direction of the *a* axis. There are no hydrogen bonds between the columns. The distances between O atoms involved in hydrogen bonding (2·489–2·891 Å) are within the distances accepted for normal hydrogen bonding in crystalline hydrates as reported by Chiari & Ferraris (1982), except the shortest one, which is between O(23*w*) and O(25*w*). As can be seen in Fig. 1, O(23*w*) has a large anisotropic thermal component perpendicular to the mirror plane. This may indicate that there is disorder, by which this molecule is displaced from the mirror plane, so that the actual O(25*w*)—O(23*w*) distance is somewhat longer than the observed average distance. This would also lengthen the short Mo—O(23*w*) distance mentioned above.

The 18-crown-6 in the complex described here lacks the usual crystallographic center of symmetry observed in other complexes and has instead a crystallographic mirror plane. This makes possible coordination to different groups above and below the ring. As mentioned by Elbasyouny *et al.* (1983) no stable binary complex of only water and 18-crown-6 has been found. In other transition metal.H_2O.18-crown-6 complexes (*e.g.* Vance *et al.*, 1980), the coordinating water molecules are part of a positively charged hydrated transition-metal moiety and other negatively charged species are present in the crystal. In the complex described here no charged species are present. Apparently the Mo^{VI} atom makes the H atoms of the coordinating water molecules sufficiently acidic to facilitate hydrogen bonding to the crown. The short distance between O(23*w*) and O(25*w*) may have the same effect on the H atoms of the 'free' water molecule.

Computing costs were provided by the OSU Computer Center.

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***trans*-Bis(*tert*-butylamine)dichloroplatinum(II), $C_8H_{22}Cl_2N_2Pt$**

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Abstract. $M_r = 412·27$, monoclinic, $P2_1/c$, $a = 6·248\ (3)$, $b = 20·569\ (21)$, $c = 10·676\ (9)$ Å, $\beta = 92·27\ (6)°$, $V = 1371\ (2)$ Å^3, $Z = 4$, $D_m = 2·01\ (2)$, $D_x = 1·997$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 11·120$ mm^{-1}, $F(000) = 784$, $T = 295$ K, $R = 0·043$, $R_w = 0·040$ for 2519 unique observed reflections. The coordination around the Pt atom is square planar. The compound, which was synthesized in our laboratory, is the *trans* isomer. The Pt—N bond lengths are 2·041 (6) and 2·053 (6) Å, while the Pt—Cl bond lengths are 2·299 (2) and 2·296 (2) Å. The structure is stabilized by intermolecular hydrogen bonds between the amino groups and the chlorine atoms.

Introduction. Continuing our studies on platinum amine compounds, we have synthesized *trans*-[Pt(*t*-butylamine)$_2$Cl$_2$] and determined its molecular and crystal structure. It was synthesized from the hot aqueous reaction of K_2PtCl_4 with the amine. Because of the bulkiness of the ligand, the *trans* isomer is obtained.

Experimental. *Synthesis*: 1 ml of *tert*-butylamine was added to 0·5 g of K_2PtCl_4 dissolved in 5 ml of hot water. The mixture was heated (~350 K) with stirring until a precipitate appeared and the solution became yellow. The mixture was then evaporated to dryness under vacuum. The residue was washed with water and

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the precipitate collected by filtration. The precipitate was again washed with water and air dried. Crystals of *trans*-[Pt(*t*-butylamine)$_2$Cl$_2$] were obtained by slow evaporation of an acetone solution of the yellow product.

Rectangular plate, dimensions (mm): 0·566 (100–$\bar{1}$00) × 0·170 (001–00$\bar{1}$) × 0·053 (010–0$\bar{1}$0); density by flotation in thallous malonate aqueous solution; precession photographs indicated $P2_1/c$; Syntex $P\bar{1}$ diffractometer; graphite-monochromatized Mo $K\alpha$ radiation; cell parameters from setting angles of 15 centred reflections ($10 < 2\theta < 21°$); 4000 independent reflections measured, $2\theta < 60°$, θ–2θ scan technique; 3 standard reflections (400, 041, 002): variations <2%; reflections with $I_{\rm net} < 2·5\sigma(I)$ unobserved, $\sigma(I)$ calculated as in Melanson, Hubert & Rochon (1975); absorption correction based on equations of crystal faces, transmission factors: 0·173 to 0·553; data corrected for Lorentz and polarization effects; 2519 unique observed reflections; atomic scattering factors of Cromer & Waber (1965) for Pt, Cl, N, C and of Stewart, Davidson & Simpson (1965) for H; anomalous-dispersion terms of Pt and Cl from Cromer (1965). Patterson map indicated position of Pt; positions of other atoms (except H) obtained by structure factor and Fourier map calculations; isotropic secondary-extinction correction (Coppens & Hamilton, 1970); anisotropic refinement on F; $w = 1/\sigma^2(F)$, H in $-NH_2$ fixed at calculated positions (N–H = 0·85 Å) with isotropic $B = 6·0$ Å^2. H in $-CH_3$ could not be located. Max. Δ/σ in final refinement cycle: <0·1; $\rho_{\rm max} = 0·82$ (close to Pt), $\rho_{\rm min} = -0·5$ e Å^{-3} in final difference Fourier synthesis. $R = 0·043$ and $R_w = 0·040$,* calculation on Cyber 171 computer with programs of Melanson, Hubert & Rochon (1975).

