

As shown in Fig. 2, the Ni(1)–F(1) and Ni(2)–F(2) bonds are tilted 4.8 and 5.6° from the normal to each NiN<sub>4</sub> plane so as to minimize the steric interaction with the axial methyl groups of C(7) and C(15), respectively.

The calculations were carried out on the HITAC M-180 computer at the Computer Center of the Institute for Molecular Science with the *Universal Crystallographic Computation Program System UNICS III* (Sakurai & Kobayashi, 1979). The TG and DSC curves were measured on a Rigaku DSC-8001, SL/C.

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### *trans*-Dichlorobis(cycloheptylamine)platinum(II)

BY J. P. BRADFORD, R. FAGGIANI AND C. J. L. LOCK

*Institute for Materials Research, McMaster University, Hamilton, Ontario, Canada L8S 4M1*

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**Abstract.** [Pt(C<sub>7</sub>H<sub>13</sub>NH<sub>2</sub>)<sub>2</sub>Cl<sub>2</sub>], triclinic,  $P\bar{1}$ ,  $Z = 2$ ,  $a = 11.445$  (3),  $b = 13.651$  (3),  $c = 5.828$  (1) Å,  $\alpha = 93.93$  (2),  $\beta = 102.55$  (2),  $\gamma = 96.78$  (2)°,  $V = 878.5$  (6) Å<sup>3</sup>,  $D_c = 1.75$ ,  $D_o = 1.73$  (2) Mg m<sup>-3</sup>. The structure was determined by heavy-atom methods and refined to  $R = 0.033$ ,  $R_w = 0.054$  based on 1838 independent reflections. The complex is *trans* even though it was prepared by the method described for the *cis* complex and bond lengths and bond angles are normal. The crystal is held together by hydrogen bonds and van der Waals forces.

**Introduction.** The crystal was prepared by the method supposed to give *cis*-dichlorobis(cycloheptylamine)-platinum(II) (Braddock, Connors, Jones, Khokhar, Melzack & Tobe, 1975; Connors, Jones, Ross, Braddock, Khokhar & Tobe, 1972). The compound was crystallized from acetone. The pale-yellow crystals were thick plates. A crystal, which was homogeneous under the polarizing microscope, was cut to a rough needle and ground into a cylinder of dimensions  $r = 0.075$ ,  $l = 0.25$  mm. Photographs suggested the crystal was triclinic and a Delaunay reduction showed no hidden symmetry. Since  $Z = 2$  the space group  $P\bar{1}$  was assumed and confirmed by the successful solution of the structure. The cell was determined by a least-squares fit of 15 well centred reflections ( $20^\circ < 2\theta < 34^\circ$ ) on a Syntex P2<sub>1</sub> diffractometer, and the density

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was determined by flotation in an iodoethane–diiodo-methane mixture. The intensities of 1838 independent reflections up to  $2\theta = 45^\circ$  were measured with Mo  $K\alpha$  radiation. The computer-controlled Syntex P2<sub>1</sub> diffractometer was operated in a  $2\theta(\text{counter})-\theta(\text{crystal})$  scan mode with a graphite monochromator. Reflections for which  $I \leq 0$  were not considered in data treatment. This left 1487 observed [ $I > 3\sigma(I)$ ] reflections which were used for the structure determination; 340 were considered unobserved [ $3\sigma(I) > I > 0$ ] and were given no weight in the refinement unless  $|F_o| > |F_c|$ . The method of data treatment has been described previously (Hughes, Krishnamachari, Lock, Powell & Turner, 1977; Lippert, Lock, Rosenberg & Zvagulis, 1977). Correction was made for absorption ( $\mu = 8.322$  mm<sup>-1</sup>,  $A^*$  limits 2.92 to 2.43) and extinction (Larson, 1967;  $g = 1.94 \times 10^{-7}$ ). The stability of the experimental system was monitored by a standard reflection (231) after every 49 reflections; it showed an e.s.d. of 1.24% with no time variation.

