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Main group complexes incorporating 1,3-bis(furyl)-1,1,3,3-tetramethyldisilazide ligands

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Abstract

The lithium salt of the bis-furyl substituted disilazide anion, Li{i} [{i} = N(SiMe$_2$R)$_2$ where R = 2-methylfuryl] has been examined as a ligand transfer reagent for the synthesis of group 2 (magnesium) and group 13 (aluminium) compounds. Salt metathesis between Li{i} and AlMe$_2$Cl afforded the expected dimethyl species, Al{i}Me$_2$ (**1**), which was isolated as a colourless oil. In contrast the corresponding aluminium dichloride, synthesized from Li{i} and AlCl$_3$, gave crystalline products as both the THF adduct Al{i}Cl$_2$(THF) (**2a**) and the base-free derivative, Al{i}Cl$_2$ (**2b**). The homoleptic magnesium bis(amide) Mg{i}$_2$ (**3**) was also synthesized. X-ray crystallographic analysis of **2a** reveals a four-coordinate distorted tetrahedral metal, in which neither of the furyl-substituents interact with the metal. In contrast, the aluminium in the base-free dichloride **2b** is five-coordinate, containing the first structurally characterized example in which the amide binds with a κ^1N,O,O′-bonding mode, involving coordination of both furyl-substituents at the N-bound metal.
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1. Introduction

Metal amides (**a**, Fig. 1) represent a fundamentally important group of compounds that have been widely studied over the years [1], due largely to the steric and electronic diversity attainable through facile derivitization of the nitrogen substituents. A prominent class of amide that has stimulated particular interest is that based around the silylamide framework, $[N(SiR'_3)_nR_{2-n}]^-$ (**b**, $n = 1$; **c**, $n = 2$), initially studied in the 1960s by Bürger and Wannagat working with the simple hexamethyldisilazide anion [2]. These investigations led to the realisation of what is referred to as the 'silicon effect' [3], in which it was noted that the use of silicon substituents leads to a reduction in the electron density at the N-bonded metal.

More recently, the coordination chemistry of silazides has been further enhanced through the incorporation of

donor functionalities (L), in particular those that are able to interact with the N-bound metal fragment, thereby forming stable chelate species (**d** and **e**). Amongst the many variations that are possible within this general class of ligand, the systems that have arguably afforded the greatest success with respect to their application in the development of transition metal, main group and lanthanoid chemistry are those based on the amido diphosphines $[N(SiMe_2-CH_2PR'_2)_2]^-$, pioneered by Fryzuk et al. [4]. X-ray crystallographic data have shown that by far the most common bonding mode for these systems are those in which both phosphine units are involved in coordination to the N-bound metal (**f**), although examples in which only one such unit is coordinated have been reported (**g**) at aluminium [5], zirconium [6] and iron [7]. To date there have been no structurally characterized examples reported in which the ligand is acting as an κ^1N-ligand through only the amide nitrogen atom.

We have recently become interested in the development of a related disilazide-based system, in which the neutral

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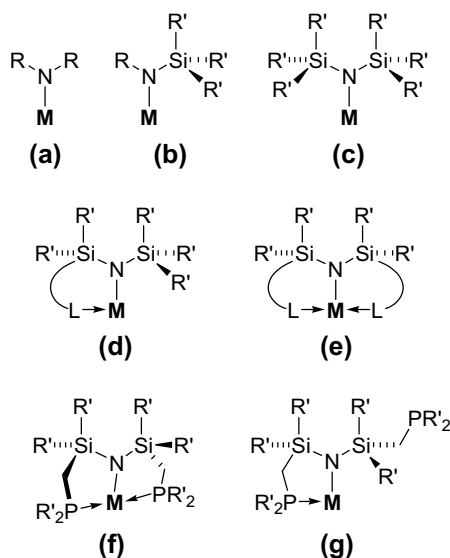 *E-mail address:* m.p.coles@sussex.ac.uk (M.P. Coles).