Discussion. The refined atomic parameters are listed in Table 1. A labelled diagram of the molecule is shown in Fig. 1. The coordination around the Pt atom is square planar. The deviations from the weighted coordination plane are: Pt −0·0005 (3); Cl(1) 0·020 (2); Cl(2) 0·019 (2); N(1) −0·024 (6); N(2) −0·023 (6) Å. The bond lengths and angles are given in Table 2. The angles around the Pt atom are close to the expected values of 90 and 180°.

As expected from the infrared spectroscopic results, the compound is the *trans* isomer. Because of the bulkiness of *tert*-butylamine, the *cis* isomer, which is usually obtained under the conditions used for the preparation of the compound, is unstable. Only the *trans* isomer is obtained. The infrared spectrum of the compound showed only one stretching Pt–Cl vibration at 322 cm^{-1}, which is typical of a *trans* Pt compound. The ν(N–H) vibrations appeared at 3250(*sh*), 3240(*vs*), 3208(*vs*) and 3128(*vs*) cm^{-1}, while the δ(NH$_2$) deformation absorbed at 1574(*vs*) cm^{-1}.

Table 1. *Positional parameters* (×10^4) *with their e.s.d.'s and temperature factors* (Å^2 × 10^3)

	x	y	z	$U_{\rm eq}$†
Pt	1556·6 (4)	2406·6 (1)	1338·7 (2)	35
Cl(1)	4125 (3)	2784 (1)	37 (2)	55
Cl(2)	−1031 (3)	2019 (1)	2612 (2)	52
N(1)	−461 (9)	3129 (3)	718 (5)	42
N(2)	3593 (9)	1692 (3)	2004 (5)	39
C(1)	−586 (15)	3786 (5)	1358 (8)	57
C(2)	1707 (15)	4064 (6)	1495 (11)	89
C(3)	−2002 (16)	4226 (5)	510 (10)	72
C(4)	−1605 (20)	3682 (6)	2646 (9)	94
C(5)	3745 (14)	1030 (5)	1401 (8)	55
C(6)	4818 (19)	1127 (5)	127 (9)	81
C(7)	5119 (16)	598 (5)	2308 (10)	71
C(8)	1476 (16)	747 (5)	1213 (11)	85

† $U_{\rm eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Bond distances* (Å) *and angles* (°)

Pt–Cl(1)	2·299 (2)	C(1)–C(2)	1·544 (13)
Pt–Cl(2)	2·296 (2)	C(1)–C(3)	1·536 (14)
Pt–N(1)	2·041 (6)	C(1)–C(4)	1·553 (14)
Pt–N(2)	2·053 (6)	C(5)–C(6)	1·553 (14)
N(1)–C(1)	1·519 (11)	C(5)–C(7)	1·548 (14)
N(2)–C(5)	1·511 (11)	C(5)–C(8)	1·538 (13)
Cl(1)–Pt–Cl(2)	179·0 (1)	N(1)–C(1)–C(4)	107·8 (7)
Cl(1)–Pt–N(1)	89·7 (2)	N(2)–C(5)–C(6)	107·1 (7)
Cl(1)–Pt–N(2)	90·7 (2)	N(2)–C(5)–C(7)	107·1 (7)
Cl(2)–Pt–N(1)	90·3 (2)	N(2)–C(5)–C(8)	108·8 (7)
Cl(2)–Pt–N(2)	89·3 (2)	C(2)–C(1)–C(3)	110·4 (8)
N(1)–Pt–N(2)	178·6 (2)	C(2)–C(1)–C(4)	112·2 (8)
Pt–N(1)–C(1)	122·9 (5)	C(3)–C(1)–C(4)	110·9 (8)
Pt–N(2)–C(5)	123·1 (5)	C(6)–C(5)–C(7)	112·0 (8)
N(1)–C(1)–C(2)	108·0 (7)	C(6)–C(5)–C(8)	111·2 (8)
N(1)–C(1)–C(3)	107·4 (7)	C(7)–C(5)–C(8)	110·5 (8)

