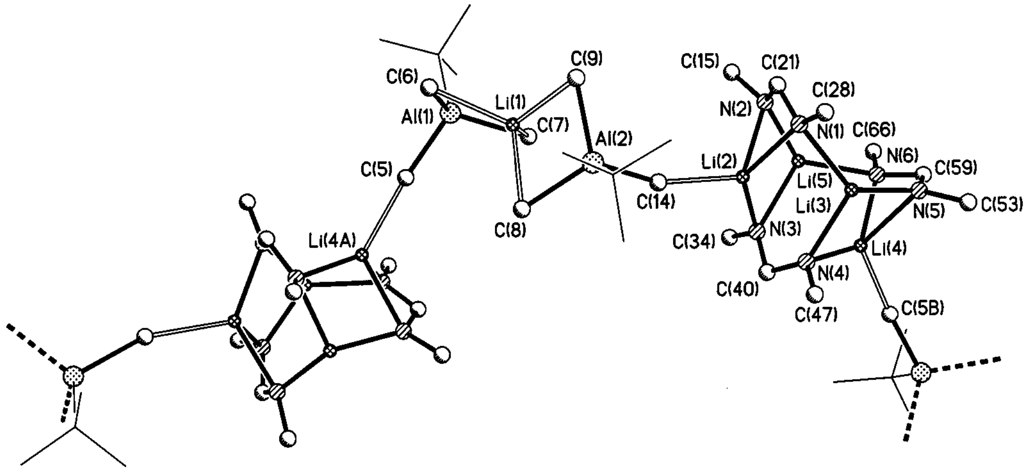

Figure 1. Polymeric structure of **5**; hydrogen atoms, phenyl rings (except the *ipso* atoms of the *N*-bonded rings) omitted for clarity; selected bond lengths (Å) and angles (°): N(1)−Li(3) 2.016(6), Li(3)−N(5) 1.992(6), N(6)−Li(5) 2.016(6), Li(5)−N(2) 1.996(6), N(2)−Li(2) 2.191(6), N(1)−Li(2) 2.089(7), N(4)−Li(3) 2.109(6), N(5)−Li(4) 2.199(6), N(6)−Li(4) 2.064(7), N(3)−Li(5) 2.124(6), Li(2)−N(2) 2.191(6), Li(4)−N(4) 2.130(6); N(2)−Li(5)−N(6) 127.0(3), N(1)−Li(3)−N(5) 125.5(3), N(1)−Li(2)−N(2) 63.7(2), Li(2)−N(2)−Li(5) 75.8(2), N(2)−Li(2)−N(3) 98.5(3), N(2)−Li(5)−N(3) 104.7(3), Li(2)−N(3)−Li(5) 74.9(2), N(5)−Li(4)−N(6) 64.0(2), N(5)−Li(3)−N(4) 105.2(3), N(5)−Li(4)−N(4) 97.7(3), Li(3)−N(5)−Li(4) 76.0(2), Li(3)−N(4)−Li(4) 75.2(2)

correlate accurately with those reported previously both in the molecular anion $[Li(Me_2Al\textit{t}Bu_2)_2]^-$ (2.25 and 2.07 Å)[7] and also in $LiAlEt_4$ (2.30 and 2.02 Å),[15] the polymeric structure of which is based on the same motif.

