

Table 2. Positional and thermal parameters with e.s.d.'s in parentheses

	$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$			$U_{eq}(\text{\AA}^2)$
	x	y	z	
Pt	0.0	0.0	0.0	0.02110 (4)
N(1)	-0.0283 (4)	0.2332 (3)	-0.0207 (5)	0.0307 (7)
N(2)	0.2755 (4)	0.1077 (3)	-0.1136 (6)	0.0308 (7)
C(1)	0.1778 (6)	0.3583 (4)	-0.0316 (8)	0.0386 (10)
C(2)	0.2724 (6)	0.2642 (4)	-0.2196 (7)	0.0372 (10)
Cl	0.6806 (1)	0.2601 (1)	0.4178 (2)	0.0390 (3)

Table 3. Bond lengths (Å), angles (°), N—H...Cl hydrogen bonds (Å) and torsion angles (°)

Pt—N(1)	2.039 (3)	Pt—N(2)	2.046 (3)
N(1)—C(1)	1.492 (4)	N(2)—C(2)	1.483 (5)
C(1)—C(2)	1.502 (6)		
N(1)...Cl <sup>ii</sup>	3.242 (4)	N(1)...Cl <sup>iii</sup>	3.213 (3)
N(2)...Cl	3.255 (4)	N(2)...Cl <sup>iii</sup>	3.365 (4)
N(1)—Pt—N(2)	83.1 (1)	Pt—N(1)—C(1)	109.3 (2)
Pt—N(2)—C(2)	109.0 (2)	N(1)—C(1)—C(2)	107.3 (3)
N(2)—C(2)—C(1)	107.5 (3)		
N(2)—Pt—N(1)—C(1)	14.0	N(1)—Pt—N(2)—C(2)	15.2
Pt—N(1)—C(1)—C(2)	-40.2	Pt—N(2)—C(2)—C(1)	-41.2
N(1)—C(1)—C(2)—N(2)	53.4		

Symmetry operations: (i)  $-1 + x, y, z$ ; (ii)  $-1 + x, y, -1 + z$ ; (iii)  $1 - x, -y, -z$ .

**Related literature.** The geometry and conformation of the en ring (en = ethylenediamine) are very similar to those in [PtCl<sub>2</sub>(en)<sub>2</sub>](ClO<sub>4</sub>)<sub>2</sub> (Sato, Haruki, Wachter & Kurita, 1990).

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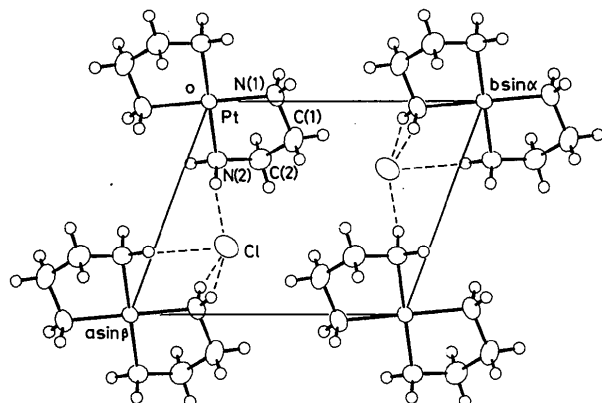


Fig. 1. A projection of the structure along *c* with the atomic numbering scheme. Thermal ellipsoids are drawn at the 60% probability level; a value of 0.8 Å<sup>2</sup> is given for the *B*<sub>iso</sub> of the H atoms in this figure. Broken lines indicate N—H...Cl hydrogen bonds.

M-380R computer at ISSP, The University of Tokyo. Computational programs used were *UNICSII* (Sakurai, 1967), *RADIEL* (Coppens, Guru Row, Leung, Stevens, Becker & Yang, 1979) and *ORTEPII* (Johnson, 1971).

