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Synthetic, spectral and structural studies of mononuclear tris(κ^2-amidate) aluminium complexes supported by tripodal ligands

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ABSTRACT

The synthesis and characterization of mononuclear tris(κ^2-amidate) aluminium complexes supported by the tripodal ligands, $[N(o\text{-PhNC(O)R})_3]^{3-}$ (R = ^{i}Pr and ^{t}Bu), are described. The molecular structures of $[Al(N(o\text{-PhNC(O)}^i\text{Pr})_3)]$ and $[Al(N(o\text{-PhNC(O)}^t\text{Bu})_3)]$ have been determined by X-ray diffraction studies. Both neutral six-coordinate aluminium complexes display coordination geometries that are intermediate between octahedral and trigonal prismatic. Solution-state NMR studies (^{1}H, ^{13}C and ^{27}Al) indicate that these structures are non-fluxional in solution. Detailed analysis of the solid-state structures shows that slight changes in the relative size of the amidate acyl substituents do not significantly impact the solid-state structures. However, large substituents may be required to prevent the formation of multinuclear species.

1. Introduction

Deprotonated organic amides (amidates) have recently been recognized as potent ligand scaffolds for metal-ion mediated catalytic and stoichiometric transformations [1,2]. Amidate ligands can interact with metal centres through a variety of coordination modes, including: (1) monodentate (where the amidate coordinates through either the *N*- or *O*-amidato donor), (2) bridging (where the *N*- and *O*-donors coordinate to two separate metal-ions), and (3) chelating (where the *N*- and *O*-donors coordinate to the same metal-ion). The possible coordination modes for amidate ligands interacting with a single metal centre are shown in Fig. 1. Studies involving deprotonated amides and aluminium centres have largely focused on the synthesis and characterization of organoaluminium amidates. The majority of these organoaluminium amidate complexes are multinuclear or oligomeric species in which the amidate donors bridge more than one aluminium centre [3–10]. However, two separate studies have identified monomeric organoaluminium complexes stabilized by *O*-amidate ligands [11,12].

Recently, Stahl and co-workers have demonstrated that the dimeric, homoleptic amidoaluminium species ($Al_2(NMe_2)_6$) can be used to efficiently catalyze the transamidation of carboxamides [13]. In subsequent mechanistic studies [14,15], they were able to use NMR spectroscopy to characterize the resting state of the catalyst, an Al(III) tris(κ^2-amidate) species.

These spectroscopic studies and the dearth of structurally characterized tris(κ^2-amidate) metal complexes [2,16–18] prompted us to explore the coordination chemistry of Al(III) with the tripodal, tris(amidate) ligand systems, $[N(o\text{-PhNC(O)R})_3]^{3-}$ (R = alkyl) recently developed in our laboratories as a means of stabilizing these species [19]. Specifically, we sought to use tris(amidate) ligand systems with bulky, acyl substituents to favour monomeric aluminium amidates. Herein, we report the synthesis and characterization of two Al(III) tris(κ^2-amidate) complexes stabilized by these ligands.

2. Results and discussion

2.1. Syntheses

The aluminium complex $[Al(N(o\text{-PhNC(O)iPr})_3)]$ is readily obtained by reacting the ligand, $N(o\text{-PhNHC(O)}^i\text{Pr})_3$ with three equivalents of potassium hydride followed by in situ transmetallation with $AlCl_3$ (Scheme 1a). The resulting complex is soluble in a variety of organic solvents including benzene, toluene and chloroform, making removal and quantification of the potassium chloride by product simple. In contrast, the aluminium complex of the ^{t}Bu ligand derivative, $[Al(N(o\text{-PhNC(O)}^t\text{Bu})_3)]$, is prepared by reacting the ligand directly with one equivalent of $AlMe_3$ in toluene (Scheme 1b). Attempts to synthesize $[Al(N(o\text{-PhNC(O)}^i\text{Pr})_3)]$ directly from the reaction of the protio ligand with $AlMe_3$ were unsuccessful and lead to a complex reaction mixture, presumably due to the insolubility of the ligand in toluene. Attempts were made to synthesize and isolate the aluminium complex of the methyl ligand congener, $[Al(N(o\text{-PhNC(O)Me})_3)]$, using analogous