Fig. 1. General classes of amide and silazide ligand.

donor groups consist of an oxygen-functionality contained within a furyl-ring. Previous results from our group have demonstrated that the lithium salts, $[Li(N\{SiMe_2R\}_2)]_n$ (R = furyl [8], 2-methylfuryl, 2-trimethylsilylfuryl) may be accessed directly from the reaction of 3 equiv. of the appropriate lithium furyl salt with 1,3-dichloro-1,1,3,3-tetram-

ethyldisilazane [9]. Synthesis of the corresponding potassium reagents was achieved *via* conversion of the Li-salts to the neutral amine and subsequent reaction with KNH_2. Salt metathesis has allowed a range of group 4 metal halide complexes to be prepared, that have been the subject of a brief study into their potential to act as pre-catalysts in the MAO mediated polymerization of ethylene [10].

During these studies, the molecular structures of selected examples have been solved by single crystal X-ray diffraction techniques, enabling the extent of furyl substituent contribution to bonding to be examined in the solid-state. Different bonding regimes have been observed to date (Fig. 2), including examples of formally terminal amide bonding involving interaction of neither furyl group (κ^1N-bonding A-type) at titanium, or a single metal–oxygen bond (κ^1-N,O-bonding B-type) at zirconium. Related bridging amides within bimetallic structures have also been noted in the solid-state, with $\kappa^{1,2}$N-κ^1O-bonding (D-type) to potassium and $\kappa^{1,2}$N-κ^1O-κ^2O'-bonding (E-type) to lithium, with participation by one and both furyl groups, respectively [9]. Several other bonding possibilities are available for this ligand, and in this contribution we wish to report the first examples of group 13 metal complexes of this ligand, including a previously unreported κ^1N,O,O'-type of bonding mode (C-type) in which both furyl groups are bonded to the N-bound metal.
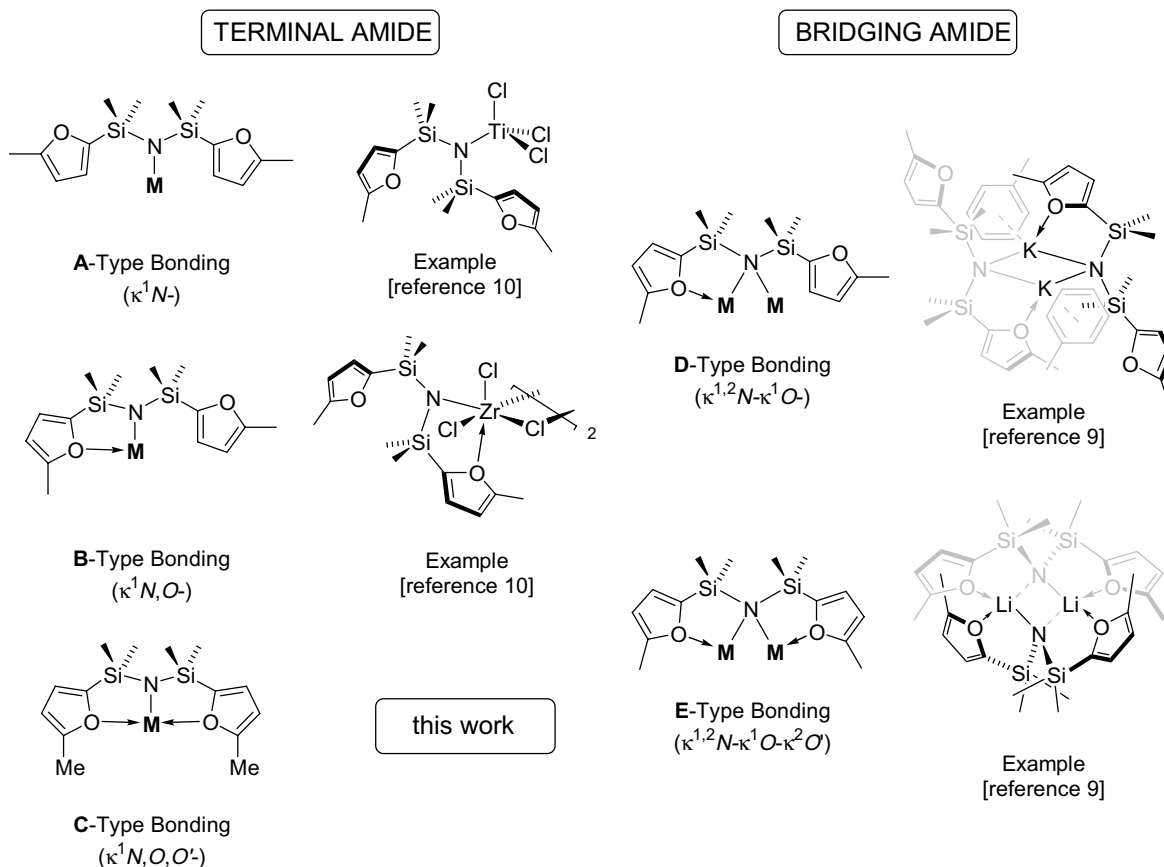