*Acta Cryst.* (1990). **C46**, 1108–1110

## Structure of an Optically Active Organoaluminium Naphthylethylamine Dimer

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**Abstract.** Bis- $\mu$ -[1-(1-naphthyl)ethylaminato-*N*]-bis[aluminum(III)], [Al<sub>2</sub>(CH<sub>3</sub>)<sub>4</sub>(C<sub>12</sub>H<sub>12</sub>N)<sub>2</sub>], *M<sub>r</sub>* = 454.62, monoclinic, *P*2<sub>1</sub>, *a* = 10.925 (4), *b* = 11.129 (4), *c* = 11.212 (4) Å,  $\beta$  = 94.18 (3)°, *V* = 1359.6 (8) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.11 g cm<sup>-3</sup>, *Mo K $\alpha$* ,  $\lambda$  = 0.71073 Å,  $\mu$  = 1.19 cm<sup>-1</sup>, *F*(000) = 488, *T* = 294 K, *R* = 0.0355 for 1615 observed reflections. The reaction of trimethylaluminum with (*R*)-(+)-1-(1-naph-

thyl)ethylamine involves cleavage of Al—C<sub>Me</sub> and N—H bonds resulting in elimination of methane and formation of an asymmetric Al<sub>2</sub>N<sub>2</sub> fragment, as the core of a dimeric molecule consisting of two (C<sub>10</sub>H<sub>7</sub>)CH(CH<sub>3</sub>)NHAl(CH<sub>3</sub>)<sub>2</sub> units. The molecule has approximate C<sub>2</sub> symmetry, with an average Al—N bond distance of 1.96 (1) Å.

**Experimental.** The title compound results from the slow addition of a trimethylaluminum/toluene solu-

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Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ ) with e.s.d.'s in parentheses

	x	y	z	$U_{eq}^*$
Al(1)	1174 (1)	4015	2043 (1)	54 (1)
Al(2)	3524 (1)	3307 (2)	2982 (1)	50 (1)
N(1)	1831 (3)	2875 (4)	3242 (3)	51 (1)
N(2)	2868 (3)	4200 (3)	1576 (3)	48 (1)
C(1)	28 (4)	2904 (5)	4509 (4)	60 (2)
C(2)	-691 (5)	2331 (5)	3640 (5)	79 (2)
C(3)	-1969 (5)	2291 (6)	3654 (7)	100 (3)
C(4)	-2530 (5)	2783 (7)	4550 (7)	98 (3)
C(5)	-1840 (5)	3365 (6)	5481 (6)	78 (2)
C(6)	-2403 (7)	3892 (7)	6457 (7)	104 (3)
C(7)	-1722 (7)	4404 (7)	7370 (7)	111 (4)
C(8)	-460 (7)	4445 (6)	7391 (5)	94 (3)
C(9)	127 (5)	3989 (5)	6439 (5)	72 (2)
C(10)	-540 (4)	3430 (5)	5479 (4)	62 (2)
C(11)	1411 (4)	2915 (5)	4474 (4)	55 (2)
C(12)	1992 (5)	1881 (5)	5220 (4)	71 (2)
C(13)	184 (4)	3219 (6)	748 (4)	82 (2)
C(14)	611 (5)	5479 (6)	2782 (5)	81 (2)
C(15)	4438 (4)	1879 (5)	2576 (5)	72 (2)
C(16)	4164 (4)	4431 (5)	4208 (4)	74 (2)
C(17)	2624 (5)	6001 (5)	292 (5)	76 (2)
C(18)	3338 (4)	5421 (4)	1374 (4)	52 (2)
C(19)	4705 (4)	5460 (4)	1224 (4)	50 (2)
C(20)	5307 (4)	4501 (5)	789 (4)	60 (2)
C(21)	6569 (5)	4560 (5)	611 (5)	74 (2)
C(22)	7226 (5)	5567 (5)	859 (4)	69 (2)
C(23)	6640 (4)	6593 (5)	1310 (4)	53 (2)
C(24)	7307 (5)	7646 (5)	1575 (4)	66 (2)
C(25)	6741 (5)	8626 (5)	2014 (4)	71 (2)
C(26)	5494 (5)	8596 (5)	2197 (5)	71 (2)
C(27)	4839 (4)	7579 (4)	1945 (4)	59 (2)
C(28)	5375 (4)	6545 (4)	1492 (4)	48 (1)