The cationic fragment of **5**, $(Li_4Am_3)^+$, takes the form of a lithium-nitrogen cluster. Whereas the lithium amide dication cluster recently reported to encapsulate a hydride ion exhibits $Li_8(amide)_6$ stoichiometry,[7] that of the monocationic moiety in **5** is $Li_4(amidinate)_3$. Thus the Li:organic ratio is retained, with the steric requirements of the *N,N′*-diphenylbenzamidinate ligand apparently requiring the formation of a smaller aggregate. The cationic component of **5** contains two types of charge-delocalised amidinate ligand, each of which interacts with the alkali metal centres through both of its sp^2-hybridised *N*-centres [C$\doteq$N$\doteq$C(Ph) = 121.6(3)−124.3(3)°]. Two amidinate ions, one on either side of the cluster, incorporate N(1)$\doteq$C(21)$\doteq$N(2) and N(5)$\doteq$C(59)$\doteq$N(6). These ligands both bond to Li(3) and Li(5), affording a concave eight-membered $(NCNLi)_2$ ring.[5e][6b,16] The Li−N interactions within this ring are short [1.992(6)−2.016(6) Å] in comparison to those between these nitrogen atoms and the remaining two lithium centres in the cluster cation [Li(2) and Li(4), 2.064(7)−2.199(6) Å]. Each of the two ligands which form this upper eight-membered ring interact individually with the Group 1 metal centres in the lower tier in an asymmetrically bidentate fashion. Thus, Li(2)−N(1) and Li(4)−N(6) average to 2.077 Å, while Li(2)−N(2) and Li(4)−N(5) average to 2.195 Å. This lengthening of the latter Li−N interactions is consistent with the bonding pattern observed in the eight-membered $(NCNLi)_2$ ring: N(1) and N(6) form relatively short bonds to Li(2) and Li(4) (mean Li−N = 2.016 Å) but interact more weakly with both Li and C centres within the ring [mean N$\doteq$C(Ph) = 1.342 Å]. Conversely, N(2) and N(5) form stronger bonds with both Li and C centres in this ring [mean Li−N = 1.994 Å; mean N$\doteq$C(Ph) = 1.325 Å] at the expense of short interactions with the two basal cations. These last two Li^+ centres are straddled by the third ligand [Li(2)−N(3) and Li(4)−N(4) = 2.113(6) and 2.130(4) Å, respectively]. However, this basal anion is orientated such that both Li(2)···N(4) and Li(4)···N(3) are extended (mean 2.583 Å) so as to be of no bonding significance. Thus, the basal tier of the cluster can be viewed as a four-membered $(LiN)_2$ ring which has undergone the fracture of two *trans*-edges to afford a pair of LiN fragments.[17] The remaining interactions between cluster base and upper $(NCNLi)_2$ ring involve the stabilisation of Li(3) and Li(5) by N(4) and N(3), respectively (mean Li−N = 2.117 Å).

The solid-state structure of **5** reveals a polymer by virtue of weak interactions between Li(2) and the terminal Me group [C(14)] in the anion and between C(5) in this anion and the analogous alkali metal centre [Li(4A)] in the adjoining cationic unit [mean Li···C(Me) = 2.295 Å] (Figure 1). These interactions raise the coordination of the otherwise low-valent Li centres in the cleaved $(LiN)_2$ ring (see above) at the base of the cluster cation. This positively charged component of **5** contrasts with normally observed ion-separated species in which otherwise isolated Li^+ centres are coordinated by a neutral Lewis base,[18] and with the recent observation of $(Li_2)^{2+}$ clusters.[19] Aggregated and charged lithium-containing clusters have also been reported in the lithium "ate" complex $(Li{\cdot}HMPA)^+{\cdot}[Li_5(N{=}CPh_2){\cdot}HMPA]^-$ [HMPA = $O{=}P(NMe_2)_3$][14b] and $Ta(4\text{-}Me\text{-}C_6H_4)_6^-$ $[Li_4Br_3(Et_2O)_7]^+$,[20] as well as in both **2** and **3**.[7]

Attempts to understand the mechanism of formation of **5** are ongoing. The observation that the amorphous solid obtained by removing the solvent from a 1:1 mixture of *N,N′*-diphenylbenzamidine and $AlMe_3$ is $AmAlMe_2$ **(4)**[21] points to the first stage intermediate in the synthesis of **5** being the aluminium mono(amidinate). However, the treatment of this species with *t*BuLi initiates a complex reaction which requires further elucidation. The isolation (see Experimental Section) and structural characterisation of the aluminium tris(amidinate) $AlAm_3$ **(6)** may also be instructive — attempts to recrystallise THF-solvated (i.e. oligomerised) **5** afforded only a co-crystalline mixture which ^1H NMR spectroscopy suggests contains **5** (or its solvated and deaggregated analogue) and **6**. Mechanical separation of the crystals allows the characterisation of pure $AlAm_3$, for which formulation the solid-state structure (Figure 2) revealed one molecule of lattice toluene.

