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Key indicators

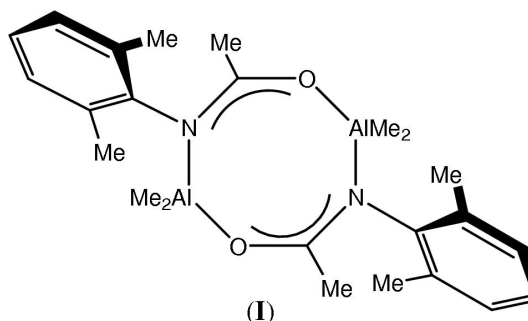
Single-crystal X-ray study
 $T = 200$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.064
 wR factor = 0.190
Data-to-parameter ratio = 16.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[μ - N -(2,6-dimethylphenyl)acetamidato]-
bis(dimethylaluminium)The structure of the title compound, $[\text{Al}_2(\text{CH}_3)_4(\text{C}_{10}\text{H}_{12}\text{NO})_2]$ or $[\text{Me}_2\text{Al}\{\mu\text{-(2,6-Me}_2\text{C}_6\text{H}_3)\text{NCMeO}\}]_2$, consists of a four-coordinate dimeric centrosymmetric eight-membered ring Al-containing species.

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Comment

The synthesis and structural characterization of alkyl-aluminium complexes containing N,O -amidate ligands, $[\text{R}^1\text{NCR}(\text{=O})]^-$ ($\text{R} =$ 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).

We report here the synthesis and the crystal structure of $[\text{Me}_2\text{Al}\{\mu\text{-(2,6-Me}_2\text{C}_6\text{H}_3)\text{NCMeO}\}]_2$, (I). The ^1H 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—N1A [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 $[\text{Me}_2\text{Al}\{\mu\text{-(C}_6\text{H}_5)\text{NCPHO}\}]_2$ (Kai *et al.*, 1971) and

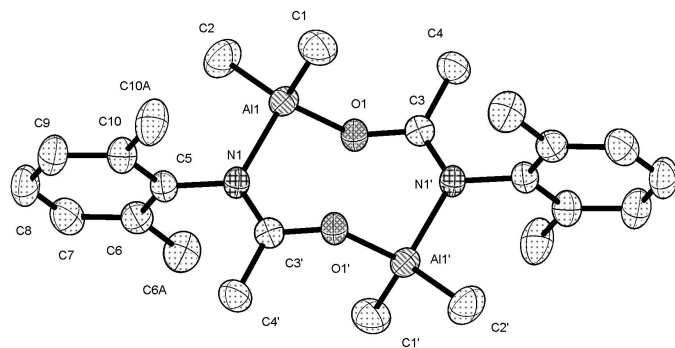


Figure 1
Molecular structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids. The molecule is located on a centre of symmetry [primed atoms are generated by $(1 - x, 2 - y, 2 - z)$]. H atoms have been omitted for clarity.

$[\text{Me}_2\text{Al}\{\mu\text{-(2,6-Pr}_2^i\text{C}_6\text{H}_3\text{)NCPHO}\}]_2$ (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, 2M 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 $\text{C}_{24}\text{H}_{36}\text{Al}_2\text{N}_2\text{O}_2$: C 65.75, H 8.22, N 6.39%. $^1\text{H NMR}$ (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–CH₃).

Crystal data

$[\text{Al}_2(\text{CH}_3)_4(\text{C}_{10}\text{H}_{12}\text{NO})_2]$
 $M_r = 438.51$
Monoclinic, $P2_1/n$
 $a = 11.028$ (2) Å
 $b = 10.4955$ (9) Å
 $c = 11.116$ (5) Å
 $\beta = 90.37$ (3) $^\circ$
 $V = 1286.6$ (6) Å³
 $Z = 2$

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

Data collection

Bruker P4 diffractometer
 ω scans
Absorption correction: none
2650 measured reflections
2257 independent reflections
1598 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

$\theta_{\text{max}} = 25.0^\circ$
 $h = 0 \rightarrow 13$
 $k = -1 \rightarrow 12$
 $l = -13 \rightarrow 13$
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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.54$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.54$ e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.023 (4)

Table 1

Selected geometric parameters (Å, $^\circ$).