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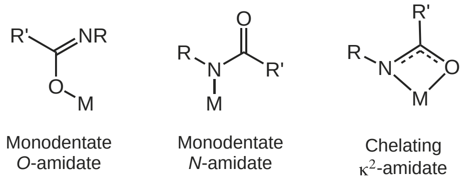
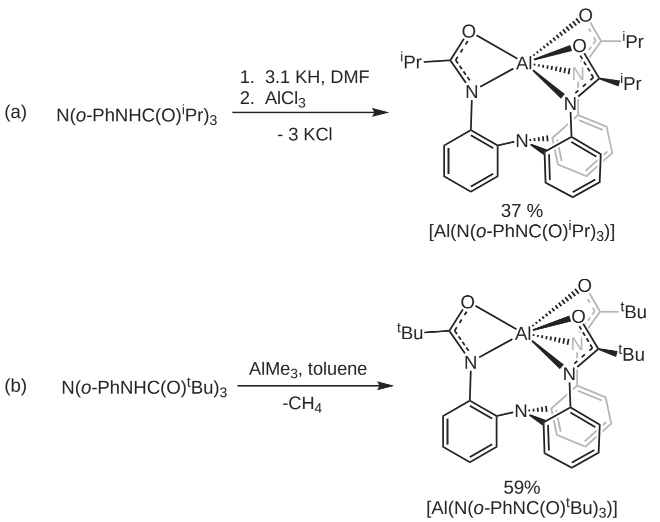


Fig. 1. Coordination modes possible for an amidate ligand ([RNC(O)R′]) interacting with a single metal centre.



Scheme 1. Synthesis of (a) [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and (b) [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)].

synthetic strategies. These reactions, however, yielded complicated product mixtures that contained a number of inseparable aluminium species.

2.2. Spectroscopy

The ^{1}H and ^{13}C spectra of [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] in deuterated chloroform are indicative of pseudo C_3-symmetric species in solution. The ^{13}C NMR spectra of both complexes exhibit single carbonyl resonances at approximately 185 ppm consistent with chelating amidate donors [2,15]. Both [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] exhibit single peaks in their ^{27}Al NMR spectra ($CDCl_3$, 25 °C) at 26 and 27 ppm, respectively. These data are consistent with non-fluxional, octahedral Al(III) centres with mixed donor ligands [20,21].

Infrared spectroscopy can also be used to determine the coordination mode of the amidate ligands. Specifically, the absence of amide ν(N–H) bands at ca. 3260 cm^{-1} and the appearance of new medium to strong intensity ν(Al–O) and ν(Al–N) bands between ~620–740 cm^{-1} are indicative of κ^2-coordination. The diagnostic ν(CO) bands of the free ligands (~1650 cm^{-1}) are also shifted to significantly lower energies (~1580 cm^{-1}) upon coordination to the aluminium centres. The ν(CO) band observed for the κ^2-amidate coordination mode is also significantly lower in energy than the ν(CO) frequency observed in four-coordinate transition metal complexes of the same ligands (i.e., [Co(N(*o*-PhNC(O)^{i}Pr)$_3$)], 1610 cm^{-1}) in which the ligand coordinates through three monodentate, *N*-amidate donors and the tertiary amine of the ligand backbone [19].

2.3. X-ray crystallography

The molecular structures of [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] were determined by X-ray diffraction studies and are shown in Fig. 2A and B, respectively. These studies confirm the chelating tris(κ^2-amidate) coordination mode of each ligand in the solid-state. Selected bond lengths and angles for both complexes are listed in Table 1.

The coordination geometries about the aluminium centres are intermediate between octahedral and trigonal prismatic. The degree of distortion between these two idealized geometries can be

Table 1
Selected average bond lengths (Å), angles (°), and metrical parameters for [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)].