Initially the Patterson map was solved incorrectly, because of the preconceived idea that we were dealing with the *cis* complex. The Pt atom was placed at  $\frac{1}{4}, 0, 0$  and electron density difference maps revealed other atoms in some peculiar geometric arrangements, including six atoms sufficiently close to Pt to be considered coordinated. The discovery that the reported structure of supposedly *cis*-dichlorobis(cyclohexyl-

Table 1. Atomic positional parameters ( $\times 10^3$ ) and temperature factors ( $\text{\AA}^2 \times 10^3$ )

	x	y	z	$U_{\text{iso}}$ or $U_{\text{eq}}^*$
Pt(1)	0	0	0	31.6 (5)*
Cl(1)	154.0 (3)	43.8 (2)	329.3 (4)	45 (2)*
N(1)	-120.0 (8)	64.1 (6)	161 (1)	42 (2)
C(1)	-86 (1)	172.8 (8)	270 (2)	42 (3)
C(2)	-189 (1)	195.6 (1)	389 (2)	60 (3)
C(3)	-184 (1)	303 (1)	483 (3)	94 (5)
C(4)	-207 (2)	376 (1)	319 (3)	114 (6)
C(5)	-107 (2)	415 (1)	190 (3)	96 (5)
C(6)	-16 (1)	342 (1)	155 (3)	82 (4)
C(7)	-68 (1)	237 (1)	72 (2)	57 (3)
Pt(2)	500	0	0	34.0 (5)*
Cl(2)	402.9 (3)	-112.2 (2)	197.6 (4)	45 (2)*
N(2)	450.8 (8)	106.4 (7)	215 (1)	46 (2)
C(11)	425 (1)	202.7 (8)	125 (2)	42 (3)
C(12)	315 (1)	182.7 (8)	-85 (2)	53 (3)
C(13)	244 (1)	272 (1)	-157 (2)	76 (4)
C(14)	322 (1)	371 (1)	-132 (3)	93 (5)
C(15)	344 (1)	426 (1)	116 (3)	86 (5)
C(16)	442 (1)	382 (1)	299 (2)	65 (4)
C(17)	409 (1)	271.6 (8)	334 (2)	56 (3)

$$* U_{\text{eq}} = \frac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{12} \cos \gamma + 2U_{13} \cos \beta + 2U_{23} \cos \alpha).$$

Table 2. Selected interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

Pt(1)—Cl(1)	2.296 (2)	Pt(2)—Cl(2)	2.303 (3)
Pt(1)—N(1)	2.065 (9)	Pt(2)—N(2)	2.059 (9)
N(1)—C(1)	1.55 (1)	N(2)—C(11)	1.49 (2)
C(1)—C(2)	1.54 (2)	C(11)—C(12)	1.54 (1)
C(2)—C(3)	1.54 (2)	C(12)—C(13)	1.58 (2)
C(3)—C(4)	1.43 (3)	C(13)—C(14)	1.51 (2)
C(4)—C(5)	1.56 (3)	C(14)—C(15)	1.54 (2)
C(5)—C(6)	1.57 (3)	C(15)—C(16)	1.58 (2)
C(6)—C(7)	1.50 (2)	C(16)—C(17)	1.55 (2)
C(7)—C(1)	1.53 (2)	C(17)—C(11)	1.54 (2)

## Possible hydrogen bonds

Cl(1)—N(1) <sup>(a)</sup>	3.48 (1)	Cl(2)—N(2) <sup>(b)</sup>	3.46 (1)
Cl(1)—Pt(1)—N(1)	91.5 (2)	Cl(2)—Pt(2)—N(2) <sup>(c)</sup>	94.7 (3)
Pt(1)—N(1)—C(1)	118.7 (6)	Pt(2)—N(2)—C(11)	119.7 (7)
N(1)—C(1)—C(2)	105.4 (8)	N(2)—C(11)—C(12)	108.2 (8)
C(1)—C(2)—C(3)	115 (1)	C(11)—C(12)—C(13)	117.6 (9)
C(2)—C(3)—C(4)	119 (1)	C(12)—C(13)—C(14)	115 (1)
C(3)—C(4)—C(5)	119 (2)	C(13)—C(14)—C(15)	113 (1)
C(4)—C(5)—C(6)	116 (1)	C(14)—C(15)—C(16)	111 (1)
C(5)—C(6)—C(7)	116 (1)	C(15)—C(16)—C(17)	115 (1)
C(6)—C(7)—C(1)	114 (1)	C(16)—C(17)—C(11)	111 (1)
C(7)—C(1)—C(2)	115 (1)	C(17)—C(11)—C(12)	115 (1)
C(7)—C(1)—N(1)	107.5 (8)	C(17)—C(11)—N(2)	107.8 (9)

