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Structure of Diphenylamine–Trichloroaluminium(III) (1/1)

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Abstract. $[AlCl_3(C_{12}H_{11}N)]$, $M_r = 302{\cdot}57$, monoclinic, $C2/c$, $a = 15{\cdot}072\ (3)$, $b = 13{\cdot}398\ (3)$, $c = 14{\cdot}603\ (3)$ Å, $\beta = 102{\cdot}95\ (2)°$, $V = 2874\ (2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}399$ g cm^{-3}, λ(Mo $K\alpha$) = 0·70926 Å, $\mu = 6{\cdot}78$ cm^{-1}, $F(000) = 1232$, $T = 291$ K, $R = 0{\cdot}0455$ for 1104 significant reflections. The structure consists of discrete molecular units, and shows no indication of hydrogen bonding. The Al–N bond length of 1·983 (4) Å is typical of a single bond.

Experimental. The title compound was unexpectedly crystallized from the reaction of $SbCl_3$, Ph_2NH and $AlCl_3$ in CH_2Cl_2, and has since been obtained from the direct reaction of Ph_2NH with $AlCl_3$ (yield 60%). A white, air-sensitive crystal 0·30 × 0·30 × 0·35 mm was mounted in a Pyrex capillary; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice constants were obtained from 25 well centred reflections in the range $20 < 2\theta < 30°$. Intensities were measured using an $\omega/2\theta$ scan mode to $2\theta_{max} = 46°$ (h_{max} 16, k_{max} 14, l_{max} 16). Three standard reflections monitored every hour showed no significant deviations in intensity, intensities reduced to a standard scale (Cameron & Cordes, 1979); Lorentz–polarization and absorption corrections were applied (*DIFABS*; Walker & Stuart, 1983). The maximum and minimum transmission factors were 1·145 and 0·709. Intensities of 2181 reflections were measured and averaged to yield 1985 unique reflections, of which 1104 were considered observed [$I > 3\sigma(I)$]. The structure was solved by direct methods (*SHELXS*86; Sheldrick, 1985) and refined initially by full-matrix least squares (*SHELX*76; Sheldrick, 1976) minimizing $\sum w(\Delta F)^2$, where $w = k/[\sigma^2(F) + gF^2]$ ($k = 1{\cdot}6229$, $g = 0{\cdot}0007$) and σ was obtained from counting statistics. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and corrected for the real part of the anomalous dispersion. All non-H atoms were refined anisotropically in two blocks; the H atoms were placed geometrically and refined isotropically with the temperature factor of the atom to which they are bound. The final cycles of refinement of 166 parameters gave $R = 0{\cdot}0455$, $wR = 0{\cdot}0468$. The largest Δ/σ was 0·587. A final difference synthesis showed no significant features and had a maximum of 0·248 and a minimum of $-0{\cdot}245$ e Å^{-3}. Table 1 lists refined fractional coordinates and Table 2 provides bond

Table 1. *Fractional atomic positional parameters and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Al(1)	0·1683 (1)	0·3767 (1)	0·5860 (1)	0·0587
Cl(1)	0·0953 (1)	0·4177 (1)	0·6860 (1)	0·0819
Cl(2)	0·1456 (1)	0·4638 (1)	0·4643 (1)	0·0926
Cl(3)	0·1521 (1)	0·2214 (1)	0·5556 (1)	0·0727
N(1)	0·3012 (2)	0·3823 (3)	0·6403 (2)	0·0476
C(11)	0·3477 (3)	0·4804 (4)	0·6397 (3)	0·0448
C(12)	0·4346 (3)	0·4783 (4)	0·6208 (3)	0·0512
C(13)	0·4799 (3)	0·5683 (5)	0·6218 (3)	0·0625
C(14)	0·4421 (4)	0·6557 (5)	0·6422 (3)	0·0648
C(15)	0·3561 (4)	0·6548 (4)	0·6621 (3)	0·0622
C(16)	0·3085 (3)	0·5665 (4)	0·6601 (3)	0·0559
C(21)	0·3297 (3)	0·3339 (4)	0·7344 (3)	0·0524
C(22)	0·3141 (3)	0·3801 (4)	0·8126 (3)	0·0654
C(23)	0·3415 (4)	0·3352 (5)	0·8981 (4)	0·0870
C(24)	0·3862 (4)	0·2472 (6)	0·9060 (5)	0·0932
C(25)	0·4035 (4)	0·1978 (4)	0·8281 (5)	0·0860
C(26)	0·3736 (3)	0·2429 (4)	0·7391 (4)	0·0705

Table 2. *Interatomic distances* (Å) *and interbond angles* (°)

Al(1)—Cl(1)	2·090 (2)	C(14)—C(15)	1·391 (9)
Al(1)—Cl(2)	2·090 (3)	C(15)—C(16)	1·381 (8)
Al(1)—Cl(3)	2·130 (2)	C(21)—C(22)	1·366 (8)
Al(1)—N(1)	1·983 (4)	C(21)—C(26)	1·381 (8)
N(1)—C(11)	1·490 (7)	C(22)—C(23)	1·363 (9)
N(1)—C(21)	1·492 (7)	C(23)—C(24)	1·350 (11)
C(11)—C(12)	1·398 (7)	C(24)—C(25)	1·390 (11)
C(11)—C(16)	1·360 (8)	C(25)—C(26)	1·412 (9)
C(12)—C(13)	1·385 (8)		
C(13)—C(14)	1·363 (9)		

