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Bis-adduct of trimethylamine with aluminium trichloride

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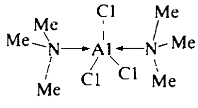
Abstract

In the molecule of trichlorobis(trimethylamine)aluminium(III), $[AlCl_3(C_3H_9N)_2]$, the metal centre is five-coordinate and the two N atoms occupy the axial positions of the trigonal bipyramid. The arrangement of the substituents bonded to Al and N is staggered.

Comment

The reaction of $AlMe_{3-n}Cl_n$ with one mole equivalent of Me_3N results in the corresponding Lewis acid–base complexes $Me_3N \rightarrow AlMe_{3-n}Cl_n$ ($n \leq 3$; Gelbrich *et al.*, 1999). The analogous reaction carried out with the basic materials in a 1:2 molar ratio may lead to the appropriate bis-adducts, such as the title compound $(Me_3N)_2AlCl_3$, (I).



$$Me_3N \rightarrow AlCl_3 \leftarrow NMe_3$$

(I)

Adduct (I) crystallizes in space group *Pnma* with four formula units per unit cell. The Al and N atoms, one Cl ligand and two methyl substituents occupy special positions on a mirror plane. As observed for the $Me_3N \rightarrow AlMe_{3-n}Cl_n$ series of mono-adducts, the arrangement of the halogen and methyl substituents with respect to the donor–acceptor bond is staggered (Gelbrich *et al.*, 1999). Analysing the coordination of the metal atom, the state of transition along the Berry pseudo-rotational pathway from trigonal bipyramidal to square pyramidal was found to be 3.8%, with a small difference of 5.4° from true Berry pseudo-rotational geometry (Holmes, 1984). Therefore, the polyhedron around aluminium is a slightly distorted trigonal bipyramid with axial N atoms. Within the equatorial $AlCl_3$ moiety, the sum of the Cl—Al1—Cl angles is 360.00 (4)°. The axial–axial N1—Al1—N2 angle is 178.76 (5)°. The increased coordination number of the metal atom results in a lengthening of Al—Cl by approximately 0.05 Å compared with the corresponding distance in the mono-adduct. The N→Al donor–acceptor bonds are nearly equal and, resulting from the higher coordination number of aluminium, are longer by 0.211 (5) Å than in $Me_3N \rightarrow AlCl_3$. As expected, the geometries around the N1 and N2 atoms and the N—C distances correspond closely in both complexes. Comparison of the N—Al distance with the values reported for $(Me_3N)_2AlH_3$ [2.18 (1) Å; Heitsch *et al.*, 1963] and for $[\{(C_5H_5)(C_5H_4CH_2NMe_2)\}Fe]_2AlH_3$ [2.168 (2) and 2.178 (2) Å; Nlate *et al.*, 1997] indicate that $AlCl_3$ is a slightly better acceptor than AlH_3. Somewhat surprisingly, the mean N→Al distances of 2.058 (3) and 2.037 (4) Å reported for the bis-adducts of $AlCl_3$ with Me_2HN (Ahmed *et al.*, 1977) and $^iPrH_2N$ (Chang *et al.*, 1997), respectively, are considerably shorter than those in (I). In connection with this, it is worth noting that, on the basis of the dissociation enthalpies of their mono-adducts with $AlMe_3$ (30 and 31 kcal mol^{-1}), the abilities of Me_3N and NMe_2H to act as electron donors have been considered to be equal (Hendrickson *et al.*, 1968).

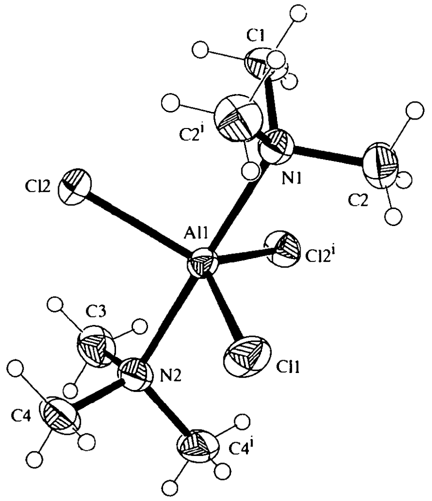


Fig. 1. The molecular structure of (I), with displacement ellipsoids at the 50% probability level. [Symmetry code: (i) $x, \frac{1}{2} - y, z$.]

The intermolecular distances indicate van der Waals-type interactions. Unit-cell parameters and the space group of (I), obtained from Weissenberg photographs at room temperature, have been reported earlier and from this, structural similarities with the chromium analogue were assumed (Beattie *et al.*, 1969). Moreover, adduct (I) is an example of a series of isostructural complexes, $(Me_3E)_2MX_{3-n}Y_n$ (E = P, N, As; M = Al, In, transition metal; X,Y = halogen, –CO, –C═O; $n \leq 3$), which will be the subject of a further contribution.

Experimental

All operations were carried out under an atmosphere of dry nitrogen using standard Schlenk techniques. Aluminium trichloride (23.4 g, 175 mmol) was dissolved in diethyl ether (200 ml). Trimethylamine (21.2 g, 360 mmol) was condensed into the solution at 253 K and the resulting solution was stirred for 1 h after which diethyl ether was removed. The residue was extracted with *n*-pentane. Crystals of (I) suitable for X-ray analysis were obtained by recrystallization from *n*-pentane at 278 K.

Crystal data

[$AlCl_3(C_3H_9N)_2$]
$M_r = 251.55$
Orthorhombic
Pnma
$a = 9.645\ (8)$ Å
$b = 9.994\ (2)$ Å
$c = 12.918\ (6)$ Å
$V = 1245.2\ (12)$ Å^3
$Z = 4$
$D_x = 1.342$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 80 reflections
$\theta = 10.3$–$19.3°$
$\mu = 0.765$ mm^{-1}
$T = 203\ (2)$ K
Platelet
0.45 × 0.40 × 0.40 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω–θ scans
Absorption correction: none
3832 measured reflections
1916 independent reflections
1666 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.014$

$\theta_{\rm max} = 30.05°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 120 min
intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.059$
$S = 1.053$
1916 reflections
103 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.1290P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.220$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.208$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0179 (15)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Al1—N1	2.1580 (16)	Al1—Cl1	2.1676 (10)
Al1—N2	2.1662 (16)	Al1—Cl2	2.1725 (5)
N1—Al1—N2	178.76 (5)	N2—Al1—Cl2	90.21 (3)
N1—Al1—Cl1	89.10 (6)	Cl1—Al1—Cl2	121.075 (16)
N2—Al1—Cl1	89.67 (7)	Cl2—Al1—Cl2$^{\rm i}$	117.85 (3)
N1—Al1—Cl2	90.42 (3)		

Symmetry code: (i) $x, \frac{1}{2} - y, z$.

Refined C—H distances are in the range 0.929 (19)–0.972 (13) Å.

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXTL/PC* (Siemens, 1990). Software used to prepare material for publication: *PLATON* (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1327). Services for accessing these data are described at the back of the journal.

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