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# Structure of a Second Polymorph of $[Al(CH_3)]_2[14]aneN_4[Al(CH_3)_3]_2$

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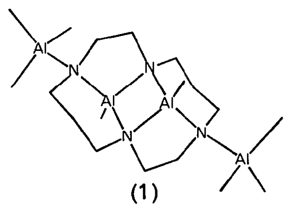
**Abstract.** Octamethyl-1$\kappa^3$,2$\kappa$,3$\kappa^3$,4$\kappa$-{$\mu_4$-[1,4,8,11-tetraazacyclotetradecanato(4−)-1:2$\kappa^2N$;2:4$\kappa^2N^4$;2:-4$\kappa^2N^{11}$;3:4$\kappa^2N^8$]}tetraaluminium, $[Al(CH_3)]_2$-$[C_{10}H_{20}N_4][Al(CH_3)_3]_2$, $M_r = 424{\cdot}58$, triclinic, $P\bar{1}$, $a = 7{\cdot}978\ (2)$, $b = 9{\cdot}428\ (2)$, $c = 9{\cdot}627\ (2)$ Å, $\alpha = 75{\cdot}46\ (2)$, $\beta = 67{\cdot}86\ (2)$, $\gamma = 73{\cdot}84\ (2)°$, $V = 635{\cdot}4\ (2)$ Å$^3$, $Z = 1$, $D_x = 1{\cdot}11$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}88$ cm$^{-1}$, $F(000) = 232$, $T = 294\ (1)$ K, $R = 0{\cdot}0314$ for 1537 observed reflections. The molecule possesses crystallographic inversion symmetry with a planar, four-membered $Al_2N_2$ ring occupying the central cavity of the macrocycle. The molecular conformation is essentially identical to that of the monoclinic form; however, the packing of the molecules in the crystal is quite different.

**Experimental.** Data quality crystals of (1) were produced by reaction of one equivalent of a 2:1 adduct (Robinson, Pennington, Lee, Self & Hrncir, 1991) of trimethylaluminium and 1,4,8,11-tetraazacyclotetradecane ($[14]aneN_4$), with two additional equivalents of trimethylaluminium at room temperature in chlorobenzene solution, followed by recrystallization from a cooled (263 K) methylene chloride/chlorobenzene solvent system. Colorless, parallelepiped crystal, dimensions 0·40 × 0·50 × 0·65 mm. Nicolet *R3mV* diffractometer, $\omega/2\theta$ scans of 2–15° min$^{-1}$, $2\theta_{max} = 45°$, $h = -9$ to 0, $k = -11$ to 11, $l = -11$ to 11, 1823 measurements, 1678 unique ($R_{int} = 0{\cdot}015$), 1537 observed [$I > 3\sigma(I)$]. Unit-cell dimensions determined by least-squares fit to settings for 33 reflections ($15 < 2\theta < 30°$). Empirical absorption correction, transmission factors: 0·98–1·00; three standards monitored (±1%), 13·2 h of X-ray exposure. Solved by direct methods; full-matrix least-squares refinement on $F$, $R = 0{\cdot}0314$, $wR = 0{\cdot}0497$, $S = 1{\cdot}74$, $(\Delta/\sigma)_{max} = 0{\cdot}01$, 171 variables including positional and anisotropic thermal parameters for the non-H atoms, positional and isotropic thermal parameters for the methylene and one set [atom C(9)] of methyl H atoms and an isotropic group thermal parameter [$U = 0{\cdot}095\ (4)$ Å$^2$] for disordered sets of half-H atoms included at optimized (staggered) positions [$d$(C—H) = 0·96 Å] for the remaining three methyl groups [atoms C(6), C(7) and C(8)]. Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0{\cdot}0005|F_o|^2]$. Final difference Fourier peaks ranged from −0·17 to 0·19 e Å$^{-3}$. Computer programs from *SHELXTL* (Sheldrick, 1985), scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B), real and imaginary anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1). Final atomic coordinates are given in Table 1,† and selected distances and angles in Table 2; the molecule is displayed in Fig. 1.