Symmetry code: the atoms are related to those given in Table 1 by the transformations; (a)  $-x, -y, 1 - z$ ; (b)  $1 - x, -y, 1 - z$ ; (c)  $1 - x, -y, -z$ .

amine)platinum(II) (Iball & Scrimgeour, 1977) is an incorrect solution of the structure of the *trans* complex (Lock, Speranzini & Zvagulis, 1980) led us to apply the same treatment to the title compound. When two

independent Pt atoms were placed at 0,0,0 and  $\frac{1}{2},0,0$ , difference maps revealed all the remaining non-hydrogen atoms and full-matrix least-squares refinement, using anisotropic temperature factors for Pt and Cl and minimizing  $\sum w(|F_o| - |F_c|)^2$  with 96 variables, proceeded smoothly to final values of  $R = 0.029$  (0.033) and  $R_w = 0.052$  (0.054) for the observed (all) reflections with a final maximum shift/error of 0.05. The weighting scheme applied was  $1/w = \sigma_F^2 + (0.04F_o)^2$ ; scattering factors were taken from Cromer & Waber (1974) and correction for anomalous scattering was applied to Pt and Cl (Cromer, 1974). A final difference map was essentially featureless, the highest peak being  $0.61 \text{ e \AA}^{-3}$  at 0.05, 0.05, 0.05 and the deepest valley being  $-0.55 \text{ e \AA}^{-3}$  at 0.80, 0.35, 0.40. The final positional parameters and isotropic temperature factors of all atoms are given in Table 1.† Selected interatomic distances and angles are given in Table 2. All calculations were carried out on CDC 6400 or Cyber 170/730 computers.‡

**Discussion.** Despite the fact that the title compound was prepared by the procedure for *cis*[Pt(C<sub>7</sub>H<sub>13</sub>NH<sub>2</sub>)<sub>2</sub>Cl<sub>2</sub>], the compound is clearly the corresponding *trans* analogue. Similar results were found for the corresponding cyclohexylamine complex (Iball & Scrimgeour, 1977; Lock, Speranzini & Zvagulis, 1980). There are a number of possible explanations for these results: (a) the preparation gives exclusively the *trans* complex, (b) the preparation gives a mixture, and crystals of the *trans* complex are better formed and are therefore automatically selected by the crystallographer for study, (c) the process of recrystallization yields the *trans* complex by conversion from the *cis* complex.

Alternative (a) appears unlikely. Biological tests of the material from the 'cis' preparation showed marked physiological differences compared to genuine *trans* material (Braddock *et al.*, 1975; Connors *et al.*, 1972). Alternative (b) is a possibility, but a very extensive study that we are undertaking on the cyclobutylamine complexes (Lock & Zvagulis, 1980) suggests that alternative (c) is the correct explanation, although we have not demonstrated it for the title compound. The

\* The value of 0.04 was chosen to make  $\langle w(|F_o| - |F_c|)^2 \rangle$  locally independent of  $F_o$  and  $\sin \theta/\lambda$ .

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35252 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

‡ Most programs for initial data treatment were from the XRAY package (Stewart, 1976). The full-matrix least-squares program, CUDLS, and the Fourier program, SYMFOU, were written locally by J. S. Stephens and J. S. Rutherford respectively. The diagrams were prepared using ORTEP II (Johnson, 1976).

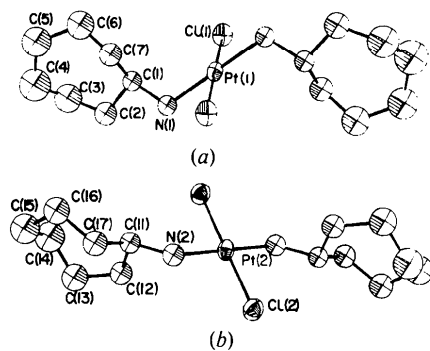


Fig. 1. Diagrams showing the numbering of the atoms in (a) molecule (1), and (b) molecule (2).

*cis-trans* interconversion of complexes in solution has been noted before by Kong & Rochon (1979). Since complexes of this type are important anticancer drugs (Braddock *et al.*, 1975; Connors *et al.*, 1972), it becomes most important when performing animal tests to use a well established recipe for making materials and to characterize fully the materials used, by techniques such as X-ray crystallography, before undertaking the tests. Interconversion of *cis* and *trans* isomers during purification can obviously occur much more easily than was suspected in the past.