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). $U_{\text{iso}}(\text{H})$ was set to $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2U_{\text{eq}}(\text{C})$ 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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References

- Fait, J. (1991). XSCANS. Bruker AXS Inc., Madison, Wisconsin, USA.
Huang, B.-H., Yu, T.-C., Huang, Y.-L., Ko, B.-T. & Lin, C.-C. (2002). *Inorg. Chem.* **41**, 2987–2994.
Huang, Y.-C., Huang, B.-H., Ko, B.-T. & Lin, C.-C. (2001). *Dalton Trans.* pp. 1359–1365.
Kai, Y., Yasuoka, N. & Kakudo, M. (1971). *J. Organomet. Chem.* **32**, 165–179.
Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
Sheldrick, G. M. (2000). SHELXTL. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2005). E61, m1210–m1211 [https://doi.org/10.1107/S1600536805015941]

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

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

Crystal data

[Al₂(CH₃)₄(C₁₀H₁₂NO)₂]

M_r = 438.51

Monoclinic, *P*2₁/*n*

Hall symbol: -*P* 2₁*y**n*

a = 11.028 (2) Å

b = 10.4955 (9) Å

c = 11.116 (5) Å

β = 90.37 (3)°

V = 1286.6 (6) Å³

Z = 2

F(000) = 472

D_x = 1.132 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 27 reflections

θ = 4.7–12.5°

μ = 0.13 mm⁻¹

T = 200 K

Block, pale yellow

0.53 × 0.41 × 0.41 mm

Data collection

Bruker P4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

2650 measured reflections

2257 independent reflections

1598 reflections with *I* > 2*s*(*I*)

*R*_{int} = 0.029

θ_{\max} = 25.0°, θ_{\min} = 2.6°

h = 0 → 13

k = -1 → 12

l = -13 → 13

2 standard reflections every 1000 reflections

intensity decay: <1%

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2 σ (*F*²)] = 0.064

wR(*F*²) = 0.190

S = 1.07

2257 reflections

137 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

w = 1/[$\sigma^2(F_o^2) + (0.065P)^2 + 3.0349P$]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