Cl(1)—Al(1)—Cl(2)	115·86 (11)	C(12)—C(13)—C(14)	121·4 (6)
Cl(1)—Al(1)—Cl(3)	110·38 (10)	C(13)—C(14)—C(15)	119·6 (6)
Cl(1)—Al(1)—N(1)	110·7 (2)	C(14)—C(15)—C(16)	120·5 (5)
Cl(2)—Al(1)—Cl(3)	112·24 (10)	C(11)—C(16)—C(15)	118·9 (5)
Cl(2)—Al(1)—N(1)	106·1 (2)	N(1)—C(21)—C(22)	120·3 (5)
Cl(3)—Al(1)—N(1)	100·32 (15)	N(1)—C(21)—C(26)	117·8 (5)
Al(1)—N(1)—C(11)	117·8 (3)	C(22)—C(21)—C(26)	121·9 (5)
Al(1)—N(1)—C(21)	114·1 (3)	C(21)—C(22)—C(23)	119·5 (6)
C(11)—N(1)—C(21)	110·5 (4)	C(22)—C(23)—C(24)	120·4 (7)
N(1)—C(11)—C(12)	116·6 (4)	C(23)—C(24)—C(25)	121·8 (7)
N(1)—C(11)—C(16)	121·2 (5)	C(24)—C(25)—C(26)	118·0 (6)
C(12)—C(11)—C(16)	122·2 (5)	C(21)—C(26)—C(25)	118·3 (6)
C(11)—C(12)—C(13)	117·6 (5)		

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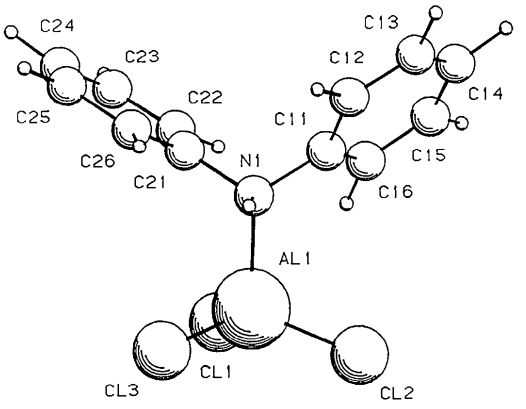

Fig. 1. View of diphenylamine–trichloroaluminium(III) (1/1).

lengths and angles.* A view (Davies, 1983) of the molecular unit is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters, torsion angles and hydrogen positions, and a view of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53657 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The structural features of $Ph_2NH.AlCl_3$ are consistent with those of $Me_3N.AlCl_3$ (Grant, Killean & Lawrence, 1969) and $(Me_2NH)_2.AlCl_3$ (Ahmed, Schwarz, Weidlein & Hess, 1977).

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Structure of DL-Dichloro(*trans*-1,2-diamino-*trans*-3,6-cyclohexanediol)platinum(II) Monohydrate, $[PtCl_2(C_6H_{14}N_2O_2)].H_2O$

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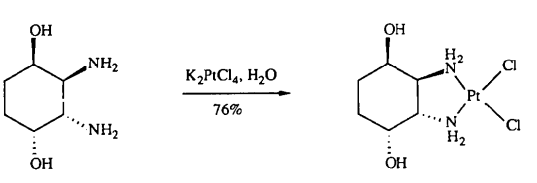
Abstract. $M_r = 430{\cdot}19$, monoclinic, $P2_1/c$, $a = 10{\cdot}919\ (7)$, $b = 10{\cdot}520\ (2)$, $c = 9{\cdot}789\ (4)$ Å, $\beta = 103{\cdot}26\ (4)°$, $V = 1094{\cdot}5$ Å^3, $Z = 4$, $D_x = 2{\cdot}610$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Cu $K\alpha$) = 13·43 mm^{-1}, $F(000) = 808$, $T = 293$ K, $R = 0{\cdot}0310$, $wR = 0{\cdot}0326$ for 1655 observed reflections. The title *trans*-(diaminocyclohexane)platinum complex is of biological interest as an antitumor agent. We resorted to X-ray analysis to establish the configuration of the substituents. A water molecule is hydrogen bonded to one of the hydroxyls.

Experimental. The reaction of *trans*-1,2-diamino-*trans*-3,6-dihydroxycyclohexane (1) with potassium tetrachloroplatinate(II) in water gave the platinum complex (2) in 76% yield (Hanessian & Wang, 1990) which was subjected to X-ray analysis.



(1) (2)

Crystal of $C_6H_{14}Cl_2N_2O_2Pt.H_2O$, bounded by {100}, {010}, {001} and with dimensions 0·04 × 0·15 × 0·19 mm, was obtained from water. Unit-cell dimensions from 25 well centered reflections in the

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