$U_{eq}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

Table 2. Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Al(1)—N(1)	1.947 (4)	Al(1)—N(2)	1.970 (3)
Al(1)—C(13)	1.958 (5)	Al(1)—C(14)	1.948 (6)
Al(2)—N(1)	1.953 (4)	Al(2)—N(2)	1.955 (4)
Al(2)—C(15)	1.949 (5)	Al(2)—C(16)	1.950 (5)
N(1)—C(11)	1.487 (6)	N(2)—C(18)	1.476 (6)
C(1)—C(11)	1.515 (6)	C(11)—C(12)	1.533 (7)
C(17)—C(18)	1.536 (7)	C(18)—C(19)	1.516 (6)
N(1)—Al(1)—N(2)	86.9 (1)	N(1)—Al(1)—C(13)	111.9 (2)
N(2)—Al(1)—C(13)	109.2 (2)	N(1)—Al(1)—C(14)	111.3 (2)
N(2)—Al(1)—C(14)	110.9 (2)	C(13)—Al(1)—C(14)	121.3 (2)
N(1)—Al(2)—N(2)	87.1 (1)	N(1)—Al(2)—C(15)	110.0 (2)
N(2)—Al(2)—C(15)	113.0 (2)	N(1)—Al(2)—C(16)	110.3 (2)
N(2)—Al(2)—C(16)	109.5 (2)	C(15)—Al(2)—C(16)	121.6 (2)
Al(1)—N(1)—Al(2)	92.4 (2)	Al(1)—N(1)—C(11)	119.9 (3)
Al(2)—N(1)—C(11)	119.4 (3)	Al(1)—N(2)—Al(2)	91.7 (2)
Al(1)—N(2)—C(18)	118.8 (3)	Al(2)—N(2)—C(18)	118.7 (3)
C(2)—C(1)—C(11)	120.6 (4)	C(10)—C(1)—C(11)	120.6 (4)
N(1)—C(11)—C(1)	113.5 (3)	N(1)—C(11)—C(12)	109.9 (4)
C(1)—C(11)—C(12)	110.7 (4)	N(2)—C(18)—C(17)	110.2 (3)
N(2)—C(18)—C(19)	113.6 (4)	C(17)—C(18)—C(19)	110.4 (4)
C(18)—C(19)—C(20)	121.5 (4)	C(18)—C(19)—C(28)	119.5 (4)

tion to (*R*)-(+)-1-naphylethylamine under a rigorously inert atmosphere, followed by heating (363 K) in an oil bath for several hours. Large colorless crystals form upon slow cooling to room temperature. Colorless parallelepiped crystal from slow evaporation of toluene solution, dimensions  $0.10 \times 0.50 \times 0.70$  mm. Data collected at room temperature, graphite-monochromated Mo  $K\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ), Nicolet R3mV diffractometer,  $\omega/2\theta$  scans of  $2-15^\circ \text{ min}^{-1}$ ,  $2\theta_{\text{max}} = 45^\circ$ ,  $h = -11$  to  $0$ ,  $k$

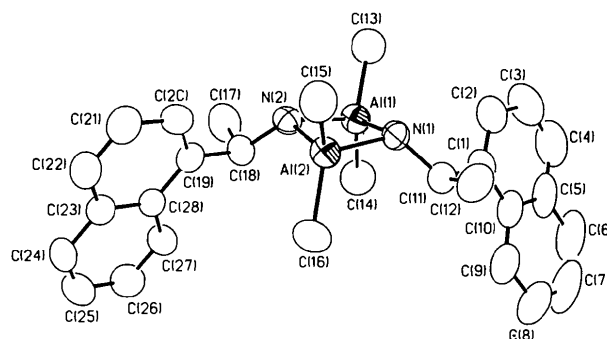


Fig. 1. Thermal ellipsoid plot (50% probability) of the molecule; H atoms omitted