Fig. 2. Classification of selected bonding modes involving the 1,3-bis(furyl)-1,1,3,3-tetramethyldisilazide ligand.

2. Experimental

2.1. General considerations

All manipulations were carried out under dry nitrogen using standard Schlenk-line and cannula techniques, or in a conventional nitrogen-filled glovebox. Solvents were dried over appropriate drying agent and degassed prior to use. NMR spectra were recorded using a Bruker Avance DPX 300 MHz spectrometer at 300.1 (^{1}H) and 75.4 (^{13}C{^{1}H}) or a Bruker AMX 500 MHz spectrometer at 500.1 (^{1}H) and 125.7 (^{13}C{^{1}H}) MHz, from samples at 25 °C in [^{2}H$_6$]-benzene, unless otherwise stated. Coupling constants are quoted in Hz. Proton and carbon chemical shifts were referenced internally to residual solvent resonances. Elemental analyses were performed by Boyer at London Metropolitan University.

AlMe$_2$Cl (2.0 M solution in hexanes, Aldrich), AlCl$_3$ (Aldrich) and MgBr$_2$(OEt$_2$) (Aldrich) were purchased from commercial sources and used without further purification. The lithium reagent Li{i} [{i} = N(SiMe$_2$R)$_2$ where R = 2-methylfuryl] was made according to published procedures [9].

2.2. Synthesis of Al{i}Me$_2$ (1)

A solution of AlMe$_2$Cl (0.34 mL of a 2.0 M solution in hexanes, 0.67 mmol) was added to a solution of Li{i} (0.20 g, 0.67 mmol) in toluene (40 mL) at room temperature. A white precipitate was observed within 5 min of addition. The resulting solution was stirred at ambient temperature for a further 24 h. The solvent was removed *in vacuo* and the residue was extracted with pentane (30 mL) and filtered through celite, affording a clear, colourless solution. Removal of all volatiles *in vacuo* afforded the product as a colourless oil. Yield 0.19 g (81%).

^{1}H NMR (300 MHz): δ 6.38 (d, $J = 3.0$, 2H, furyl-CH_α), 5.77 (m, $J = 3.0$, 2H, furyl-CH_β), 2.10 (s, 6H, furyl-CH_3), 0.37 (s, 12H, SiMe_2), −0.52 (s, 6H, AlMe_2). ^{13}C NMR (75 MHz): δ 161.5 (furyl-C4), 155.4 (furyl-C3), 120.0 (furyl-C2), 108.1 (furyl-C1), 13.6 (furyl-CH$_3$), 3.5 (SiMe_2), −6.8 (AlMe_2). Mass spectrometry: (EI, m/z): 334 [M−CH$_3$]$^+$.