	[Al(N(*o*-PhNC(O)^{i}Pr)$_3$)]	[Al(N(*o*-PhNC(O)^{t}Bu)$_3$)]
Al–O$_{(av)}$	1.927(1)	1.895(3)
Al–N$_{(av)}$	1.967(1)	1.980(4)
C–O$_{(av)}$	1.300(2)	1.301(5)
C–N$_{(av)}$	1.306(2)	1.302(5)
O–Al–N$_{(av)}$	67.07	67.10
Al···N$_{tert}$[a]	3.14	3.15
N···O$_{(av)}$[b]	2.15	2.15
ϕ(O/N)[c]	35.7	36.3
$b_{(av)}$[d]	1.10	1.10

[a] Through space distance between Al(III) centre and non-coordinated, tertiary amine of the ligand.

[b] Bite distance between the chelating *O*- and *N*-donors.

[c] Ligand twist angle.

[d] Normalized bite of the ligand.

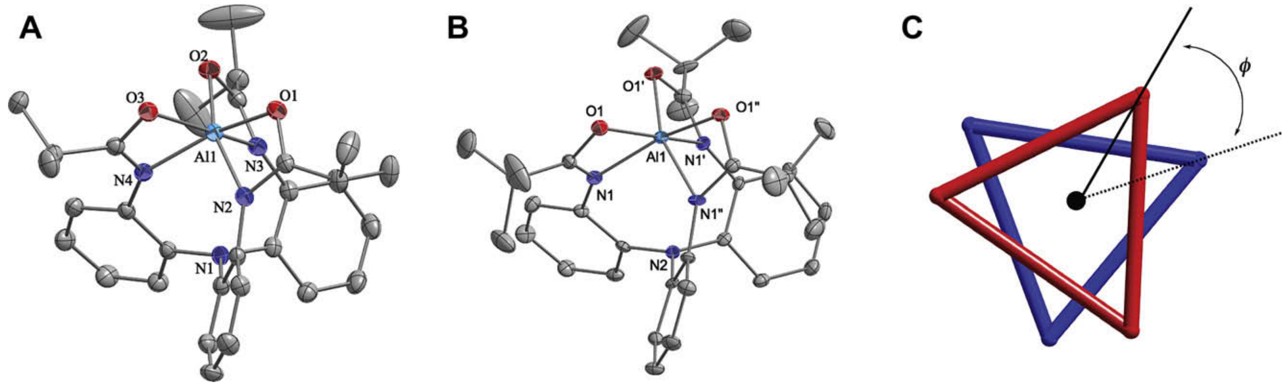


Fig. 2. Thermal ellipsoid diagrams of (A) [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and (B) [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] (one of two independent molecules in the asymmetric unit) drawn at 35% probability. Hydrogen atoms have been omitted for clarity. (C) Illustration of the dihedral angle (trigonal twist angle ϕ) between the trigonal faces formed by the *O*-amidate (red) and N-amidate (blue) donors.

quantified by measuring the dihedral or twist angle (ϕ) between the two trigonal coordination planes [22–25]. A twist angle of 60° is indicative of ideal octahedral coordination, whereas a twist angle of 0° indicates an ideal trigonal prismatic geometry. The average trigonal twist angles for [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] are 35.7° and 36.3°, respectively, indicating a significant degree of distortion away from octahedral geometry. This distortion can be explained by considering the relatively small chelate bite angles (O–Al–N) and bite distances (non-bonded, N···O distance) afforded by the chelating κ^2-amidate ligands. These parameters can be described by the normalized bite, *b*, of the bidentate, chelating ligands ($b = 2 \sin(\alpha/2)$, where α = the O–Al–N bond angle) [26]. Both [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] give rise to normalized bite values of 1.10. This value is similar to normalized bite values (1.12–1.23) observed for tris(*N*,*N*-disubsubstitued-dithiocarbamato) metal complexes with similar, four-membered chelate rings [27]. Complexes exhibiting *b* values of this magnitude are expected to significantly distort away from an idealized octahedral geometry in order to reduce electrostatic repulsion between the donor atoms [26,27].

