

A dimeric alkyl complex supported by an
O,S,S,O-tetradentate diphenolate ligandHaiyan Ma,^a Gianluca Melillo,^b
Leone Oliva,^b Thomas P.
Spaniol^{a*} and Jun Okuda^a^aInstitute of Inorganic Chemistry, Aachen
University of Technology (RWTH), Prof.-Pirlet-
Str. 1, D-52056 Aachen, Germany, and^bDipartimento di Chimica, Università di Salerno,
Via S. Allende, I-84081 Baronissi (SA), ItalyCorrespondence e-mail:
thomas.spaniol@ac.rwth-aachen.de

Key indicators

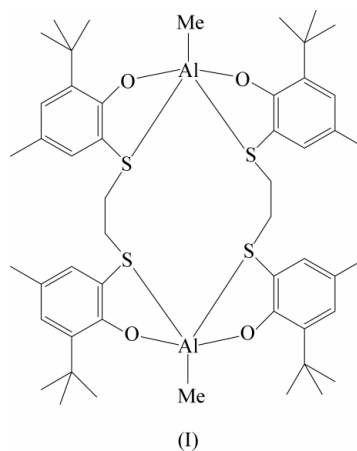
Single-crystal X-ray study
T = 110 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.041
wR factor = 0.109
Data-to-parameter ratio = 22.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title dinuclear compound, bis[μ -6,6'-di-*tert*-butyl-4,4'-dimethyl-2,2-(ethylenedithio)diphenolato]bis[methylaluminium(I)], $[\text{Al}_2(\text{CH}_3)_2(\text{C}_{24}\text{H}_{32}\text{O}_2\text{S}_2)]$ or $[\text{AlMe}(\text{etbmp})]_2$ [where etbmpH_2 is 6,6'-di-*tert*-butyl-4,4'-dimethyl-2,2-(ethylenedithio)diphenol], was obtained by reaction of trimethylaluminium with etbmpH_2 , as shown by X-ray diffraction of a single crystal. The molecule possesses a crystallographic twofold axis.

Received 10 December 2004
Accepted 20 December 2004
Online 8 January 2005

Comment

Reaction of trimethylaluminium with 1,4-dithiabutanediyl-bis(6-*tert*-butyl-4-methylphenol) (etbmpH_2) gives a compound of composition $\text{AlMe}(\text{etbmp})$ (Ma *et al.*, 2005). However, in one reaction carried out independently, a dinuclear compound of composition $[\text{AlMe}(\text{etbmp})]_2$, (I), was obtained, as shown by X-ray diffraction. The dinuclear compound does not seem to be interconvertible with the mononuclear compound. A related structure was reported for another dimeric aluminium complex, where two (*R*)-(SalBinap) ligands span two metal centres and are additionally bridged by two methoxy groups (Ovitt & Coates, 2002). Fig. 1 shows the molecular structure of the compound with crystallographic C_2 symmetry.



Experimental

Crystals were obtained by evaporation of a hexane solution of (I).

Crystal data

$[\text{Al}_2(\text{CH}_3)_2(\text{C}_{24}\text{H}_{32}\text{O}_2\text{S}_2)]$
 $M_r = 917.33$
 Orthorhombic, $Pccn$
 $a = 10.7048 (17) \text{ \AA}$
 $b = 16.380 (3) \text{ \AA}$
 $c = 28.722 (5) \text{ \AA}$
 $V = 5036.2 (14) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.210 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 Cell parameters from 8096
 reflections
 $\theta = 1.9\text{--}28.3^\circ$
 $\mu = 0.27 \text{ mm}^{-1}$
 $T = 110 (2) \text{ K}$
 Irregular fragment, colourless
 $0.60 \times 0.56 \times 0.30 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.857$, $T_{\max} = 0.925$

50262 measured reflections

6270 independent reflections

5520 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\text{max}} = 28.3^\circ$

$h = -14 \rightarrow 14$

$k = -21 \rightarrow 21$

$l = -38 \rightarrow 37$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.109$

$S = 1.07$

6270 reflections

280 parameters

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 2.2698P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.50 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.24 \text{ e } \text{\AA}^{-3}$

Table 1

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

Al—O1	1.7645 (11)	Al—S1	2.5673 (6)
Al—O2	1.7755 (11)	Al—S2	2.5897 (6)
Al—C25	1.9441 (16)		
O1—Al—O2	131.03 (5)	C25—Al—S1	104.42 (6)
O1—Al—C25	113.77 (7)	O1—Al—S2	84.76 (4)
O2—Al—C25	115.16 (7)	O2—Al—S2	80.24 (4)
O1—Al—S1	82.16 (4)	C25—Al—S2	106.15 (6)
O2—Al—S1	87.74 (4)	S1—Al—S2	149.42 (2)

All H atoms were placed in idealized positions (C—H 0.98 \AA) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2$ or 1.5 times $U_{\text{eq}}(\text{C})$. Torsional refinement was applied for the H atoms of the methyl groups.

Data collection: *SAINT-Plus* (Bruker, 1999); cell refinement: *SMART* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

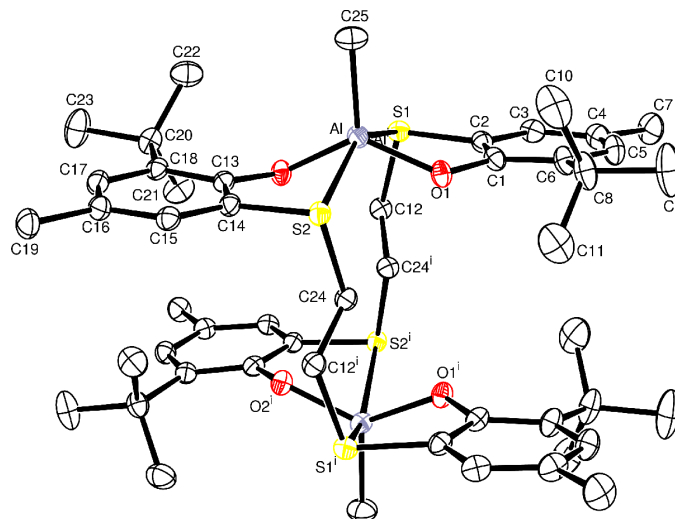


Figure 1

Displacement ellipsoid plot (30% probability) for (I). H atoms are omitted for clarity. [Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, z$].

Support from the DFG and the Fonds der Chemischen Industrie is gratefully acknowledged.

References

- Bruker (1999). *SAINT-Plus*. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2001). *SMART*. Version 5.624. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Ma, H., Melillo, G., Oliva, L., Spaniol, T. P., Englert, U. & Okuda, J. (2005). *Dalton Trans.* Submitted.
- Ovitt, T. M., Coates, G. W. (2002). *J. Am. Chem. Soc.* **124**, 1316–1326.
- Sheldrick, G. M. (1985). *SHELXS86*. University of Göttingen, Germany.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, University of Göttingen, Germany.