2.3. Synthesis of Al{i}Cl$_2$(THF) (2a)

Pre-cooled THF (−78 °C, 40 mL) was added to a dry mixture of AlCl$_3$ (0.20 g, 1.50 mmol) and Li{i} (0.45 g, 1.50 mmol) and the resulting solution was allowed to warm to ambient temperature and stirred for 24 h. The volatile components were removed *in vacuo* and the residue was extracted with pentane (40 mL) concentrated and cooled to −45 °C. The product was isolated as colourless crystals, although upon exposure to vacuum the solid became a sticky oil, precluding the acquisition of meaningful analytical data. Due to this change of state, the precise yield was not calculated; however, the yield for **2b** was calculated at

60% based on the amount of AlCl$_3$ used in this stage of the preparation, therefore the yield for **2a** must be greater than this amount.

2.4. Synthesis of Al{i}Cl$_2$ (2b)

A sample of **2a** was prepared according to the procedure outlined above. The crude sticky solid was sublimed at 100–120 °C at 10^{-6} mbar. The sublimate was crystallized from a saturated pentane solution at −45 °C, affording the base-free compounds as colourless crystals. Yield 0.31 g (60%, based on AlCl$_3$ used in the synthesis of **2a**). Anal. Calc. for C$_{14}$H$_{22}$NAlCl$_2$O$_2$Si$_2$ (390.39): C, 43.08; H, 5.64; N, 3.59. Found: C, 43.17; H, 5.64; N, 3.70%. ^{1}H NMR (300 MHz): δ 6.27 (d, $J = 3$, 2H, CH_α), 5.65 (m, $J = 2$, 2H, CH_β), 2.39 (s, 6H, furyl-CH_3), 0.35 (s, 12H, SiMe_2). ^{13}C NMR (75 MHz): δ 159.9 (furyl-C4), 157.2 (furyl-C3), 120.2 (furyl-C2), 108.8 (furyl-C1), 14.1 (furyl-CH$_3$), 2.9 (SiMe_2). Mass spectrometry: (EI, m/z): 389–393 [M ^{35}Cl/^{37}Cl isotopes]$^+$.

2.5. Synthesis of Mg{i}$_2$ (3)

THF (30 mL) was added to a dry mixture of MgBr$_2$(OEt$_2$) (0.20 g, 0.77 mmol) and Li{i}$_2$ (0.46 g, 1.55 mmol), and the resulting solution was stirred at ambient temperature for 24 h. The solvent was removed *in vacuo* and the residue was extracted with pentane (30 mL) and filtered through celite, affording a clear, colourless solution. Removal of all volatiles *in vacuo* afforded the product as a colourless oil. Yield 0.36 g (77%). ^{1}H NMR (300 MHz): δ 6.38 (d, $J = 3.0$, 4H, furyl-CH_α), 5.73 (m, $J = 3.0$, 4H, furyl-CH_β), 2.02 (s, 12H, furyl-CH_3), 0.46 (s, 24H, SiMe_2). ^{13}C NMR (75 MHz): δ 164.0 (furyl-C4), 154.9 (furyl-C3), 119.4 (furyl-C2), 107.3 (furyl-C1), 13.2 (furyl-CH$_3$), 4.3 (SiMe_2). Mass spectrometry: (EI, m/z): 608 [M]$^+$, 593 [M−CH$_3$]$^+$.

2.6. Crystallographic details

Details of the crystal data, intensity collection and refinement for complexes **2a** and **2b** are collected in Table 1. Crystals were covered in an inert oil and suitable single crystals were selected under a microscope and mounted on a Kappa CCD diffractometer. The structures were refined with SHELXL-97 [11].

3. Results and discussion

During a recent study of titanium and zirconium halide compounds incorporating ligand {i}, we attempted to generate the corresponding methyl species using a range of main group metal reagents known to promote this transformation in related systems [10]. Instead of proceeding as intended, however, the predominant pathway involved transfer of the amide from the transition element to the main group metal, affording the compounds Al{i}Me$_2$ (1)

Table 1
Crystal structure and refinement data for Al{i}Cl$_2$(THF) (**2a**) and Al{i}Cl$_2$ (**2b**)