The primary coordination spheres of [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] and [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] are very similar (Table 1). The Al–O$_{\text{(amidate)}}$ bond lengths of [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] (1.927 and 1.895 Å, respectively) are significantly longer than the Al–O$_{\text{(amidate)}}$ bond length (1.77 Å) observed in the previously reported mononuclear aluminium complex [Me$_2$Al-(PhNC(O)PH)(ONMe$_3$)] that contains an *O*-amidate donor and has been characterized by X-ray diffraction [11]. The delocalization of anionic charge within the chelating κ^2-amidate OCN group is apparent from the similarities in C–O and C–N bond lengths (Table 1).

In the structure of [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)], each amidate acyl substituent is oriented so that either a methyl group or a methine proton of the isopropyl group is positioned between adjacent phenyl rings of the ligand backbone (Fig. 2A). Replacing the isopropyl substituents with larger *tert*-butyl substituents does not result in a significant change in solid-state structure, suggesting that subtle changes in the amidate substituents do not significantly alter the coordination geometries of these species. However, attempts to synthesize the analogous [Al(N(*o*-PhNC(O)Me)$_3$)] complex using similar synthetic strategies were unsuccessful and gave rise only to complex reaction mixtures by spectroscopic analysis. These results indicate that the bulky acyl substituents may be required to prevent the formation of multinuclear aluminium species. Similar observations have been reported for tris(amido) aluminium complexes in which small amido substituents give rise to dimeric aluminium species in solution [28,29].

The six coordinate geometries observed for [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] were not unexpected, as hard metal-ions tend to favour either the κ^2-amidate binding mode or a binding mode in which the *O*-amidate donor spans two different metal centres [1]. However, given the large number of well-characterized four-coordinate azaalumatranes supported by tripodal tetradentate, tris(amido) [28] ligand systems that contain bulky amide substituents i.e., [Al(N(CH$_2$CH$_2$NSiMe$_3$)$_3$], four-coordinate geometries and/or fluxional solution-state behaviour in these systems could not be ruled out.

3. Conclusions

In conclusion, the aluminium complexes [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)] have been prepared and completely characterized. The structures of both complexes have been determined by X-ray diffraction and are very similar. Each complex is six-coordinate and the primary coordination sphere about each aluminium centre consists of three chelating, κ^2-amidate ligands. The small bite angle and bite distance afforded by the amidate ligands give rise to overall coordination geometries that are best described as intermediate between octahedral and trigonal prismatic. Spectroscopic studies indicate that the six-coordinate geometry is maintained in solution.

4. Experimental

4.1. General considerations

All manipulations were conducted in an MBraun Labmaster 130 drybox under a nitrogen atmosphere. All reagents used were purchased from commercial vendors and used as received unless otherwise noted. Anhydrous solvents were purchased from Sigma–Aldrich and further purified by sparging with Ar gas followed by passage through activated alumina columns. Deuterated chloroform (CDCl$_3$-d_1) was purchased from Aldrich and degassed and dried according to standard procedures prior to use. Elemental analyses were performed by Atlantic Microlabs, Norcross, GA. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Unity Plus 600 MHz spectrometer at ambient temperature. Chemical shifts were referenced to residual solvent peaks. The ^{27}Al NMR spectrum was recorded in CDCl$_3$-d_1 on a Varian Unity Plus 600 MHz spectrometer (156.2 MHz) and referenced to an external standard (0.2 M Al(NO$_3$)$_3$ in D$_2$O). The ^{27}Al background signal [30] was determined using a CDCl$_3$ blank. The background signal appeared as a very broad singlet centred at 57 ppm. Infrared spectra were recorded as KBr pellets on a Varian Scimitar 800 Series FT-IR spectrophotometer. X-ray diffraction studies were carried out in the X-ray Crystallography Centre at Emory University on a Bruker Smart 1000 CCD diffractometer. The Mass Spectrometry Centre at Emory University recorded Mass spectra on a JEOL JMS-SX102/SX102A mass spectrometer. The synthesis and characterization of the ligand, [N(*o*-PhNHC(O)^{i}Pr)$_3$)], [19] and the ligand precursor, N(*o*-PhNH$_2$)$_3$, have been previously reported [19,31].