= 0 to 12,  $l = -12$  to 12, 2007 measurements, 1892 unique ( $R_{\text{int}} = 0.007$ ), 1615 observed [ $I > 2.58\sigma(I)$ ]. Unit-cell dimensions determined by least-squares fit to settings for 33 reflections ( $18 < 2\theta < 30^\circ$ ). Empirical absorption correction, transmission factors 0.92–1.00; three standards monitored ( $\pm 2\%$ ), 17.1 h of X-ray exposure. Solved by direct methods, full-matrix least-squares refinement on  $F$ ,  $R = 0.0355$ ,  $wR = 0.0418$ ,  $S = 1.13$ , 290 variables including positional and anisotropic thermal parameters for non-H atoms, H atoms, located by standard techniques, included in structure-factor calculation at optimized (C—H =  $0.96 \text{ \AA}$ ) difference-map positions, with separate isotropic group thermal parameters for the methyl H atoms [ $U = 0.102 (4) \text{ \AA}^2$ ] and the remaining H atoms [ $U = 0.078 (4) \text{ \AA}^2$ ]. Function minimized was  $\sum w(|F_o| - |F_c|)^2$ , where  $w = 1/[\sigma^2(F_o) + 0.0005|F_o|^2]$ ,  $(\Delta/\sigma)_{\text{max}} = 0.01$ , final difference-map peaks of  $0.13$  and  $-0.11 \text{ e \AA}^{-3}$ . Computer programs SHELXTL (Sheldrick, 1986), scattering factors from Cromer & Waber (1974), real and imaginary anomalous-dispersion corrections from Cromer (1974).

Final atomic coordinates are listed in Table 1,\* selected bond distances and angles are given in Table 2. The molecule is displayed in Fig. 1, which also gives the atom-numbering scheme.

**Related literature.** The crystal structure of the related (*S*)-(–)-1-phenylethylamine derivative is reported by Robinson, Sangokoya & Rogers (1988).

\* Tables of crystallographic details, bond distances, bond angles, anisotropic thermal parameters, H-atom coordinates and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52438 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of a Condensation Product of Triethylaluminium and a Multidentate Open-Chain Amine

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**Abstract.**  $\mu_4$ -[1,4-Bis(3-aminopropyl)piperazinato-*N, N', N'', N'''*]decaethyltetraaluminium, [Al<sub>4</sub>(C<sub>2</sub>H<sub>5</sub>)<sub>10</sub>(C<sub>10</sub>H<sub>22</sub>N<sub>4</sub>)], *M<sub>r</sub>* = 596.98, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 8.283 (1), *b* = 13.657 (2), *c* = 17.361 (2) Å,  $\beta$  = 102.63 (1)°, *V* = 1916.4 (4) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.03 g cm<sup>-3</sup>, Mo *K*α,  $\lambda$  = 0.71073 Å,  $\mu$  = 1.40 cm<sup>-1</sup>, *F*(000) = 664, *T* = 294 K, *R* = 0.0516 for 1687 observed reflections. The molecule possesses crystallographic inversion symmetry; each piperazine heteroatom and terminal N atom of the attached aminopropyl group is bridged by a diethylaluminium unit. In addition a terminal triethylaluminium group is bonded to each of the aminopropyl N atoms.

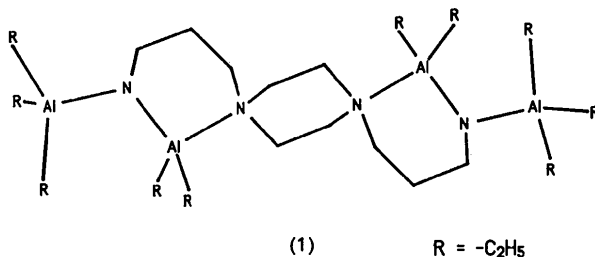
**Experimental.** The title compound (1) is formed by the reaction of 1,4-bis(3-aminopropyl)piperazine and triethylaluminium in a methylene chloride/pentane solution, and involves the cleavage of an ethyl group from two triethylaluminium molecules and one H atom from each amino group of the piperazine derivative, with subsequent generation of two equivalents of ethane gas. Colorless parallelepiped crystal, dimensions 0.20 × 0.25 × 0.65 mm. Data collected at room temperature, graphite-monochromated Mo *K*α radiation ( $\lambda$  = 0.71073 Å), Nicolet R3mV diffractometer,  $\omega/2\theta$  scans of 2–15° min<sup>-1</sup>,  $2\theta_{\max}$  = 45°, *h* = 0 to 9, *k* = -15 to 0, *l* = -19 to 19, 3852 measurements, 2509 unique (*R*<sub>int</sub> =

