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

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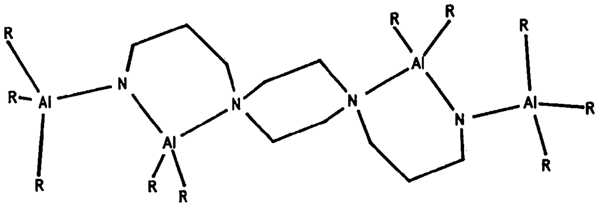
**Abstract.** $\mu_4$-[1,4-Bis(3-aminopropyl)piperazinato-*N*, *N*′,*N*″,*N*‴]decaethyltetraaluminium, $[Al_4(C_2H_5)_{10}(C_{10}H_{22}N_4)]$, $M_r = 596{\cdot}98$, monoclinic, $P2_1/n$, $a = 8{\cdot}283\,(1)$, $b = 13{\cdot}657\,(2)$, $c = 17{\cdot}361\,(2)$ Å, $\beta = 102{\cdot}63\,(1)°$, $V = 1916{\cdot}4\,(4)$ Å$^3$, $Z = 2$, $D_x = 1{\cdot}03$ g cm$^{-3}$, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}40$ cm$^{-1}$, $F(000) = 664$, $T = 294$ K, $R = 0{\cdot}0516$ 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 $K\alpha$ radiation ($\lambda = 0{\cdot}71073$ Å), Nicolet *R*3*mV* diffractometer, $\omega/2\theta$ scans of 2–15° min$^{-1}$, $2\theta_{max} = 45°$, $h = 0$ to 9, $k = -15$ to 0, $l = -19$ to 19, 3852 measurements, 2509 unique ($R_{int} = 0{\cdot}020$), 1687 observed [$I > 3\sigma(I)$]. Unit-cell dimensions determined by least-squares fit to settings for 49 reflections ($27 < 2\theta < 43°$). Empirical absorption correction ($\mu = 1{\cdot}40$ cm$^{-1}$), 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*, $R = 0{\cdot}0516$, $wR = 0{\cdot}0663$, $S = 1{\cdot}79$, $(\Delta/\sigma)_{max} = 0{\cdot}01$, 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 [$U_H = 0{\cdot}136\,(6)$ Å$^2$]. Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0{\cdot}0005|F_o|^2]$. Final



(1) R = $-C_2H_5$

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

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

| | $x$ | $y$ | $z$ | $U_{eq}$(Å$^2$) |
|---|---|---|---|---|
| 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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Table 2. *Selected bond distances* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Al(1)—N(1) | 2·073 (4) | Al(1)—N(2) | 1·906 (4) |
| Al(1)—C(6) | 1·967 (5) | Al(1)—C(8) | 1·967 (6) |
| Al(2)—N(2) | 2·025 (4) | Al(2)—C(10) | 1·985 (5) |
| Al(2)—C(12) | 1·995 (6) | Al(2)—C(14) | 2·000 (5) |
| N(1)—C(1) | 1·510 (7) | N(1)—C(2) | 1·493 (6) |
| N(1)—C(3) | 1·508 (6) | N(2)—C(5) | 1·491 (6) |
| C(1)—C(2*A*) | 1·520 (7) | C(3)—C(4) | 1·526 (8) |
| C(4)—C(5) | 1·517 (8) | | |