(1)

Table 1. *Atomic coordinates* (× 10$^4$) *and equivalent isotropic thermal parameters* (Å$^2$ × 10$^3$) *for* $[Al(CH_3)]_2[C_{10}H_{20}N_4][Al(CH_3)_3]_2$

| | $x$ | $y$ | $z$ | $U_{eq}$* |
|---|---|---|---|---|
| Al(1) | −184 (1) | 1128 (1) | 746 (1) | 33 (1) |
| Al(2) | −3750 (1) | 3005 (1) | 3077 (1) | 45 (1) |
| N(1) | 513 (2) | 935 (2) | −1377 (2) | 34 (1) |
| N(2) | −2773 (2) | 1913 (2) | 1263 (2) | 36 (1) |
| C(1) | −546 (3) | 2144 (2) | −2266 (2) | 47 (1) |
| C(2) | −2615 (3) | 2436 (3) | −1513 (2) | 49 (1) |
| C(3) | −3313 (3) | 3000 (2) | −13 (2) | 47 (1) |
| C(4) | −3659 (3) | 585 (2) | 1666 (3) | 43 (1) |
| C(5) | −2522 (3) | −798 (2) | 2302 (2) | 44 (1) |
| C(6) | 1474 (3) | 1866 (3) | 1287 (3) | 57 (1) |
| C(7) | −2824 (4) | 1619 (3) | 4685 (3) | 71 (1) |
| C(8) | −2742 (4) | 4852 (3) | 2346 (3) | 70 (1) |
| C(9) | −6472 (3) | 3418 (3) | 3634 (3) | 62 (1) |

* Equivalent isotropic $U$ defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

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† Lists of structure factors, anisotropic thermal parameters and H-atom parameters, as well as a summary of crystallographic details and figures comparing conformation and packing of the two forms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53766 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Bond distances* (Å) *and angles* (°) *for* $[Al(CH_3)]_2[C_{10}H_{20}N_4][Al(CH_3)_3]_2$

| | | | |
|---|---|---|---|
| Al(1)—N(1) | 1·948 (2) | Al(1)—N(2) | 1·903 (2) |
| Al(1)—C(6) | 1·929 (3) | Al(1)—N(1*a*) | 1·947 (2) |
| Al(2)—N(2) | 2·037 (2) | Al(2)—C(7) | 1·990 (3) |
| Al(2)—C(8) | 1·978 (3) | Al(2)—C(9) | 1·978 (3) |
| N(1)—C(1) | 1·505 (3) | N(1)—C(5*a*) | 1·498 (2) |
| N(2)—C(3) | 1·505 (3) | N(2)—C(4) | 1·504 (3) |
| C(1)—C(2) | 1·507 (3) | C(2)—C(3) | 1·515 (3) |
| C(4)—C(5) | 1·502 (3) | | |

| | | | |
|---|---|---|---|
| N(1)—Al(1)—N(2) | 102·3 (1) | N(1)—Al(1)—C(6) | 117·0 (1) |
| N(2)—Al(1)—C(6) | 124·1 (1) | N(1)—Al(1)—N(1*a*) | 90·3 (1) |
| N(2)—Al(1)—N(1*a*) | 92·8 (1) | C(6)—Al(1)—N(1*a*) | 123·0 (1) |
| N(2)—Al(2)—C(7) | 106·3 (1) | N(2)—Al(2)—C(8) | 106·0 (1) |
| C(7)—Al(2)—C(8) | 112·8 (1) | N(2)—Al(2)—C(9) | 105·1 (1) |
| C(7)—Al(2)—C(9) | 113·3 (1) | C(8)—Al(2)—C(9) | 112·7 (1) |
| Al(1)—N(1)—C(1) | 114·5 (1) | Al(1)—N(1)—Al(1*a*) | 89·7 (1) |
| C(1)—N(1)—Al(1*a*) | 124·5 (2) | Al(1)—N(1)—C(5*a*) | 116·9 (1) |
| C(1)—N(1)—C(5*a*) | 107·1 (1) | Al(1*a*)—N(1)—C(5*a*) | 103·9 (1) |
| Al(1)—N(2)—Al(2) | 111·8 (1) | Al(1)—N(2)—C(3) | 113·3 (1) |
| Al(2)—N(2)—C(3) | 106·9 (1) | Al(1)—N(2)—C(4) | 105·8 (1) |
| Al(2)—N(2)—C(4) | 109·5 (1) | C(3)—N(2)—C(4) | 109·6 (2) |
| N(1)—C(1)—C(2) | 114·8 (1) | C(1)—C(2)—C(3) | 115·2 (2) |
| N(2)—C(3)—C(2) | 115·5 (2) | N(2)—C(4)—C(5) | 111·3 (2) |
| C(4)—C(5)—N(1*a*) | 110·8 (1) | | |