	2a	**2b**
Formula	C$_{18}$H$_{30}$AlCl$_2$NO$_3$Si$_2$	C$_{14}$H$_{22}$AlCl$_2$NO$_2$Si$_2$
Formula weight	462.49	390.39
Temperature (K)	173(2)	173(2)
Wavelength (Å)	0.71073	0.71073
Crystal size (mm)	0.25 × 0.20 × 0.10	0.30 × 0.25 × 0.15
Crystal system	Triclinic	Orthorhombic
Space group	$P\bar{1}$ (No. 2)	$P2_12_12_1$ (No. 19)
a (Å)	7.7326(4)	8.5431(11)
b (Å)	9.8184(5)	13.9999(15)
c (Å)	16.4479(7)	16.595(2)
α (°)	91.306(3)	90
β (°)	96.615(3)	90
γ (°)	102.424(2)	90
V (Å^3)	1209.93(10)	1984.8(4)
Z	2	4
D_c (Mg m^{-3})	1.27	1.31
Absorption coefficient (mm^{-1})	0.42	0.50
θ Range for data collection (°)	3.46–26.02	3.42–25.99
Reflections collected	12 754	14 756
Independent reflections [R_{int}]	4745 [0.049]	3795 [0.060]
Reflections with $I > 2\sigma(I)$	3745	3204
Data/restraints/parameters	4745/0/246	3795/0/202
Goodness-of-fit on F^2	1.008	1.028
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.042$, $wR_1 = 0.095$	$R_1 = 0.039$, $wR_1 = 0.076$
R indices (all data)	$R_1 = 0.061$, $wR_1 = 0.105$	$R_1 = 0.055$, $wR_1 = 0.082$
Largest difference in peak/hole (e Å^{-3})	0.41 and −0.26	0.22 and −0.26

and Mg{i}$_2$ (**3**) illustrated in Scheme 1. Although initially isolated as dark brown (Al) or red (Mg) oils by this procedure, suggesting contamination by unknown transition-metal side products, distillation of the crude products under high vacuum (100–120 °C, 10^{-6} mbar) showed that in their pure state the products are colourless, although remain as oils. Indeed this was verified using a more rational synthetic approach whereby the pure compounds **1** and **3** were made directly from the salt metathesis reaction of Li{i} with AlMe$_2$Cl and MgBr$_2$(OEt$_2$), respectively.
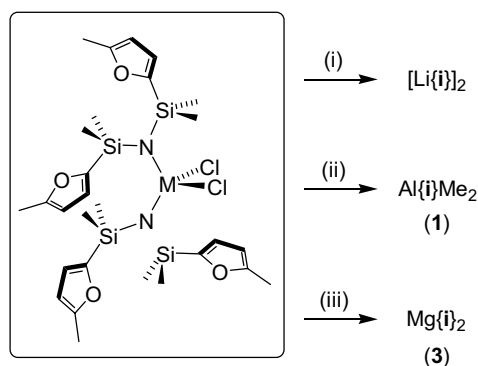
The ^{1}H and ^{13}C NMR resonances were as predicted for these compounds, and indicated a symmetrical environment for the ligand with single sets of resonances for the Si*Me*$_2$ and furyl components. Assuming only a single metal is present (in agreement with the highest mass fragments from EI$^+$ mass spectral data) this bonding may be assigned to a static structure containing either A-type (κ^1N-) in which neither furyl group is involved with bonding to the main group element, or C-type (κ^1N,O,O′-) with the ligand acting as an 'XL$_2$' donor to the metal. We have found that this solution-state measurement is, however, not a reliable indication of the bonding mode of the ligand in the solid-state, illustrated clearly by the compound Zr{i}Cl$_3$ that has similar spectroscopic properties and yet adopts a non-symmetric κ^1N,O-mode (B-type) in the solid-state (determined crystallographically). These data imply that a fluxional process is occurring in solution in which the two furyl groups of the ligand are interchanging positions at the metal, or that a monomer–dimer equilibrium is present involving a similar exchange process.