$\Delta\rho_{\max}$ = 0.54 e Å⁻³

$\Delta\rho_{\min}$ = -0.54 e Å⁻³

Extinction correction: SHELXL97,

*F_c** = *kF_c*[1 + 0.001*xF_c*² $\lambda^3/\sin(2\theta)$]^{-1/4}

Extinction coefficient: 0.023 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
All	0.56700 (10)	1.01577 (11)	0.83404 (10)	0.0346 (4)
O1	0.6307 (2)	1.0135 (2)	0.9835 (2)	0.0328 (6)
N1	0.4303 (3)	0.8969 (3)	0.8447 (2)	0.0305 (7)
C1	0.5088 (4)	1.1888 (4)	0.7972 (4)	0.0541 (12)
H1A	0.4511	1.2149	0.8567	0.081*
H1B	0.4708	1.1891	0.7193	0.081*
H1C	0.5761	1.2467	0.7974	0.081*
C2	0.6846 (4)	0.9348 (5)	0.7268 (4)	0.0571 (12)
H2A	0.7042	0.8514	0.7566	0.086*
H2B	0.7569	0.9856	0.7237	0.086*
H2C	0.6502	0.9277	0.6475	0.086*
C3	0.6447 (3)	1.0996 (3)	1.0659 (3)	0.0295 (8)
C4	0.7501 (4)	1.1884 (4)	1.0544 (4)	0.0422 (10)
H4A	0.7497	1.2485	1.1195	0.063*
H4B	0.7443	1.2331	0.9793	0.063*
H4C	0.8241	1.1402	1.0568	0.063*
C5	0.4160 (3)	0.8022 (3)	0.7517 (3)	0.0307 (8)
C6	0.4677 (3)	0.6807 (4)	0.7681 (3)	0.0371 (9)
C7	0.4593 (4)	0.5944 (4)	0.6738 (4)	0.0465 (11)
H7A	0.4930	0.5137	0.6825	0.056*
C8	0.4015 (4)	0.6264 (4)	0.5674 (4)	0.0501 (11)
H8A	0.3963	0.5673	0.5053	0.060*
C9	0.3515 (4)	0.7460 (4)	0.5530 (4)	0.0478 (11)
H9A	0.3137	0.7669	0.4806	0.057*
C10	0.3567 (3)	0.8355 (4)	0.6445 (3)	0.0381 (9)
C6A	0.5284 (4)	0.6454 (4)	0.8838 (4)	0.0518 (11)
H6D	0.5565	0.5589	0.8799	0.078*
H6E	0.4724	0.6541	0.9490	0.078*
H6F	0.5962	0.7013	0.8968	0.078*
C10A	0.3007 (5)	0.9660 (4)	0.6288 (4)	0.0565 (12)
H10D	0.2638	0.9720	0.5506	0.085*
H10E	0.3625	1.0300	0.6367	0.085*
H10F	0.2402	0.9789	0.6893	0.085*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
All	0.0355 (7)	0.0404 (7)	0.0280 (6)	-0.0045 (5)	0.0018 (4)	-0.0007 (5)
O1	0.0343 (14)	0.0327 (13)	0.0314 (13)	-0.0004 (11)	-0.0006 (10)	-0.0060 (11)
N1	0.0320 (16)	0.0324 (16)	0.0270 (14)	-0.0010 (13)	-0.0022 (12)	-0.0049 (12)
C1	0.063 (3)	0.048 (3)	0.051 (3)	-0.008 (2)	-0.011 (2)	0.011 (2)
C2	0.049 (3)	0.078 (3)	0.045 (2)	-0.003 (2)	0.014 (2)	-0.009 (2)
C3	0.0275 (18)	0.0294 (18)	0.0316 (18)	0.0031 (15)	-0.0027 (14)	0.0008 (15)
C4	0.038 (2)	0.039 (2)	0.049 (2)	-0.0064 (18)	0.0086 (18)	-0.0060 (18)
C5	0.0284 (19)	0.0312 (18)	0.0325 (18)	-0.0006 (15)	0.0005 (14)	-0.0055 (15)
C6	0.033 (2)	0.038 (2)	0.040 (2)	-0.0002 (16)	0.0039 (16)	-0.0028 (17)
C7	0.043 (2)	0.037 (2)	0.060 (3)	0.0024 (18)	0.010 (2)	-0.011 (2)
C8	0.049 (3)	0.054 (3)	0.047 (2)	0.000 (2)	0.006 (2)	-0.023 (2)
C9	0.043 (2)	0.061 (3)	0.039 (2)	-0.001 (2)	-0.0021 (18)	-0.014 (2)
C10	0.036 (2)	0.041 (2)	0.037 (2)	0.0018 (17)	-0.0050 (16)	-0.0058 (17)
C6A	0.057 (3)	0.047 (2)	0.051 (2)	0.015 (2)	-0.003 (2)	0.000 (2)
C10A	0.069 (3)	0.058 (3)	0.042 (2)	0.018 (2)	-0.018 (2)	-0.007 (2)

Geometric parameters (\AA , $^\circ$)

All—O1	1.800 (3)	C5—C10	1.400 (5)
All—N1	1.961 (3)	C5—C6	1.408 (5)
All—C2	1.961 (4)	C6—C7	1.387 (5)
All—C1	1.968 (5)	C6—C6A	1.494 (6)
O1—C3	1.296 (4)	C7—C8	1.382 (6)
N1—C3 ⁱ	1.298 (4)	C7—H7A	0.9300
N1—C5	1.443 (4)	C8—C9	1.379 (6)
C1—H1A	0.9600	C8—H8A	0.9300
C1—H1B	0.9600	C9—C10	1.386 (5)
C1—H1C	0.9600	C9—H9A	0.9300
C2—H2A	0.9600	C10—C10A	1.512 (6)
C2—H2B	0.9600	C6A—H6D	0.9600
C2—H2C	0.9600	C6A—H6E	0.9600
C3—N1 ⁱ	1.298 (4)	C6A—H6F	0.9600
C3—C4	1.496 (5)	C10A—H10D	0.9600
C4—H4A	0.9600	C10A—H10E	0.9600
C4—H4B	0.9600	C10A—H10F	0.9600
C4—H4C	0.9600		
O1—All—N1	103.37 (13)	C10—C5—C6	121.6 (3)
O1—All—C2	107.46 (17)	C10—C5—N1	119.1 (3)
N1—All—C2	105.81 (18)	C6—C5—N1	119.3 (3)
O1—All—C1	109.21 (16)	C7—C6—C5	117.9 (4)
N1—All—C1	110.47 (17)	C7—C6—C6A	121.1 (4)
C2—All—C1	119.3 (2)	C5—C6—C6A	121.0 (3)
C3—O1—All	133.4 (2)	C8—C7—C6	121.1 (4)
C3 ⁱ —N1—C5	120.0 (3)	C8—C7—H7A	119.5