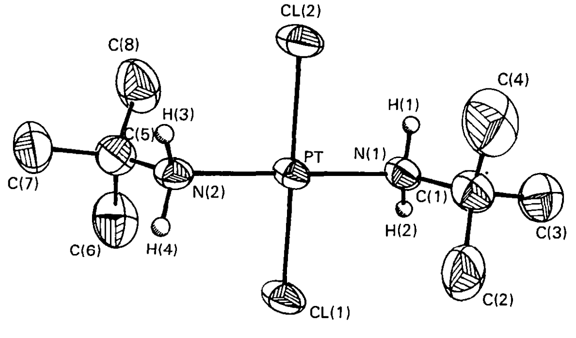

Fig. 1. Labelled diagram of the molecule *trans*-[Pt{NH$_2$–C(CH$_3$)$_3$}$_2$Cl$_2$]. The ellipsoids correspond to 50% probability.

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* Lists of structure factors, anisotropic thermal parameters H-atom parameters and hydrogen-bonding data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39867 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The Pt—Cl bonds [2·299 (2) and 2·296 (2) Å] and the Pt—N bonds [2·041 (6) and 2·053 (6) Å] are normal and agree well with published values found in *trans*-aminochloroplatinum(II) compounds (Rochon, Kong & Melanson, 1984; Lock & Zvagulis, 1981). The two *trans* organic ligands are arranged so that one ligand is above the platinum plane, while the second is below the plane. The whole molecule has an approximate centre of symmetry (Figs. 1 and 2).

The structure of the organic ligands is normal. All the angles are close to the tetrahedral values (107–112°). The angles around the bonded N atoms [Pt—N—C = 122·9 (5) and 123·1 (5)°] are quite far from the tetrahedral value, but similar values (113–122°) have been observed for these types of compounds (Rochon, Kong & Melanson, 1984; Lock & Zvagulis, 1981; Rochon & Melanson, 1982).

A projection down the x axis of the packing of the molecules is shown in Fig. 2. The crystal consists of layers of molecules parallel to the ac plane. The layers are held together in the **b** direction by van der Waals forces between the $-CH_3$ groups. Inside the layers, the structure is stabilized by intermolecular hydrogen bonding between the amino groups and the chlorine atoms. The N···Cl distances vary from 3·335 (6) to 3·504 (6) Å and the C—N···Cl angles from 100·3 (4) to 133·1 (5)°.

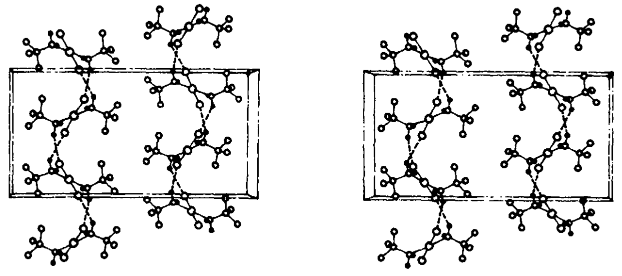

Fig. 2. Stereoscopic diagram of the packing in the *trans*-$[Pt\{NH_2-C(CH_3)_3\}_2Cl_2]$ crystal (b axis horizontal and c axis vertical).

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1,1,1,2,2,2,3,3,3-Nonacarbonyl-μ_3-cyclopropylmethylidyne-*triangulo*-tricobalt, $(\mu_3\text{-}c\text{-}C_3H_5C)Co_3(CO)_9$

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Abstract. $M_r = 481·98$, triclinic, $P\bar{1}$, $a = 7·8772\ (6)$, $b = 14·3778\ (17)$, $c = 15·7851\ (16)$ Å, $\alpha = 103·700\ (9)$, $\beta = 100·877\ (7)$, $\gamma = 99·365\ (8)°$, $V = 1664·9\ (7)$ Å^3, $Z = 4$, $D_x = 1·923$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 29·93$ cm^{-1}, $F(000) = 944$, $T = 296$ K, $R = 2·7\%$, $R_w = 3·6\%$, $R_{all} = 3·7\%$, $S = 1·67$ for 3691 observations with $I > 3\sigma(I)$. The final structure clearly shows the staggering of the Co—Co—Co and C—C—C rings in a nearly threefold symmetric environment. There are no intermolecular contacts shorter than 2·70 Å.

Introduction. A recently developed and important class of organometallic clusters is the alkylidynetricobalt nonacarbonyl series (Seyferth, 1976). Such compounds have been shown to exhibit interesting reactivity (Palyi, Piacenti & Marko, 1970; Seyferth, 1976; Seidler, Bryndza, Frommer, Stuhl & Bergman, 1983; and references therein), and have even found use as catalyst precursors for certain transformations (Seyferth & Withers, 1983).

Sutton & Dahl (1967) first structurally characterized a member of this class using film data; more recently,

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