Two independent molecules exist in the unit cell and these are illustrated in Fig. 1. Equivalent bond lengths in the two molecules do not differ significantly, except that C(3)–C(4) [1.43 (3) Å] is shorter than both C(5)–C(6) and C(15)–C(16) [1.57 (3), 1.58 (2) Å]. Most angles do not differ significantly. Within equivalent sets of angles of the amine only C(16)–C(17)–C(11) [111 (1)°] is smaller than C(11)–C(12)–C(13) [117.6 (9)°], and C(14)–C(15)–C(16) [111 (1)°] smaller than both C(4)–C(5)–C(6) [116 (1)°] and C(3)–C(4)–C(5) [119 (2)°]. We assume these small differences are caused by packing forces. There are significant differences in the Cl–Pt–N angles [(1), 91.5 (2); (2), 94.7 (3)°]. This difference is probably caused by steric hindrance. The larger angle is associated with the Cl atom which comes relatively close to C(11) [3.29 (1) Å]. The closest Cl–C distances in the other molecule [C(1)⋯Cl(1) 3.58 (1), C(1)⋯Cl(1) 3.64 Å] are much longer. This effect has been observed previously (Zanotti, Del Pra, Bombieri & Tamburro, 1978).

As shown in Fig. 1(a) the amines for the first molecule are oriented such that the rings lie roughly at right angles to the square plane of the ligand and such that the H atoms of C(7) would cover the axial positions above and below the square plane. The amines in the second group are arranged in a much more open arrangement, the mean plane through the ring lying at a smaller angle to the square plane, and such that the ring gives less protection to the axial

Table 3. Torsional and dihedral angles (°)

Torsional angles			
C(1)Pt(1)N(1)C(1)	+52.9 (5)	Cl(2)Pt(2)N(2)C(11)	–148.2 (5)
Pt(1)N(1)C(1)C(2)	–176.3 (6)	Pt(2)N(2)C(11)C(12)	+63.2 (6)
Pt(1)N(1)C(1)C(7)	+60.1 (6)	Pt(2)N(2)C(11)C(17)	–172.0 (6)
N(1)C(1)C(2)C(3)	–172.5 (8)	N(2)C(11)C(12)C(13)	+160.2 (8)
N(1)C(1)C(7)C(6)	–170.9 (9)	N(2)C(11)C(17)C(16)	+153.0 (8)
C(1)C(2)C(3)C(4)	+70 (1)	C(11)C(12)C(13)C(14)	+37 (1)
C(2)C(3)C(4)C(5)	–77 (1)	C(12)C(13)C(14)C(15)	–89 (1)
C(3)C(4)C(5)C(6)	+27 (1)	C(13)C(14)C(15)C(16)	+77 (1)
C(4)C(5)C(6)C(7)	+44 (1)	C(14)C(15)C(16)C(17)	–61 (1)
C(5)C(6)C(7)C(1)	–86 (1)	C(15)C(16)C(17)C(11)	+76 (1)
C(6)C(7)C(1)C(2)	+71 (1)	C(16)C(17)C(11)C(12)	–86 (1)
C(7)C(1)C(2)C(3)	–54 (10)	C(17)C(11)C(12)C(13)	+40 (1)
Dihedral angles			
N(1)C(1)C(2)C(7)	61.1 (9)	N(2)C(11)C(12)C(17)	59.7 (9)
N(1)C(1)C(7)C(2)	62.1 (9)	N(2)C(11)C(17)C(12)	120.7 (9)

Angles given are dihedral angles between the planes defined by the first three atoms and the last three atoms. Thus for *ABCD*, the dihedral angle is between the planes *ABC* and *BCD*.

Plane 1	Distance from plane (Å)
Pt(1)Cl(1)N(1)	C(1) 1.08 (2); C(2) 0.75 (2); C(3) 1.78 (2); C(4) 3.05 (2); C(5) 4.14 (3); C(6) 3.60 (3); C(7) 2.45 (2)
Plane 2	
Pt(2)Cl(2)N(2)	C(11) –0.68 (2); C(12) –2.18 (2); C(13) –3.17 (2); C(14) –2.64 (2); C(15) –1.85 (2); C(16) –0.40 (2); C(17) –0.34 (2)

positions. This is most easily demonstrated by considering the distances the C atoms are out of the square plane (Table 3): C(5) is 4.14 Å out of the plane in molecule (1) whereas for molecule (2) the atom most out of the plane is C(13) (3.17 Å). Within the rings the arrangement of the C atoms is the open chair or crown-type structure. The torsion angles in the rings do not show a good match. The best correspondence is obtained if the numbering correspondence is C(1)–C(7) ≡ C(15)–C(17), C(11)–C(14) and the signs are changed.

