Acta Crystallographica Section C
**Crystal Structure Communications**
ISSN 0108-2701

# Bis[($\mu$-cyclopentylamino-*N*:*N*)-dimethylaluminium(III)]

**Fiona J. Craig, Alan R. Kennedy* and Robert E. Mulvey**

Department of Pure and Applied Chemistry, University of Strathclyde, 295 Cathedral Street, Glasgow G1 1XL, Scotland
Correspondence e-mail: a.r.kennedy@ccsun.strath.ac.uk

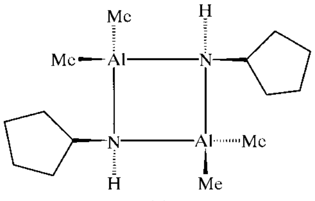
Received 2 September 1999
Accepted 4 October 1999

Reaction of $AlMe_3$ with $NH_2(C_5H_9)$ caused the evolution of methane and produced the dimeric species bis($\mu$-cyclopentylamino-*N*:*N*)bis[dimethylaluminium(III)], $[Al(CH_3)_2(C_5H_{10}N)]_2$, which was found to adopt a *cis* configuration of cyclopentyl groups about a bent AlNAlN ring (which has twofold crystallographic symmetry) instead of the more common *trans* arrangement.

## Comment

Diorganoaluminium amides [$R_2$AlN(H)$R'$], where the amide function derives from a primary source $R'NH_2$, have proved useful synthetically in providing access to a range of more exotic organoaluminium compounds and structures. This chemistry centres on the reactivity of the remaining N—H bond, and the products obtained are markedly dependent on the nature of the $R'$ substituent (Waggoner & Power, 1991). The structures of these primary amides have also attracted considerable attention in their own right. It has been established that in amide-bridged dimeric systems larger organic *R* ligands bound to Al favour a configuration with mutually *cis*, rather than mutually *trans*, $R'$ groups (Schaur *et al.*, 1992). Thus, when *R* is methyl a *trans* arrangement is favoured and indeed only three *cis* examples have been documented. A mixture of both configurations cocrystallizing in a 2:1 *trans*:*cis* ratio was found for $R'$ = isopropyl (Amirkhalili *et al.*, 1981) and the *cis* isomer is also known from two cases where $R'$ is chiral, –CHMePh (Robinson *et al.*, 1988) or –CHMe(naphthyl) (Pennington *et al.*, 1990). It has been suggested that in the latter cases the *cis* configuration is adopted as the 2/*m* geometry shown by the *trans* isomer is not accessible for optically active compounds whilst the *cis* configuration still allows the largest substituents on $R'$ to be mutually *trans.* It was reported that no interconversion of the isomers was found in solution. It is hence of interest here that the crystal structure of the cycloalkylamide [$Me_2Al\{\mu$-N(H)$R'\}_2AlMe_2$], (I), where $R'$ is a simple cyclopentyl group, shows it to adopt only the *cis* configuration.

The two halves of dimeric (I) are related by a twofold rotation axis. The AlNAlN ring is bent about the Al···Al vector (Al1—*Cp*—Al1* 171.5°, where *Cp* is the ring centroid) as are the three other known *cis* isomers (range 170.6 to 172.7°). The cyclopentyl groups lie *endo* with respect to this fold and adopt an envelope configuration with N1 as an equatorial substituent. In contrast, the known *trans* isomers have planar AlNAlN rings with two exceptions where $R'$ is excessively large [$R'$ = biphenyl (Byers *et al.*, 1992); $R'$ = 2,6-diisopropylphenyl (Waggoner & Power, 1991)]. The aluminium centre is pseudo-tetrahedral with the largest deviation from ideal geometry being the closure of the internal ring angle N1—Al1—N1* to 86.8 (1)°. This is compensated for by separating the methyl groups and thus C1—Al1—C2 expands to 120.6 (2)°. N1 is also pseudo-tetrahedral (as opposed to the flat $CNAl_2$ fragment found for $R'$ = 2,6-diisopropylphenyl above) and subtends an endocyclic ring angle of 92.0 (1)°. There are no intermolecular contacts significantly shorter than the sum of the van der Waals radii.



