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Bis(μ-*N*-methylethylamido)bis[*tert*-butylchloroaluminium]

Elmar Hecht‡

Universität Leipzig, Institut für Anorganische Chemie, Johannisallee 29, D-04103 Leipzig, Germany

‡ Present address: SusTech Darmstadt GmbH & Co KG, Petersenstraße 20, D-64287 Darmstadt, Germany

Correspondence e-mail:
elmar.hecht@sustech.de

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Key indicators

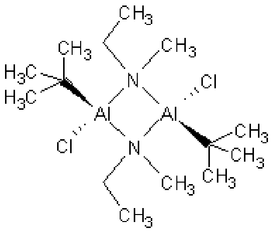
Single-crystal X-ray study
T = 213 K
Mean σ(C–C) = 0.007 Å
R factor = 0.060
wR factor = 0.124
Data-to-parameter ratio = 23.5

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

The dimeric title coordination compound, $[Al_2(C_4H_9)_2Cl_2(C_3H_8N)_2]$, comprises tetrahedral Al atoms each bonded to two N atoms of the amide ligands, one C atom of the *tert*-butyl group and one Cl atom. The Al atoms are bridged by amide moieties, creating a planar four-membered Al_2N_2 ring, which may be considered as the main structural feature. A twofold rotation axis passes through the centre of the ring. The Al—N bond distance are 1.943 (4) and 1.962 (4) Å. Pseudosymmetry in space group $P4_2/nmc$ was considered but the space group $P\overline{4}2_1c$ was confirmed by the structure solution and refinement.

Comment

The chemistry of compounds containing Al—N bonds flourished over the past several years due mainly to current interest in developing optimum AlN precursors. The structures of several complexes of aluminium and gallium with amide ligands have been reported (Chang & Ameerunisha, 1999; Dümichen *et al.*, 1999; Carmalt, 2001). Compounds of that type usually form dimeric molecules comprising a central four-membered Al_2N_2 ring as the main structural feature. The Al atom in the title compound, (I), adopts a tetrahedral geometry, formed by two N atoms of the amide group [Al—N = 1.943 (2) and 1.962 (4) Å], one Cl atom [Al—Cl = 2.145 (1) Å] and one C atom of a *tert*-butyl group [Al—C = 1.983 (3) Å]. As a result of the bridging effect of the amide ligands, dimeric molecules are formed, which have a crystallographic two-fold rotation axis perpendicular to the central ring.



(I)

Experimental

The title compound, (I), was prepared by a method analogous to the procedure of Dümichen *et al.* (1999), by reaction of equimolar amounts (20 mmol) of *tert*-butyldichloroaluminium and lithium *N*-methylethylamide in toluene (50 ml). The reaction mixture was refluxed for 2 h and the solvent removed *in vacuo* to produce a colourless precipitate. The solid formed was collected and dried *in vacuo*. Suitable crystals were obtained by cooling a saturated solution of (I) in *n*-pentane. Analysis calculated for $C_{14}H_{34}Al_2Cl_2N_2$: C 47.30, H 9.57, Al 15.20%; found: C 47.21, H 9.63, Al 15.08%.

Crystal data

$[Al_2(C_4H_9)_2Cl_2(C_3H_8N)_2]$
$M_r = 355.29$
Tetragonal, $P\overline{4}2_1c$
$a = 12.0110\ (1)$ Å
$c = 14.4768\ (3)$ Å
$V = 2088.48\ (5)$ Å^3
$Z = 4$
$D_x = 1.130$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 8923 reflections
$\theta = 2$–$25°$
$\mu = 0.39$ mm^{-1}
$T = 213\ (2)$ K
Block, colourless
$0.40 \times 0.30 \times 0.20$ mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.860$, $T_{\max} = 0.926$
8923 measured reflections

2162 independent reflections
1693 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.094$
$\theta_{\max} = 27.2°$
$h = -6 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.124$
$S = 1.17$
2162 reflections
92 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 1.7272P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}
Absolute structure: Flack (1983), 832 Friedel reflections
Flack parameter = 0.5 (2)

Table 1
Selected geometric parameters (Å, °).

Al1—N1^{i}	1.943 (4)	Al1···Al1^{i}	2.7782 (18)
Al1—N1	1.962 (4)	N1—C5	1.500 (4)
Al1—C1	1.983 (3)	N1—C6	1.502 (4)
Al1—Cl1	2.1455 (12)		
N1^{i}—Al1—N1	87.56 (12)	C5—N1—Al1^{i}	111.3 (3)
N1^{i}—Al1—C1	122.36 (18)	C6—N1—Al1^{i}	119.4 (3)
N1—Al1—C1	119.58 (19)	C5—N1—Al1	111.2 (3)
N1^{i}—Al1—Cl1	107.28 (10)	C6—N1—Al1	114.3 (3)
N1—Al1—Cl1	107.48 (10)	Al1^{i}—N1—Al1	90.71 (11)
C1—Al1—Cl1	110.08 (11)		

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

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.97 and 0.98 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. There is pseudosymmetry which emulates space group $P4_2/nmc$. Program *XPREP* of the *SHELXTL* package [Sheldrick (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA] only suggested $P\overline{4}2_1c$ because of the reflection conditions. *PLATON* [Spek (2003). *J. Appl. Cryst.* **36**, 7–13], however, suggested the higher symmetry space group. The correct space group ($P\overline{4}2_1c$) was assigned by examination of the reflection conditions. The Flack (1983) parameter, with a high standard uncertainty, indicates probable inversion twinning.

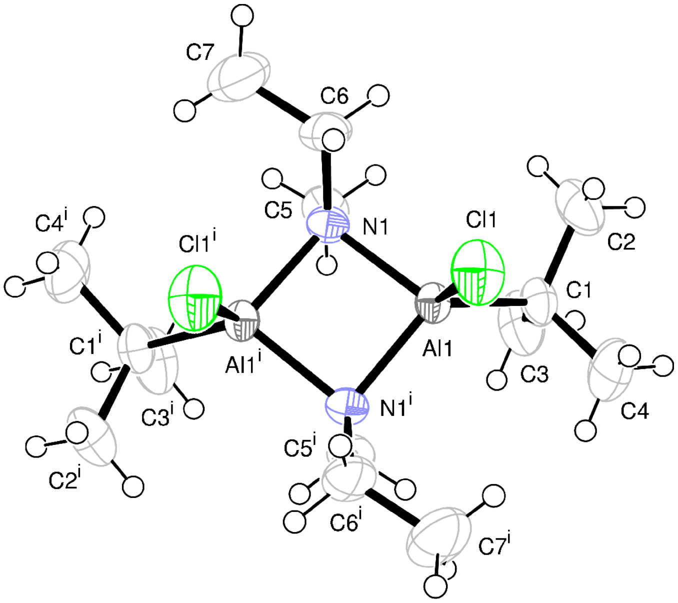


Figure 1
View of the dimeric molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Symmetry operator for generating equivalent atoms: (i) $1 - x, -y, z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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