4.2. Synthesis of 2,2′2′′-tris(pivalamidotriphenyl)amine

A suspension of N(*o*-PhNH$_2$)$_3$ (1.79 g, 6.2 mmol) in dichloromethane (CH$_2$Cl$_2$, 80 mL) was cooled to 0 °C under an atmosphere of N$_2$. Distilled triethylamine (3.20 mL, 23.0 mmol) was then added, followed by pivaloyl chloride (2.71 mL, 22.0 mmol). The mixture stirred for 20 h as it slowly warmed to room temperature. The crude reaction mixture was washed with aqueous HCl (0.1 M, 3 × 50 mL), dried over magnesium sulfate, and concentrated in vacuo. The resulting solid was washed with diethyl ether (~10 mL) and ethyl acetate (~10 mL), and the white solid was collected by filtration (69%, 2.29 g). ^{1}H NMR (δ, CDCl$_3$, 300 MHz): 7.95 (s, 3H, NH), 7.79 (dd, 3H, *J* = 1.2, 7.8 Hz, ArH), 7.11 (td, 3H, *J* = 1.2, 7.5 Hz, ArH), 7.02 (td, 3H, *J* = 1.2 Hz, *J* = 7.5 Hz, ArH), 6.84 (dd, 3H, *J* = 1.2, 7.8 Hz, ArH), 0.95 (s, 27H, C(CH$_3$)$_3$). ^{13}C NMR (δ, CDCl$_3$, 300 MHz): 176.64, 137.94, 131.56, 126.01, 125.67, 124.78, 124.42, 39.44, 27.22. HRESI-MS: C$_{33}$H$_{43}$O$_3$N$_4$ *m/z* Calc. 543.33297; found: 543.33352 [M + 1]$^{+}$. FT-IR (KBr, cm^{-1}): ν(NH) 3258, ν(CO) 1653, 3387, 3309, 2959, 2909, 2871, 1680, 1653, 1594, 1516, 1479, 1439, 1303, 1265, 1160, 928, 756, 734, 625, 480.

4.3. Synthesis of [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)]

To a stirred solution of N(*o*-PhNHC(O)^{i}Pr)$_3$ (98 mg, 0.20 mmol) in DMF (2 mL) was added KH (26 mg, 0.65 mmol) as a solid. When H$_2$ evolution ceased, AlCl$_3$ (26 mg, 0.20 mmol) was added as a solid. Upon complete dissolution of the metal salt, solvent was removed in vacuo. The colorless solid was extracted into toluene

Table 2
Crystal and refinement data for complexes [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)][a].

	[Al(N(*o*-PhNC(O)^{i}Pr)$_3$)]	[Al(N(*o*-PhNC(O)^{t}Bu)$_3$)]
Formula	$C_{30}H_{33}AlN_4O_3$	$C_{33}H_{39}AlN_4O_3$
Formula weight	524.58	566.66
Lattice, space group	orthorhombic, *Pbca*	trigonal, $P3_1c$
a (Å)	15.9429(3)	11.2793(8)
b (Å)	9.8814(2)	11.2793(8)
c (Å)	34.5625(7)	28.563(4)
α (°)	90	90
β (°)	90	90
γ (°)	90	120
Z	8	4
V (Å^3)	5444.91(19)	3147.0(6)
Temperature (K)	173(2)	173(2)
Radiation (λ, Å)	0.71073	0.71073
Reflections measured	36660	38246
Unique reflections [R_{int}]	5585	3881
R_1 ($I > 2\sigma(I)$)	0.0499	0.0713
wR_2, all data	0.1375	0.1777