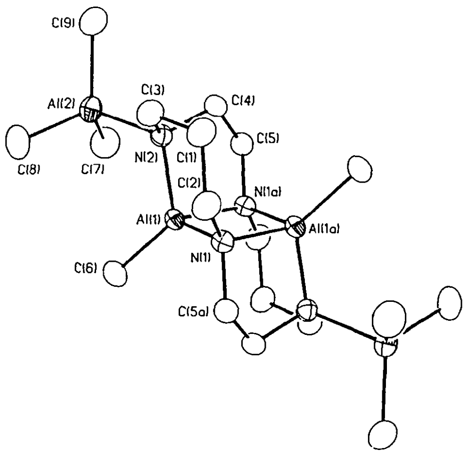

Fig. 1. Thermal ellipsoid plot (35% probability) of the molecule; H atoms are omitted.

**Related literature.** The triclinic form of the title compound represents the second polymorph of a product resulting from reaction of trimethylaluminium with [14]ane$N_4$. Reaction of one equivalent of [14]ane$N_4$ with four equivalents of trimethylaluminium, carried out at 393 K in toluene, gives monoclinic crystals of (1) (Robinson, Rae, Campana & Byram, 1987). In addition, an isomorphous gallium derivative (Lee, Pennington, Robinson & Rogers, 1990) and a mixed aluminium/gallium derivative, $[Me_3Ga]_2[14]aneN_4[MeAl]_2$ (Robinson, Pennington, Lee, Self & Hrncir, 1990), which is isomorphous with the monoclinic form, have also been isolated.

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# Structure of 1,2,2,2,3,3,3,4,4,4-Decacarbonyl-1,2:3,4-di-$\mu$-hydrido-1-tricyclohexylphosphine-3-triisopropylphosphineplatinumtriosmium(2*Pt–Os*)(3*Os–Os*)

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**Abstract.** $[Os_3Pt(\mu\text{-}H)_2(CO)_{10}\{P(C_6H_{11})_3\}\{P(i\text{-}C_3H_7)_3\}]$, $M_r = 1488·5$, monoclinic, $P2_1/c$, $a = 10·678\,(2)$, $b = 15·924\,(4)$, $c = 27·237\,(5)$ Å, $\beta = 100·92\,(2)°$, $V = 4547\,(2)$ Å$^3$, $Z = 4$, $D_x = 2·17$ g cm$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 115·85$ cm$^{-1}$, $F(000) = 2776$, $T = 298$ K, $R = 0·035$ for 3227 unique observed reflections. The metal skeleton is of the butterfly form, with a non-bonding Pt···Os(3) distance of 3·837 (1) Å, and a butterfly angle, defined by the torsion angle Pt—Os(1)—Os(2)—Os(3), of 98·2 (1)°. The pseudo-octahedral ligand sphere on Os(3) is skewed out of the $Os_3$ plane by *ca* 11·5°.

**Experimental.** Pale yellow prisms from dichloromethane/hexane solution: crystal dimensions *ca* 0·4

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