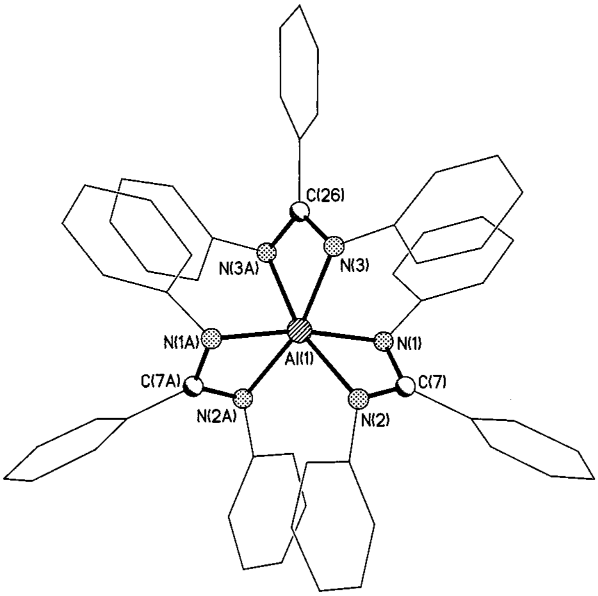

Figure 2. Molecular structure of monomeric **6**·PhMe; hydrogen atoms and lattice toluene molecule omitted for clarity; selected bond lengths (Å) and angles (°): Al(1)−N(1) 1.997(3), Al(1)−N(2) 1.981(3), Al(1)−N(3) 1.985(3); N(1)−Al(1)−N(2) 66.7(1), N(3)−Al(1)−N(3A) 66.8(2)

While the isolation and characterisation of **4**, the novel polymer **5** and also of **6** cast some light on the complex processes occurring in systems of the type discussed here, further mechanistic investigations are clearly required and will be reported subsequently. More generally, we are seeking to vary the choice of *N*-centred organic residue and organoaluminium reagent in an attempt to generate new compounds related to **5**, and so investigate the extent of this chemistry.

Experimental Section

General: All operations were carried out using standard Schlenk techniques. *N,N'*-diphenylbenzamidine was used as received from Lancaster. Toluene (freshly distilled from sodium), hexane and THF (freshly distilled from sodium-potassium alloy) and trimethylaluminium and *tert*-butyllithium were added direct to the nitrogen-filled Schlenk tube using standard syringe techniques. − NMR spectroscopy: Bruker DRX 400 (400.137 MHz for ^1H). For ^1H NMR spectroscopy, $[D_6]$benzene and $[D_8]$THF as solvents, TMS at 25 °C as external standard.

Dimethylaluminium *N,N'*-Diphenylbenzamidinate, $4 \cdot {}^1/_3$PhMe: *N,N'*-diphenylbenzamidine (0.272 g, 1 mmol) was dissolved in toluene (2 mL) and cooled to −78 °C, whereupon $AlMe_3$ (0.5 mL, 1 mmol, 2 M in toluene) was added. The resultant mixture was warmed to room temperature and stirred for 30 minutes. Reflux afforded a yellow solution from which $\mathbf{4} \cdot {}^1/_3$PhMe was deposited as an amorphous solid upon removal of the solvent. Yield 73%, m.p. 176−178 °C. − $C_{70}H_{71}Al_3N_6$ (1076): calcd. C 78.07, H 6.60, N 7.81; found C 76.53, H 6.43, N, 8.35. − ^1H NMR (400 MHz, C_6D_6): δ = 7.19−6.49 (m, 16 H, Ph + $^1/_3$*Ph*Me), 2.15 (s, 1 H, $^1/_3$Ph*Me*), −0.01, −0.28 (m, 6 H, AlMe).