In the case of aluminium, in which there is scope for derivitization of the remaining two ligands at the metal, complexes related to the dimethyl species **1** that possessed more favourable crystalline properties were targeted, thereby allowing examination of the bonding mode of the ligand in the solid-state. Given that group 13 halides are often more crystalline than their methyl analogues (e.g. AlCl$_3$ mp = 190 °C; AlMe$_3$ mp = 15 °C) a logical starting point was to target the aluminium compound, Al{i}Cl$_2$. The synthesis was achieved from a stoichiometric reaction between Li{i} and AlCl$_3$ in THF and the resultant complex was crystallized from pentane at low temperature. Upon exposure to vacuum, these crystals rapidly decrepitated, precluding the acquisition of full analytical data. However, it was possible to quickly mount a single crystal in an inert oil and obtain the molecular structure by X-ray diffraction (**2a**). Given the apparent facile loss of solvent, a sample of the crude material was sublimed prior to crystallization, affording more stable colourless crystals (**2b**) that were fully analysed.

Scheme 1. (i) M = Ti: LiMe, Et$_2$O, room temperature; (ii) M = Zr: AlMe$_3$, toluene, reflux; (iii) M = Zr: MgMe$_2$, toluene, reflux.
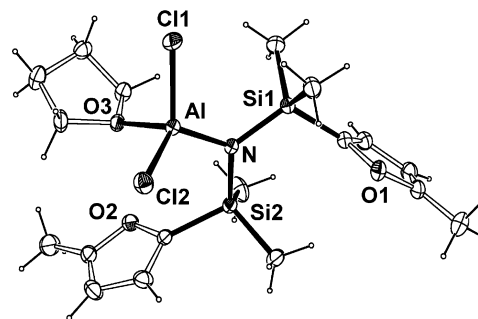

Fig. 3. ORTEP representation of the molecular structure of Al{i}Cl$_2$(THF) (**2a**) with ellipsoids at the 30% probability level.

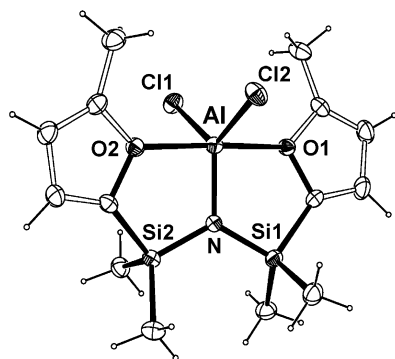Fig. 4. ORTEP representation of the molecular structure of Al{i}Cl$_2$ (2b) with ellipsoids at the 30% probability level.

The molecular structures of **2a** and **2b** are illustrated in Figs. 3 and 4, respectively; crystal structure and refinement data are collected in Table 1 and selected bond lengths and angles in Tables 2 and 3.

As originally isolated from solution, the compound retains a molecule of THF at the aluminium centre, corresponding to the molecular formula Al{i}Cl$_2$(THF) (**2a**). The geometry is distorted tetrahedral at aluminium, with angles in the range 99.88(6)–118.69(7)°. All other bond parameters are similar to those in the previously reported N-bonded 1-aza-allyl complex, Al[N(SiMe$_3$)C(Ph)C(Si-Me$_3$)$_2$]Cl$_2$(THF) [12], in which a four-coordinate centre consisting of an 'AlNCl$_2$(THF)' unit is present. The amide ligand is present in an A-type bonding pattern with no interactions between the furyl oxygen atoms and the metal. This is entropically disfavoured and is likely the cause of the facile loss of THF upon exposure to vacuum, as noted in the 1-aza-allyl system described above.

Table 2
Selected bond lengths (Å) and angles (°) for Al{i}Cl$_2$(THF) (**2a**)

Al–N	1.7997(19)	Al–O(3)	1.8513(16)
Al–Cl(1)	2.1466(9)	N–Si(1)	1.741(2)
Al–Cl(2)	2.1300(10)	N–Si(2)	1.742(2)
N–Al–Cl(1)	115.23(7)	N–Al–Cl(2)	118.69(7)
N–Al–O(3)	109.01(8)	Cl(1)–Al–O(3)	99.88(6)
Cl(2)–Al–O(3)	104.64(6)	Cl(1)–Al–Cl(2)	107.24(4)
Al–N–Si(1)	120.0(11)	Al–N–Si(2)	118.94(11)
Si(1)–N–Si(2)	120.96(11)		

