

Crystallographic report

[Methylaluminum- μ -oxo-dimethylaluminum-trimethylethylenediamide]₂

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Dimeric and centrosymmetric [MeAlO·Me₂AlNMe(CH₂)₂NMe₂]₂ comprises two different kinds of aluminum center. One is tetrahedrally coordinated by two methyl groups, the nitrogen atom of one ligand molecule and one bridging oxygen atom, and the other is coordinated by one methyl group, two bridging oxygen atoms and two nitrogen atoms, derived from the amide ligand molecule in a distorted trigonal bipyramidal fashion. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; aluminum; amide

COMMENT

The investigation of the hydrolysis of aluminum amides is a contribution to the study of the properties of nitrogen donor ligand complexes of aluminum.^{1–3} The title compound **I** was prepared by reaction of Me₂AlNMe(CH₂)₂NMe₂ with water and exists as dimeric and centrosymmetric molecules exhibiting two different kinds of aluminum geometry. **I** (Fig. 1) may be described as a methyl alumoxane unit coordinated to the starting material Me₂AlNMe(CH₂)₂NMe₂. Tetrahedrally coordinated aluminum atoms are surrounded by two methyl groups and nitrogen atoms of the amide ligand. The second independent aluminum atom is coordinated in a distorted trigonal bipyramidal fashion by one methyl group, two bridging oxygen atoms and two nitrogen atoms of the amide ligands.

EXPERIMENTAL

The starting compound Me₂AlNMe(CH₂)₂NMe₂ was prepared by the literature procedure.¹ To a solution of Me₂AlNMe(CH₂)₂NMe₂ (1.4 g, 0.01 mol) in diethyl ether (30 ml), water (0.1 ml, 0.005 mol) was added by syringe with continuous stirring. After 1 week, colorless crystals of **I** were formed at –60 °C. The solid was collected and dried in vacuum. Yield: 0.9 g (46% yield). Intensity data for **I** were collected at 213 K on a Bruker SMART CCD diffractometer

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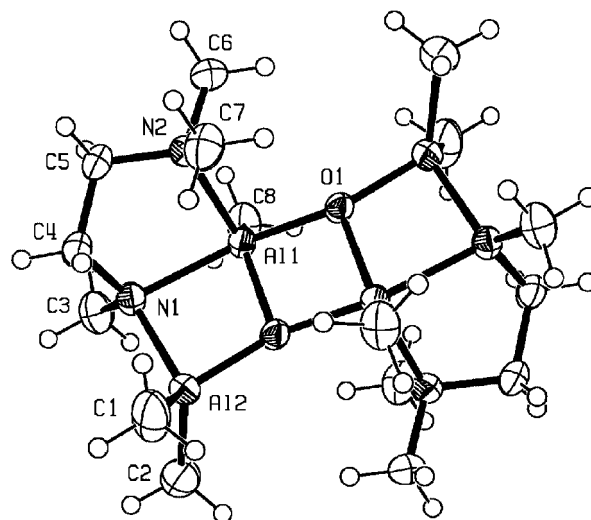


Figure 1. Molecular structure of **I**. Key geometric parameters: Al1–O1 1.845(2), Al1–N1 2.137(3), Al1–N2 2.057(3), Al1–C8 1.972(4), Al1–O1ⁱ 1.832(2), Al2–O1ⁱ 1.779(2), Al2–N1 1.935(3), Al2–C1 1.967(5), Al2–C2 1.959(5) Å; O1–Al1–N1 154.7(1), O1–Al1–N2 91.5(1), O1–Al1–C8 103.8(1), N1–Al1–N2 82.7(1), N1–Al1–C8 101.2(1), N2–Al1–C8 110.5(1), Al1–O1ⁱ–Al2 152.4(1), O1–Al1–Al2 117.9(1), Al2–Al1–C8 120.5(1), N2–Al1–Al2 108.4(1), N1–Al1–Al2 43.1(1), O1–Al2–N1 88.1(1)°. Symmetry operation: *i* = 1 – *x*, –*y*, –*z*.

for a colorless crystal 0.30 × 0.30 × 0.40 mm³; C₁₆H₄₄Al₄N₄O₂, *M* = 432.8, monoclinic, *P*2₁/*n*, *a* = 9.842(2), *b* = 9.563(2), *c* = 13.910(3) Å,

$\beta = 103.22(3)^\circ$, $V = 1274.5(5) \text{ \AA}^3$, $Z = 2$ (dimers), 1840 unique data ($\theta_{\text{max}} = 23.4^\circ$), $R = 0.054$ (1300 data with $[I \geq 2\sigma(I)]$), $wR = 0.147$ (all data). Programs used: SAINT, SHELXS97, SHELXL97, WinGX, and ORTEP3. CCDC deposition number: 238047.

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