| | | | |
|---|---|---|---|
| N(1)—Al(1)—N(2) | 98·3 (2) | N(1)—Al(1)—C(6) | 104·5 (2) |
| N(2)—Al(1)—C(6) | 116·0 (2) | N(1)—Al(1)—C(8) | 105·6 (2) |
| N(2)—Al(1)—C(8) | 109·6 (2) | C(6)—Al(1)—C(8) | 119·8 (2) |
| N(2)—Al(2)—C(10) | 105·9 (2) | N(2)—Al(2)—C(12) | 105·2 (2) |
| C(10)—Al(2)—C(12) | 114·5 (3) | N(2)—Al(2)—C(14) | 107·5 (2) |
| C(10)—Al(2)—C(14) | 112·7 (2) | C(12)—Al(2)—C(14) | 110·4 (2) |
| Al(1)—N(1)—C(1) | 106·7 (3) | Al(1)—N(1)—C(2) | 111·7 (3) |
| C(1)—N(1)—C(2) | 106·3 (4) | Al(1)—N(1)—C(3) | 109·3 (3) |
| C(1)—N(1)—C(3) | 109·9 (4) | C(2)—N(1)—C(3) | 112·7 (4) |
| Al(1)—N(2)—Al(2) | 114·8 (2) | Al(1)—N(2)—C(5) | 113·8 (4) |
| Al(2)—N(2)—C(5) | 109·7 (3) | N(1)—C(1)—C(2*A*) | 112·9 (4) |
| N(1)—C(2)—C(1*A*) | 114·7 (4) | N(1)—C(3)—C(4) | 113·5 (4) |
| C(3)—C(4)—C(5) | 115·2 (4) | N(2)—C(5)—C(4) | 114·5 (4) |

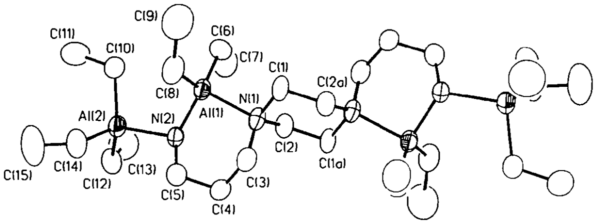

Fig. 1. Thermal ellipsoid plot (35% probability) and atomic numbering for $[Al(C_2H_5)_2]_2[C_{10}H_{22}N_4][Al(C_2H_5)_3]_2$.

difference map peaks ranged from $-0.23$ to $0.26\ e\ Å^{-3}$. Computer programs of Sheldrick (1986), scattering factors from Cromer & Waber (1974), and real and imaginary anomalous-dispersion corrections from Cromer (1974). Final atomic coordinates are given in Table 1,* selected distances and angles are reported in Table 2. The molecule is displayed in Fig. 1.

**Related literature.** Synthesis and structure of the methyl derivative: Robinson, Moise, Pennington & Sangokoya (1988). For related structures of other multidentate open-chain amine/organoaluminium products see Sangokoya, Moise, Pennington, Self & Robinson (1989), Robinson, Moise, Pennington & Sangokoya (1989), Robinson, Sangokoya, Moise & Pennington (1988), Robinson, Moise, Pennington & Sangokoya (1988) and Robinson & Sangokoya (1987).

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* A summary of crystallographic details, tables of anisotropic thermal parameters, distances and angles, H-atom coordinates and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52598 (14 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 an Iridium Bis(phosphine) Diene Complex, a Catalyst for Homogeneous Hydrogenation

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**Abstract.** ($\eta^4$-1,5-Cyclooctadiene)bis{tris(4-methoxyphenyl)phosphine}iridium(I) hexafluorophosphate, $[Ir(C_8H_{12})(C_{21}H_{21}O_3P)_2][PF_6]$, $M_r = 1150{\cdot}1$, monoclinic, space group $P2_1/c$, $a = 11{\cdot}757\,(5)$, $b = 19{\cdot}759\,(7)$, $c = 20{\cdot}468\,(8)$ Å, $\beta = 93{\cdot}85\,(4)$°, $V = 4744{\cdot}0$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}61$ g cm$^{-3}$, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 29{\cdot}7$ cm$^{-1}$, $F(000) = 2312$, $T = 295$ K; $R = 0{\cdot}039$, $wR = 0{\cdot}055$ for 3793 reflections with $|F^2| > 3\sigma(F^2)$. The geometry at iridium is approximately square planar, with the expected distortions due to the size of the phosphine ligands: Ir—P1 2·359 (3), Ir—P2 2·341 (3) Å, P1—Ir—P2

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