Table 3
Selected bond lengths (Å) and angles (°) for Al{i}Cl$_2$ (**2b**)

Al–N	1.824(2)	Al–Cl(1)	2.1459(11)
Al–Cl(2)	2.1389(12)	Al–O(1)	2.101(2)
Al–O(2)	2.149(2)	N–Si(1)	1.725(2)
N–Si(2)	1.719(2)		
O(1)–Al–O(2)	177.64(9)	O(1)–Al–N	89.31(9)
O(1)–Al–Cl(1)	92.78(6)	O(1)–Al–Cl(2)	89.46(7)
O(2)–Al–N	88.37(9)	O(2)–Al–Cl(1)	88.00(7)
O(2)–Al–Cl(2)	92.11(7)	N–Al–Cl(1)	118.85(9)
N–Al–Cl(2)	121.78(9)	Cl(1)–Al–Cl(2)	119.34(5)
Al–N–Si(1)	118.94(13)	Al–N–Si(2)	118.16(13)
Si(1)–N–Si(2)	122.76(14)		

The molecular formula of the compound crystallized after sublimation corresponds to the monomeric base-free species, Al{i}Cl$_2$ (**2b**), in which the THF has been lost from the metal coordination sphere. The most notable feature of the structure is the bonding of the amide ligand, that has compensated for the loss of THF by adopting a previously unobserved κ^1N,O,O′- (C-type) mode with both furyl oxygen atoms bonded to the metal. The resultant geometry at aluminium is therefore close to an ideal trigonal bipyramid (τ value [13] = 0.97) with the furyl substituents axial and the amide nitrogen and chlorides in equatorial positions. The corresponding amido diphosphine compound, Al(N-{SiMe$_2$CH$_2$P^{i}Pr$_2$}$_2$)Cl$_2$, crystallizes with an analogous arrangement of groups around the metal, with trans apical phosphine donors [5]. In this compound, a pronounced twisting is noted in the backbone of the ligand (dihedral angle between AlNSi$_2$ and AlNP$_2$ planes = 37.8°) compared with the corresponding value in **2b** of 19.8°.

The aluminium–nitrogen distance is somewhat longer than in the THF-adduct [1.824(2) Å and 1.7997(19) Å, respectively] likely to reflect the increased coordination number in the five-coordinate system. The Al–O$_{furyl}$ distances in **2b** [Al–O(1) = 2.101(2) Å; Al–O(2) = 2.149(2) Å] are considerably longer compared with the Al–O$_{THF}$ distance in **2a** [Al–O(3) = 1.8513(16) Å]. In this case, this is most likely due to a combination of the better electron donating ability of THF vs. a furyl-group in addition to the changes in coordination number, the latter feature clearly indicated in the compounds AlCl$_3$(THF) [14], and AlCl$_3$(THF)$_2$ [15] and with Al–O$_{THF}$ bond lengths of 1.798(6) Å and 1.990(1) Å, respectively.

In summary, we have shown that the 1,3-bis(furyl)-1,1,3,3-tetramethyldisilazide ligand readily transfers from a group four metal centre to a main-group element, to afford the aluminium dimethyl compound Al{i}Me$_2$ (**1**) and the homoleptic magnesium diamide, Mg{ii}$_2$ (**3**). Crystalline samples of related aluminium dichlorides show monodentate coordination to the metal in the four-coordinate THF adduct, Al{i}Cl$_2$(THF) (**2a**), and for the first time, structural evidence for interaction of both furyl substituents in a κ^1N,O,O′-bonding mode in trigonal bipyramidal Al{i}Cl$_2$ (**2b**). Investigation of the bonding of this anion to the group 3 elements yttrium and scandium and the chemistry associated with these complexes are ongoing in our laboratory and will be reported in due course.

Appendix A. Supplementary material

CCDC 631480 and 631481 contain the supplementary crystallographic data for **2a** and **2b**. The data can be obtained free of charge via htpp://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallo-

graphic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2007.02.042.

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