C21	0.1948 (3)	−0.1174 (4)	0.0055 (2)	0.0287 (9)
C22	0.2689 (3)	−0.2034 (5)	0.0335 (3)	0.0402 (11)
C23	0.2545 (4)	−0.3162 (5)	0.0611 (3)	0.0456 (12)
C24	0.1655 (4)	−0.3455 (4)	0.0607 (3)	0.0434 (12)
C25	0.0913 (4)	−0.2625 (5)	0.0332 (3)	0.0455 (12)
C26	0.1056 (3)	−0.1497 (4)	0.0059 (2)	0.0342 (10)
C27	0.2621 (5)	−0.0326 (5)	−0.1020 (3)	0.0458 (13)
C28	0.0957 (4)	0.0790 (5)	−0.0943 (3)	0.0411 (11)
C31	0.3806 (3)	0.0369 (4)	0.2358 (2)	0.0325 (10)
C32	0.4451 (4)	0.0785 (5)	0.3054 (3)	0.0521 (14)
C33	0.5116 (4)	−0.0019 (6)	0.3558 (3)	0.061 (2)
C34	0.5142 (4)	−0.1222 (6)	0.3361 (3)	0.060 (2)
C35	0.4521 (5)	−0.1652 (5)	0.2672 (3)	0.0579 (15)
C36	0.3854 (4)	−0.0860 (5)	0.2176 (3)	0.0475 (13)
C37	0.1760 (4)	0.0676 (6)	0.1578 (3)	0.0483 (13)
C38	0.2914 (5)	0.2751 (5)	0.2199 (3)	0.0502 (14)

Table 2. *Selected geometric parameters (Å, °)*

Tc—N	2.104 (3)	Tc—P1	2.4784 (13)
Tc—Cl2	2.3216 (13)	Tc—P3	2.4823 (14)
Tc—Cl1	2.3449 (13)	N—C1	1.142 (5)
Tc—P2	2.4430 (12)	S—C1	1.624 (4)
N—Tc—Cl2	91.29 (10)	P2—Tc—P1	94.79 (4)
N—Tc—Cl1	91.25 (10)	N—Tc—P3	84.14 (9)
Cl2—Tc—Cl1	177.21 (4)	Cl2—Tc—P3	93.96 (4)
N—Tc—P2	176.32 (9)	Cl1—Tc—P3	85.16 (4)
Cl2—Tc—P2	86.84 (5)	P2—Tc—P3	99.14 (4)
Cl1—Tc—P2	90.69 (5)	P1—Tc—P3	165.16 (4)
N—Tc—P1	82.09 (9)	C1—N—Tc	171.3 (3)
Cl2—Tc—P1	91.88 (4)	N—C1—S	179.1 (4)
Cl1—Tc—P1	89.61 (4)		

Scattering factors, dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography* (1992, Vol. C, Tables 6.1.1.4, 4.2.6.8 and 4.2.4.2).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*, *SDP* (Frenz, 1983), *HKL* (Kretschmar, 1994). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trimethyl(1-methyl-4-phenylpiperazine-*N*¹)aluminium

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Abstract

The X-ray single-crystal structure determination of the title compound, $[Al(CH_3)_3(C_{11}H_{16}N_2)]$, reveals that the Al atom is four-coordinate in a distorted tetrahedral environment and is bonded to the N atom of the piperazine molecule carrying the methyl group, with an Al—N bond length of 2.050 (2) Å. The piperazine ring adopts a chair conformation.

Comment

Alkylaluminium compounds have been used extensively as one of the major precursors in the preparation of semiconductor compounds by metal–organic chemical vapour deposition (MOCVD). Recently, there has been increasing interest in the preparation of alkylaluminium complexes with N or P donor ligands, which are either used directly as MOCVD precursors or applied in

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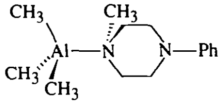
the ultra-purification of trialkylaluminium compounds (Bradley, Chudzynska & Frigo, 1988; Foster, Rushworth, Cole-Hamilton, Jones & Stagg, 1988; Bradley *et al.*, 1988; Jones, Wright, Oliver & Cockayne, 1990; Looney & Parkin, 1990). In order to obtain a trialkylaluminium complex of suitable stability and volatility, we embarked on the present research and the title compound, (1), was studied by X-ray diffraction.