(I)

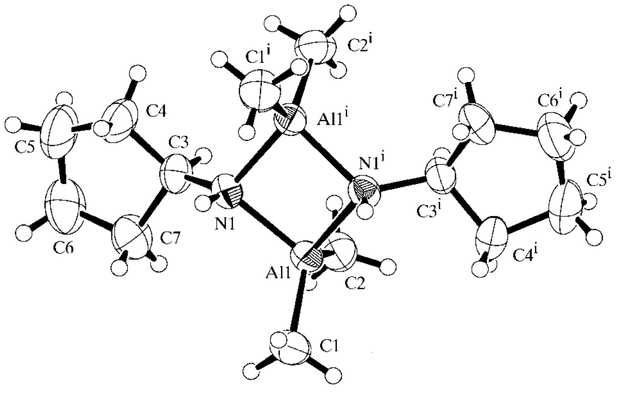


**Figure 1**
An *ORTEP* view of (I) with ellipsoids at the 40% probability level and H atoms shown as small spheres of arbitrary size. [Symmetry code: (i) 1−*y*, 1−*x*, $\frac{1}{2}$−*z*.]

## Experimental

Synthesis was carried out by adaption of the deprotonation/alkane elimination method of Waggoner & Power (1991). A suitable crystal was obtained from toluene solution and mounted in a glass capilliary.

*Crystal data*

$[Al_2(CH_3)_4(C_5H_{10}N)_2]$
$M_r$ = 282.38
Tetragonal, $P4_12_12$
$a$ = 12.380 (2) Å
$c$ = 12.018 (3) Å
$V$ = 1841.9 (7) Å$^3$
$Z$ = 4
$D_x$ = 1.018 Mg m$^{-3}$

Mo *Kα* radiation
Cell parameters from 16 reflections
$\theta$ = 10.6–13.7°
$\mu$ = 0.147 mm$^{-1}$
$T$ = 295 K
Block, colourless
0.42 × 0.40 × 0.35 mm

*Data collection*

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
2580 measured reflections
2133 independent reflections
1225 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\rm max} = 27.50°$

$h = -16 \rightarrow 16$
$k = -10 \rightarrow 11$
$l = -15 \rightarrow 15$
3 standard reflections
every 150 reflections
intensity decay: 1.12%

*Refinement*

Refinement on $F$
$R = 0.046$
$wR = 0.046$
$S = 1.408$
1225 reflections
86 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.16$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Al1—N1 | 1.943 (3) | Al1—C1 | 1.957 (4) |
| Al1—N1$^{\rm i}$ | 1.956 (3) | Al1—C2 | 1.959 (4) |
| | | | |
| N1—Al1—N1$^{\rm i}$ | 86.8 (1) | C1—Al1—C2 | 120.6 (2) |
| N1—Al1—C1 | 111.6 (2) | Al1—N1—Al1$^{\rm i}$ | 92.0 (1) |
| N1—Al1—C2 | 109.9 (2) | Al1—N1—C3 | 121.3 (2) |
| N1$^{\rm i}$—Al1—C1 | 112.2 (1) | Al1$^{\rm i}$—N1—C3 | 121.1 (2) |
| N1$^{\rm i}$—Al1—C2 | 110.7 (1) | | |

Symmetry code: (i) $1 - y, 1 - x, \frac{1}{2} - z$.

The amide-H atom was refined isotropically [N—H 0.80 (3) Å] and all other H atoms were placed in idealized positions. Reflections and their Friedel mates were collected but no reliable conclusion could be reached as to the absolute configuration.

Cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1985); data reduction: *TEXSAN* (Molecular Structure Corporation, 1993); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

We thank the EPSRC for providing a studentship to FJC.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1061). Services for accessing these data are described at the back of the journal.

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