The packing is illustrated in Fig. 2. All Pt atoms lie in the *ac* plane and the molecules are oriented such that the rings point very roughly along the *b* direction so that in the *b* direction intermolecular contact is between the hydrocarbon groups at  $y = \frac{1}{2}$  and the forces are van der Waals. In the *c* direction both sets of translationally equivalent molecules (1) and (2) are stacked such that the square planes are tilted like tiles on a roof but the orientation of the stacks is different. Molecule (1) is canted so that it is inclined about 30° to the *ac* plane while molecule (2) is canted about 35° to the *bc* plane. The rings of translationally equivalent molecules in the *c* direction provide van der Waals contacts, but, in addition, there are weak hydrogen bonds [N(1)⋯Cl(1) 3.48 (1) Å, N(2)⋯Cl(2) 3.46 (1) Å] between adjacent molecules.

In the *a* direction there are again van der Waals contacts between the rings on adjacent (1) and (2) molecules with possible very weak hydrogen bonds

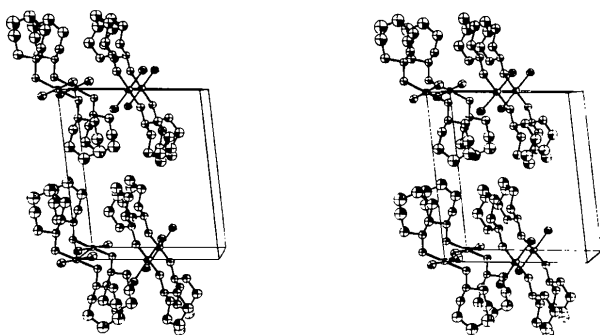


Fig. 2. The packing of the molecules within the unit cell. The contents of four cells are shown.  $\mathbf{a}$  and  $\mathbf{c}^* \times \mathbf{a}$  are parallel to the bottom and side of the page. The view is down  $\mathbf{c}^*$ .

[N(2)···Cl(1) 3.62 Å, N(1)···Cl(2) 3.61 Å] between adjacent molecules. The packing in this complex results in shortest Pt···Pt distances of 5.723 (2) and 5.828 (1) Å, considerably greater than the 3.4–3.5 Å typically found in *cis*-diamineplatinum(II) complexes (Lock, Speranzini & Zvagulis, 1980, and references therein). This difference in *trans* and *cis* complexes has already been noted by Srivasta, Froehlich & Eichhorn (1978).

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## Structure of Potassium Sucrose Octasulfate Heptahydrate

BY Y. NAWATA, K. OCHI, M. SHIBA AND K. MORITA

*Research Laboratories, Chugai Pharmaceutical Co. Ltd, Takada, Toshima, Tokyo 171, Japan*

AND Y. IITAKA

*Faculty of Pharmaceutical Sciences, University of Tokyo, Hongo, Bunkyo, Tokyo 113, Japan*

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**Abstract.**  $\text{C}_{12}\text{H}_{14}\text{O}_{35}\text{S}_8^{8-} \cdot 8\text{K}^+ \cdot 7\text{H}_2\text{O}$ ,  $M_r = 1413.58$ , orthorhombic,  $P2_12_12_1$ ,  $a = 16.865$  (12),  $b = 18.649$  (5),  $c = 14.051$  (3) Å,  $Z = 4$ , and  $D_x = 2.12$  Mg m $^{-3}$ . In the sucrose moiety, the furanosyl ring takes a  $^5T_4$  twist form while the pyranosyl ring takes a  $^4C_1$  chair. The  $\text{K}^+$  ion is surrounded by five to seven O

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atoms including three to seven from the sulfate groups. The final  $R$  is 0.056.

**Introduction.** A basic aluminum salt of sucrose sulfate (I) accelerates the healing of gastric and duodenal ulcers (Mayberry, Williams, Rhodes & Lawrie, 1978).