and filtered to remove KCl (3 equiv.). X-ray quality crystals were obtained by vapour diffusion of hexanes into a concentrated toluene solution. (38 mg, 37%). ^{1}H NMR (δ), $CDCl_3$, 600 MHz: 7.45 (dd, 3H, *J* = 7.8, 1.2 Hz, ArH), 7.09 (dt, 3H, *J* = 7.8, 1.2 Hz, ArH), 7.04 (dt, 3H, *J* = 7.8, 1.8 Hz, ArH), 6.95 (dd, 3H, *J* = 7.8, 1.8 Hz, ArH), 2.94 (7, 3H, *J* = 7.2 Hz, CH), 1.28 (d, 9H, *J* = 7.2 Hz, CH_3), 0.55 (d, 9H, *J* = 6.6 Hz, CH_3). ^{13}C NMR (δ (ppm), $CDCl_3$, 600 MHz): 184.84, 141.22, 140.48, 130.67, 126.29, 124.25, 122.25, 28.32, 19.14, 17.97. ^{27}Al NMR (δ, $CDCl_3$, 600 MHz): 26 ppm ($W_{1/2}$ = 3000 Hz). FT-IR (KBr, cm^{-1}): ν(CO) 1580, 1474, 1435, 1373, 1295, 1271, 1079, 976, 751, 507. *Anal.* Calc. for [Al^{III}(N(*o*-PhNC(O)^{i}Pr)$_3$)]: C, 68.69; H, 6.34; N, 10.68. Found: C, 68.35; H, 6.41; N, 10.57%.

4.4. Synthesis of [Al(N(o-PhNC(O)^{t}Bu)$_3$)]

To a stirred suspension of N(*o*-PhNHC(O)^{t}Bu)$_3$ (250 mg, 0.46 mmol) in toluene (4 mL) was added $AlMe_3$ via syringe (2.0 M in toluene, 0.23 mL, 0.46 mmol). After 20 min of stirring, the reaction mixture became homogeneous and solvent was removed in vacuo; leaving a colorless solid. X-ray quality crystals were obtained by vapour diffusion of petroleum ether into a concentrated toluene solution of the complex (154 mg, 59%). ^{1}H NMR (δ), $CDCl_3$, 600 MHz: 7.24 (dd, 3H, *J* = 7.8, 1.2 Hz, ArH), 7.01 (m, 9H, ArH), 0.917 (s, 27H, CH_3C). ^{13}C NMR (δ), $CDCl_3$, 600 MHz: 184.706, 141.409, 140.62, 129.45, 125.17, 125.00, 124.21, 38.51, 28.15. ^{27}Al NMR (δ (ppm), $CDCl_3$, 600 MHz): 27 ($W_{1/2}$ = 3000 Hz). FT-IR (KBr, cm^{-1}): ν(CO) 1562, 1480, 1363, 1302, 1190, 968, 760, 749, 623, 529. *Anal.* Calc. for [AlN(*o*-PhNC(O)^{t}Bu)$_3$]: C, 69.94; H, 6.94; N, 9.89. Found: C, 69.97; H, 6.84; N, 9.80%.

4.5. X-ray structure determinations

Suitable crystals of [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)]and[Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] were coated with Paratone N oil, suspended in small fiber loops and placed in a cooled nitrogen gas stream at 173 K on a Bruker D8 APEX II CCD sealed tube diffractometer with graphite monochromated Mo Kα (0.71073 Å) radiation. Data were measured using a series of combinations of phi and omega scans with 10 s frame exposures and 0.5° frame widths.

The structures were solved using Direct methods and difference Fourier techniques (SHELXTL, V6.12) [32]. Hydrogen atoms were placed their expected chemical positions using the HFIX command and were included in the final cycles of least squares with isotropic U_{ij}'s related to the atom's ridden upon. All non-hydrogen atoms were refined anisotropically. Structure solution, refinement, graphics and generation of publication materials were performed by using SHELXTL, V6.12 software. Additional details of data collection and structure refinement are given in Table 2.

Supplementary data

CCDC 703388 and 729894 contains the supplementary crystallographic data for [Al(N(*o*-PhNC(O)^{i}Pr)$_3$)] and [Al(N(*o*-PhNC(O)^{t}Bu)$_3$)]. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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