0.020), 1687 observed [*I* > 3σ(*I*)]. Unit-cell dimensions determined by least-squares fit to settings for 49 reflections (27 < 2θ < 43°). Empirical absorption correction ( $\mu$  = 1.40 cm<sup>-1</sup>), transmission factors 0.95–1.00; three standards monitored (±1%), 31.1 h of X-ray exposure. Solved by direct methods; full-matrix least-squares refinement on *F*, *R* = 0.0516, *wR* = 0.0663, *S* = 1.79, ( $\Delta/\sigma$ )<sub>max</sub> = 0.01, 217 variables including positional parameters and anisotropic thermal parameters for all non-H atoms; H atoms located by standard techniques; those of the piperazine ring and the aminopropyl group refined isotropically; ethyl H atoms included in structure factor calculations at idealized positions (C—H = 0.96 Å) with group isotropic thermal parameter [*U*<sub>H</sub> = 0.136 (6) Å<sup>2</sup>]. Function minimized was  $\sum w(|F_o| - |F_c|)^2$ , where  $w = 1/[\sigma^2(F_o) + 0.0005|F_o|^2]$ . Final

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

*U*<sub>eq</sub> is defined as one third of the trace of the orthogonalized

	<i>U</i> <sub>ij</sub> tensor.			<i>U</i> <sub>eq</sub> (Å <sup>2</sup> )
	<i>x</i>	<i>y</i>	<i>z</i>	
Al(1)	0.1232 (2)	0.2264 (1)	0.4494 (1)	0.042 (1)
Al(2)	0.2248 (2)	0.3514 (1)	0.3035 (1)	0.048 (1)
N(1)	0.0085 (4)	0.0918 (2)	0.4516 (2)	0.040 (1)
N(2)	0.1411 (5)	0.2250 (3)	0.3418 (2)	0.043 (2)
C(1)	-0.0599 (7)	0.0900 (3)	0.5254 (3)	0.046 (2)
C(2)	0.1293 (6)	0.0093 (3)	0.4592 (3)	0.044 (2)
C(3)	-0.1307 (6)	0.0825 (4)	0.3797 (3)	0.050 (2)
C(4)	-0.0749 (8)	0.0927 (4)	0.3020 (3)	0.053 (2)
C(5)	-0.0133 (7)	0.1936 (4)	0.2857 (3)	0.053 (2)
C(6)	0.3322 (6)	0.2145 (4)	0.5285 (3)	0.058 (2)
C(7)	0.4870 (7)	0.1724 (5)	0.5056 (4)	0.083 (3)
C(8)	-0.0416 (7)	0.3245 (4)	0.4637 (3)	0.067 (2)
C(9)	-0.0195 (11)	0.3675 (5)	0.5452 (4)	0.126 (4)
C(10)	0.3575 (8)	0.4163 (4)	0.3988 (3)	0.073 (2)
C(11)	0.4392 (10)	0.5123 (5)	0.3836 (3)	0.116 (4)
C(12)	0.3541 (6)	0.3107 (4)	0.2245 (3)	0.061 (2)
C(13)	0.5350 (8)	0.2902 (5)	0.2540 (4)	0.100 (3)
C(14)	0.0272 (7)	0.4301 (4)	0.2515 (3)	0.068 (2)
C(15)	0.0692 (8)	0.5156 (5)	0.2068 (4)	0.110 (4)



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