(1)

The crystal structure of (1) is presented in Fig. 1. Although there are two N atoms in the donor ligand, the Al atom is coordinated to only one of them [N(1)]. The coordinative capability of alkylamines should reasonably be greater than that of aromatic amines. The Al atom is four-coordinate in a distorted tetrahedral environment. The C—Al—N bond angles [103.6 (1)–105.4 (1)°] are consistently 9–10° smaller than the C—Al—C angles [113.6 (2)–115.1 (2)°], implying steric repulsion between the methyl groups on the Al atom and rather weak Al—N bonding. The N—Al bond length is 2.050 (2) Å, which agrees well with the value reported for trimethyl(quinuclidine)aluminium (Whitt, Parker & Atwood, 1971). The six-membered piperazine ring has a chair form, with the N(1)—Al and N(2)—C(5) bonds in equatorial directions. The bond angles C(2)—N(2)—C(5) and C(3)—N(2)—C(5) are around 117° and the bond angle C(2)—N(2)—C(3) [112.0 (2)°] is much larger than that of C(1)—N(1)—C(4) [106.5 (2)°], implying a near sp^2-hybrid orbital for atom N(2); this accounts for the strong conjugation of electrons between the N atom and the aromatic ring and also the weak coordinative activity of the N(2) atom.

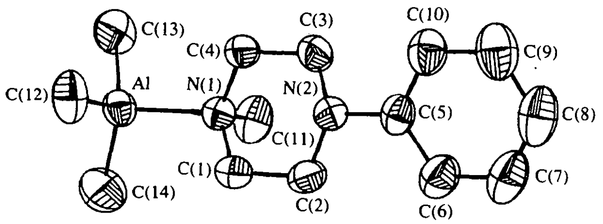


Fig. 1. The molecular structure of $[Me_3Al\{N(CH_3)(CH_2CH_2)_2NPh\}]$ showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

Experimental

All manipulations were performed in a glove box under purified nitrogen. Solvents were heated under reflux with sodium benzophenone and distilled under nitrogen prior to use. 1-Methyl-4-phenylpiperazine (1.76 g, 10 mmol) was dissolved in a mixture of benzene (5 ml) and petroleum ether (333–363 K, 1 ml). To this solution was added dropwise a solution of $AlMe_3$ (2 ml, 20 mmol) in benzene (10 ml) at room temperature. The mixture was then stirred at 323 K for 2 h. After removing about half of the solvent mixture and cooling, the precipitates were filtered off and dried under vacuum to give the pure title product (1.1 g, 43% yield). Crystals suitable for analysis were obtained by recrystallization from benzene.

Crystal data

$[Al(CH_3)_3(C_{11}H_{16}N_2)]$
$M_r = 248.35$
Monoclinic
$P2_1/c$
$a = 10.130 (5)$ Å
$b = 9.107 (2)$ Å
$c = 17.620 (4)$ Å
$\beta = 106.57 (3)°$
$V = 1558.1 (9)$ Å^3
$Z = 4$
$D_x = 1.06$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.82–14.82°$
$\mu = 0.110$ mm^{-1}
$T = 296$ K
Rectangular block
1.0 × 0.65 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3014 measured reflections
2939 independent reflections
1963 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.011$
$\theta_{max} = 25°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 20$
3 standard reflections monitored every 300 reflections
intensity decay: 35.8%

Refinement

Refinement on F
$R = 0.055$
$wR = 0.067$
$S = 1.69$
1963 reflections
230 parameters
Only coordinates of H atoms refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.16$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Al	0.28777 (8)	0.2117 (1)	0.02968 (5)	4.20 (4)
N(1)	0.3263 (2)	0.3587 (2)	0.1215 (1)	3.53 (8)
N(2)	0.2119 (2)	0.5718 (2)	0.2107 (1)	3.8 (1)
C(1)	0.2483 (3)	0.3168 (3)	0.1785 (2)	4.1 (1)
C(2)	0.2551 (3)	0.4272 (3)	0.2431 (2)	4.4 (1)
C(3)	0.2864 (4)	0.6191 (3)	0.1552 (2)	4.6 (1)
C(4)	0.2774 (3)	0.5077 (3)	0.0917 (2)	4.0 (1)
C(5)	0.1897 (3)	0.6788 (3)	0.2633 (2)	3.9 (1)
C(6)	0.2022 (3)	0.6480 (4)	0.3430 (2)	5.2 (1)
C(7)	0.1760 (4)	0.7543 (5)	0.3919 (2)	6.2 (2)
C(8)	0.1373 (3)	0.8914 (5)	0.3655 (2)	6.4 (2)
C(9)	0.1255 (3)	0.9256 (4)	0.2883 (2)	6.0 (2)
C(10)	0.1517 (3)	0.8203 (4)	0.2377 (2)	4.9 (1)

