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Bis[μ-*N*-(2,6-dimethylphenyl)acetamidato]-bis(dimethylaluminium)

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The structure of the title compound, $[Al_2(CH_3)_4(C_{10}H_{12}NO)_2]$ or $[Me_2Al\{\mu\text{-}(2,6\text{-}Me_2C_6H_3)NCMeO\}]_2$, consists of a four-coordinate dimeric centrosymmetric eight-membered ring Al-containing species.

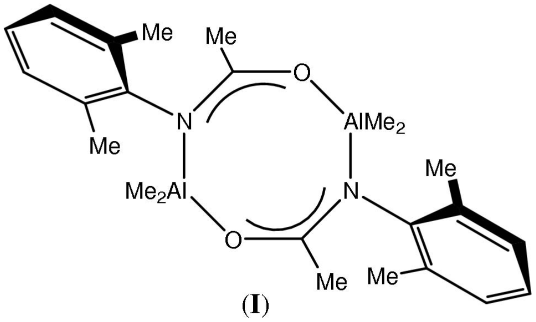
Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.006 Å
R factor = 0.064
wR factor = 0.190
Data-to-parameter ratio = 16.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The synthesis and structural characterization of alkylaluminium complexes containing *N*,*O*-amidate ligands, $[R^{I}NCR(=O)]^-$ (*R* = alkyl or aryl), has recently received attention due, in part, to the rich variety of bonding modes that are accessible. For example, the ligand can bridge, chelate or act as a monodentate ligand to a single metal centre, the precise bonding mode being dependent on the acidity and the steric bulk of the amide precursor employed (Huang *et al.*, 2002). Furthermore, the reactions of amides with R_3Al have allowed access to aluminium diketimates, some of which are not obtainable by more conventional synthetic routes (Huang *et al.*, 2001).



(I)

We report here the synthesis and the crystal structure of $[Me_2Al\{\mu\text{-}(2,6\text{-}Me_2C_6H_3)NCMeO\}]_2$, (I). The ^{1}H NMR spectrum gives methyl resonances in a ratio of 12:6:12, corresponding to the aromatic (δ 2.40), acetamide (δ 1.70) and aluminium methyls (δ −0.15), respectively. The X-ray analysis of (I) reveals a dimeric structure based on a centrosymmetric eight-membered ring. The bridging amidate ligand coordinates to the two Al atoms through both the N and the O atoms. The geometry at each Al atom can be best described as distorted tetrahedral, with two methyl C atoms, an N atom and an O atom occupying the coordination sites. The C3—O1 [1.296 (4) Å] and C3—N1*A* [1.298 (4) Å] bond lengths suggest some delocalization within the OCN moiety. The Al1—O1 [1.800 (3) Å], Al1—N1 [1.961 (3) Å], Al1—C1 [1.968 (5) Å] and Al1—C2 [1.961 (4) Å] bond distances in (I) are comparable with the corresponding distances observed in the related structures $[Me_2Al\{\mu\text{-}(C_6H_5)NCPhO\}]_2$ (Kai *et al.*, 1971) and

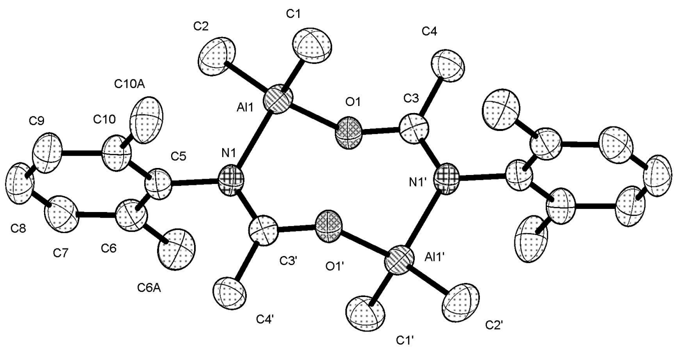


Figure 1
Molecular structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids. The molecule is located on a centre of symmetry [primed atoms are generated by $(1 - x, 2 - y, 2 - z)$]. H atoms have been omitted for clarity.

$[Me_2Al\{\mu\text{-}(2,6\text{-}Pr_2{}^iC_6H_3)NCPhO\}]_2$ (Huang *et al.*, 2002). The benzene rings are arranged orthogonal to the puckered eight-membered ring. There are no intermolecular packing interactions of note.

Experimental

Under an atmosphere of nitrogen, trimethylaluminium (3.07 ml, 6.13 mmol, 2*M* solution in toluene) was added to a solution of *N*-(2,6-dimethylphenyl)acetamide (0.50 g, 3.06 mmol) in toluene (30 ml), and the reaction mixture was heated to reflux for 12 h. On cooling to room temperature, the volatiles were removed under reduced pressure and the residue dried overnight. Slow cooling of a hot acetonitrile (40 ml) solution containing the complex gave pale-yellow crystals of the title compound suitable for single-crystal X-ray diffraction analysis (yield 0.50 g, 75%). Analysis found: C 65.89, H 8.31, N 6.57%; calculated for $C_{24}H_{36}Al_2N_2O_2$: C 65.75, H 8.22, N 6.39%. ^{1}H NMR (C_6D_6): δ 7.20–7.05 (*m*, 6H, Ar—H), 2.40 (*s*, 12H, Ar—Me), 1.70 [*s*, 6H, MeC(O)] and −0.15 (*s*, 12H, Al—CH_3).

Crystal data

$[Al_2(CH_3)_4(C_{10}H_{12}NO)_2]$
$M_r = 438.51$
Monoclinic, $P2_1/n$
$a = 11.028$ (2) Å
$b = 10.4955$ (9) Å
$c = 11.116$ (5) Å
$\beta = 90.37$ (3)°
$V = 1286.6$ (6) Å^3
$Z = 2$

$D_x = 1.132$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 27 reflections
$\theta = 4.7\text{–}12.5°$
$\mu = 0.13$ mm^{-1}
$T = 200$ (2) K
Block, pale yellow
0.53 × 0.41 × 0.41 mm

Data collection

Bruker *P*4 diffractometer
ω scans
Absorption correction: none
2650 measured reflections
2257 independent reflections
1598 reflections with $I > 2s(I)$
$R_{\text{int}} = 0.029$

$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 13$
$k = -1 \rightarrow 12$
$l = -13 \rightarrow 13$
2 standard reflections every 1000 reflections
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.190$
$S = 1.07$
2257 reflections
137 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 3.0349P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.54$ e Å^{-3}
$\Delta\rho_{\min} = -0.54$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.023 (4)

Table 1
Selected geometric parameters (Å, °).

Al1—O1	1.800 (3)	Al1—C2	1.961 (4)
Al1—N1	1.961 (3)	Al1—C1	1.968 (5)
O1—Al1—N1	103.37 (13)	O1—Al1—C1	109.21 (16)
O1—Al1—C2	107.46 (17)	N1—Al1—C1	110.47 (17)
N1—Al1—C2	105.81 (18)	C2—Al1—C1	119.3 (2)

All H atoms were included in calculated positions and treated as riding on the bonded atom (C—H = 0.93 and 0.96 Å). U_{iso}(H) was set to $1.5U_{\text{eq}}$(C) for methyl H atoms and $1.2U_{\text{eq}}$(C) for all other H atoms.

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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