Compound 5: As for **4** but after stirring at room temperature for 30 minutes the solution was returned to −78 °C and treated with *t*BuLi (0.49 mL, $^5/_6$ mmol, 1.7 M in pentane). The resultant suspension was warmed to room temperature and dissolved at reflux with the addition of toluene (20 mL). The yellow solution was stored at +70 °C for two days whereupon colourless cubes of **5** were deposited. Yield 35% (based on *t*BuLi consumed), m.p. decomp. from 220 °C. − $C_{71}H_{81}Al_2Li_5N_6$ (1107): calcd. C 76.96, H 7.32, N 7.59; found C 77.95, H 6.69, N, 7.18. − ^1H NMR (400 MHz, $[D_8]$THF): δ = 7.05, 7.02 (br. m, 18 H, Ph), 6.83 (br. s, 12 H, Ph), 6.48 (br. s, 15 H, Ph), 0.70 (m, $^3J_{H-Al}$ = 5.0 Hz, 18 H, *t*Bu), −1.33 (sext., $^2J_{H-Al}$ = 5.7 Hz, 18 H, AlMe).

Aluminium Tris(*N,N'*-diphenylbenzamidinate), 6·PhMe: As for **5** utilising an initial solvent mixture of 5:1 toluene/THF (1:0.2 mL). The suspension produced after *t*BuLi addition and reflux was filtered and the resultant solution stored at room temperature for 1 week, whereupon a complex co-crystalline mixture was obtained. Mechanical separation allowed the characterisation of **6**·PhMe. M.p. 298−300 °C. − $C_{64}H_{53}Al_3N_6$ (933): calcd. C 82.31, H 5.68, N 9.00; found C 80.29, H 5.55, N 9.27. − ^1H NMR (400 MHz, C_6D_6): δ = 7.34−6.89 (m, 16 H, Ph + $^1/_3$*Ph*Me), 2.32 (s, 1 H, $^1/_3$Ph*Me*).

Crystal Data for 5: $C_{71}H_{81}Al_2Li_5N_6$, M = 1107.08, monoclinic, space group *Cc*, a = 13.7160(3), b = 23.9360(7), c = 20.6990(6) Å, β = 90.5220(19)°, U = 6795.3(3) Å^3, Z = 4, D_c = 1.082 g cm^{-3}, Mo-K_α (λ = 0.71070 Å), μ = 0.086 mm^{-1}, T = 180(2) K. 26396 reflections (10837 unique, $\theta < 50.10°$, R_{int} = 0.0481). Refinement on F^2 values of all data gave $wR2$ = 0.1453, conventional R = 0.0547 on F values of 8799 reflections with $F^2 > 2\sigma(F^2)$, 754 parameters. Residual electron density extrema 0.49 and −0.28 e Å^{-3}.

Crystal Data for 6·PhMe: $C_{64}H_{53}AlN_6$, M = 933.10, monoclinic, space group *C2/c*, a = 18.2750(12), b = 18.7770(11), c = 16.2890(9) Å, β = 112.547(3)°, U = 5162.3(5) Å^3, Z = 4, D_c = 1.201 g cm^{-3}, Mo-K_α (λ = 0.71070 Å), μ = 0.086 mm^{-1}, T = 180(2) K. 8348 reflections (4517 unique, $\theta < 49.94°$, R_{int} = 0.0358). Refinement on F^2 values of all data gave $wR2$ = 0.2166, conventional R = 0.0707 on F values of 3185 reflections with $F^2 > 2\sigma(F^2)$, 307 parameters. Residual electron density extrema 0.69 and −0.64 e Å^{-3}.

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-145946 (**5**) and -145945 (**6**·PhMe). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

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