C(11)	0.4778 (3)	0.3602 (5)	0.1611 (2)	5.8 (2)
C(12)	0.0872 (4)	0.2203 (5)	−0.0201 (2)	6.4 (2)
C(13)	0.3970 (4)	0.2872 (5)	−0.0376 (3)	6.2 (2)
C(14)	0.3508 (6)	0.0245 (5)	0.0819 (3)	8.0 (3)

Table 2. *Selected geometric parameters (Å, °)*

Al—C(14)	1.956 (5)	Al—C(13)	1.962 (4)
Al—C(12)	1.971 (4)	Al—N(1)	2.050 (2)
N(1)—C(4)	1.488 (3)	N(1)—C(1)	1.494 (3)
N(1)—C(11)	1.494 (4)	N(2)—C(5)	1.405 (3)
N(2)—C(3)	1.461 (4)		
C(14)—Al—C(13)	115.1 (2)	C(14)—Al—C(12)	113.6 (2)
C(14)—Al—N(1)	103.6 (2)	C(13)—Al—C(12)	113.9 (2)
C(13)—Al—N(1)	103.6 (1)	C(12)—Al—N(1)	105.4 (1)
C(4)—N(1)—C(1)	106.5 (2)	C(4)—N(1)—C(11)	110.5 (3)
C(4)—N(1)—Al	110.3 (2)	C(1)—N(1)—C(11)	110.0 (2)
C(1)—N(1)—Al	110.2 (2)	C(11—N(1)—Al	108.3 (2)

The single crystal was sealed in a glass capillary under nitrogen. *CAD*-4 *Software* (Enraf–Nonius, 1989) was used for data collection and cell refinement. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983) and subsequent Fourier techniques, and refined by full-matrix least-squares methods. The H atoms were found by difference Fourier methods. H-atom coordinates were used for correction of the structure. All calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1989). *ORTEP*II (Johnson, 1976) was used to produce the figure.

This work was supported by the National Natural Science Foundation of China and the Starting Fund for Returned Scholars.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: KH1055). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trifluoromethyl(triphenylphosphine)gold(I)

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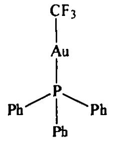
Abstract

The title compound, $[Au(CF_3)\{P(C_6H_5)_3\}]$, is the first crystallographically characterized mononuclear gold(I) complex with an Au—CF_3 bond. The Au coordination is nearly linear with P—Au—C 178.56 (11)°, Au—P 2.2859 (9) and Au—C 2.045 (4) Å.

Comment

The structures of linear gold(I) complexes Ph_3PAuX have attracted much interest with regard to the type of ligand X and the *trans*-positioned Au—P bond (Ahrland, Dreisch, Norén & Oskarsson, 1987; Barron, Engelhardt, Healy, Oddy & White, 1987; Jones & Lautner, 1988). The complexes with X = Cl, Br, I, CN, SCN and $OCOCH_3$ are effectively isostructural with orthorhombic packing, $P2_12_12_1$, whereas the monoclinic complexes with X = ONO_2 and CH_3 differ in their packing. We have investigated the related compound, (I), with X = trifluoromethyl, which, according to a search in the Cambridge Structural Database (Allen *et al.*, 1991), is also the first crystallographically characterized example of a mononuclear complex with an Au^I—CF_3 bond.



(I)

The title molecule shows the expected linear coordination of the Au^I atom with a P—Au—C angle of 178.56 (11)°. The most interesting geometric features are the Au—P and Au—C bond distances. The former, 2.2859 (9) Å, has the same length as those in the complexes with X = CN [2.278 (2) Å; Jones & Lautner, 1988] and X = CH_3 [2.279 (8) Å; Gavens, Guy, Mays & Sheldrick, 1977], although these three ligands differ appreciably in their electronic properties. It has already been stated by Barron *et al.* (1987) that de-