C3 ⁱ —N1—A11	121.5 (2)	C6—C7—H7A	119.5
C5—N1—A11	118.4 (2)	C9—C8—C7	120.2 (4)
A11—C1—H1A	109.7	C9—C8—H8A	119.9
A11—C1—H1B	109.3	C7—C8—H8A	119.9
H1A—C1—H1B	109.5	C8—C9—C10	121.1 (4)
A11—C1—H1C	109.4	C8—C9—H9A	119.4
H1A—C1—H1C	109.5	C10—C9—H9A	119.4
H1B—C1—H1C	109.5	C9—C10—C5	118.2 (4)
A11—C2—H2A	109.5	C9—C10—C10A	121.0 (4)
A11—C2—H2B	109.5	C5—C10—C10A	120.9 (3)
H2A—C2—H2B	109.5	C6—C6A—H6D	109.8
A11—C2—H2C	109.5	C6—C6A—H6E	109.8
H2A—C2—H2C	109.5	H6D—C6A—H6E	109.5
H2B—C2—H2C	109.5	C6—C6A—H6F	108.8
O1—C3—N1 ⁱ	119.1 (3)	H6D—C6A—H6F	109.5
O1—C3—C4	117.6 (3)	H6E—C6A—H6F	109.5
N1 ⁱ —C3—C4	123.2 (3)	C10—C10A—H10D	109.5
C3—C4—H4A	109.7	C10—C10A—H10E	109.5
C3—C4—H4B	109.4	H10D—C10A—H10E	109.5
H4A—C4—H4B	109.5	C10—C10A—H10F	109.4
C3—C4—H4C	109.3	H10D—C10A—H10F	109.5
H4A—C4—H4C	109.5	H10E—C10A—H10F	109.5
H4B—C4—H4C	109.5		
N1—A11—O1—C3	-117.9 (3)	C10—C5—C6—C7	0.6 (5)
C2—A11—O1—C3	130.5 (3)	N1—C5—C6—C7	-175.8 (3)
C1—A11—O1—C3	-0.3 (4)	C10—C5—C6—C6A	-178.7 (4)
O1—A11—N1—C3 ⁱ	45.6 (3)	N1—C5—C6—C6A	4.9 (5)
C2—A11—N1—C3 ⁱ	158.4 (3)	C5—C6—C7—C8	-0.3 (6)
C1—A11—N1—C3 ⁱ	-71.1 (3)	C6A—C6—C7—C8	179.1 (4)
O1—A11—N1—C5	-132.9 (2)	C6—C7—C8—C9	0.3 (6)
C2—A11—N1—C5	-20.1 (3)	C7—C8—C9—C10	-0.7 (7)
C1—A11—N1—C5	110.3 (3)	C8—C9—C10—C5	1.1 (6)
A11—O1—C3—N1 ⁱ	101.3 (4)	C8—C9—C10—C10A	-179.4 (4)
A11—O1—C3—C4	-80.4 (4)	C6—C5—C10—C9	-1.0 (6)
C3 ⁱ —N1—C5—C10	97.9 (4)	N1—C5—C10—C9	175.4 (3)
A11—N1—C5—C10	-83.5 (4)	C6—C5—C10—C10A	179.4 (4)
C3 ⁱ —N1—C5—C6	-85.5 (4)	N1—C5—C10—C10A	-4.1 (6)
A11—N1—C5—C6	93.0 (4)		

Symmetry code: (i) $-x+1, -y+2, -z+2$.