

scheme are shown in Fig. 1 and a stereoscopic view of the unit cell and four molecules is given in Fig. 2. Positional parameters and the equivalent isotropic values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.\* Bond lengths, bond angles and torsion angles are given in Table 2.

**Discussion.** The important feature of the structure revealed by the present study is the transition of the 1,2,4,5-tetrastannacyclohexane ring from the boat forms observed previously (Preut, Bleckmann, Mitchell & Fabisch, 1984; Preut & Mitchell, 1989) to a chair form. The presence of two methyl groups on each ring C atom apparently destabilizes the boat form because of unfavourable 1,4-axial methyl interactions similar to the 1,4-hydrogen interaction which destabilizes the boat form of cyclohexane. While the C—Sn—C angles vary only slightly between the octa-, deca- and dodecamethyl tetrastannacyclohexanes, the Sn—C—Sn angle decreases steadily [average values: octamethyl 115.5 (15), deca-

methyl 112.2 (4)°]. Although the torsion angles are similar for the two boat forms [octamethyl: 0 (1), 5 (2), -54 (2), 0 (1), 55 (2), -55 (2)°; *cis*-dodecamethyl: -1.5 (1), 51.7 (1), -48.8 (1), -3.5 (1), 54.6 (1), -51.5 (1)°] they are very different for the two chair structures [octaphenyl: -39.2 (3), 42.7 (4), -42.3 (4), 39.2 (3), -42.7 (4), 42.3 (4)°; title compound: -55.9 (2), 55.8 (2), -56.1 (2), 55.9 (2), -55.8 (2), 56.1 (2)°].

#### References

- LE PAGE, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.  
 MEUNIER-PIRET, J., VAN MEERSSCHE, M., GIELEN, M. & JURKSCHAT, K. (1983). *J. Organomet. Chem.* **252**, 289–294.  
 MITCHELL, T. N., FABISCH, B., WICKENKAMP, R., KUIVILA, H. G. & KAROL, T. (1986). *Silicon Germanium Tin Lead Compd.* **9**, 57–66.  
 NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.  
 PREUT, H., BLECKMANN, P., MITCHELL, T. & FABISCH, B. (1984). *Acta Cryst.* **C40**, 370–372.  
 PREUT, H. & MITCHELL, T. (1989). *Acta Cryst.* **C45**, 35–37.  
 SHELDRIK, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet R3m/V crystallographic systems. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Germany.  
 SPEK, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. SAYRE, p. 528. Oxford: Clarendon Press.  
 WILLIAMS, D. E. (1984). *PCK83. A Crystal and Molecular Packing Analysis Program*. QCPE Program No. 481. Department of Chemistry, Indiana Univ., USA.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53716 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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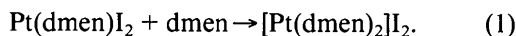
## Structure of Bis(*N,N*-dimethyl-1,2-ethanediamine)platinum(II) Diiodide

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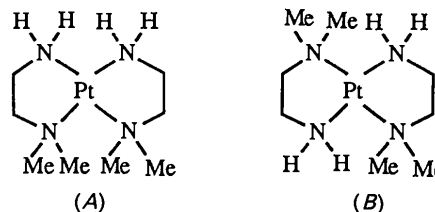
**Abstract.** [Pt(C<sub>8</sub>H<sub>24</sub>N<sub>4</sub>)]I<sub>2</sub>, *M<sub>r</sub>* = 625.2, monoclinic, *P*<sub>2</sub><sub>1</sub>/*c*, *a* = 5.712 (1), *b* = 15.901 (5), *c* = 8.740 (1) Å, β = 93.263 (9)°, *V* = 792.5 Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 2.62 (2), *D<sub>x</sub>* = 2.62 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.7107 Å, μ = 127.9 cm<sup>-1</sup>, *F*(000) = 568, *T* = 293 K, final *R* = 0.037 for 1882 counter-measured reflections. The structure consists of centrosymmetric square-planar molecules. Pt—N distances are 2.053 (5) and 2.045 (5) Å and the chelate N—Pt—N angle is 83.7 (2)°.

**Introduction.** The complex bis(*N,N*-dimethyl-1,2-ethanediamine)platinum(II) diiodide, [Pt(dmen)]<sub>2</sub>I<sub>2</sub>, has been prepared by reaction (1).



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*cis* (*A*) or *trans* (*B*) stereochemistry is possible for the complex cation, and this cannot be readily determined by spectroscopic and physicochemical methods. Thus, the X-ray crystal structure of the compound has been determined and the X-ray powder pattern of the bulk material has been compared with the single-crystal measurements to ascertain whether the synthesis is stereoselective.



**Experimental.** *Synthesis.* Pt(dmen)I<sub>2</sub> (3.72 mmol) was dissolved with warming in dmen (5 ml) and the resulting solution was stirred for 0.75 h. During this time the initially yellow solution became colourless and a white precipitate formed. The solvent was removed under vacuum and the residue recrystallized from water. Found: C 15.2, H 3.8, N 9.0%. [Pt(C<sub>8</sub>H<sub>24</sub>N<sub>4</sub>)]I<sub>2</sub> requires: C 15.4, H 3.9, N 9.0%.

*Crystal data.* A suitable tabular crystal (0.375 × 0.10 × 0.10 mm) was selected from recrystallized product. *D<sub>m</sub>* measured in 1,1,2,2-tetrabromoethane-carbon tetrachloride. The X-ray powder diffraction pattern of the bulk material was indexed completely using the single-crystal unit cell. No lines resulting from either another isomer or impurities were detected. Accordingly, the reaction given in (1) is 100% stereoselective.

Space group *P2<sub>1</sub>/c* was assigned from systematic absences *0k0 k* odd and *h0l l* odd; cell parameters determined from 25 reflections ( $6 < \theta < 12^\circ$ ) with a Philips PW1100 automatic four-circle diffractometer equipped with a graphite monochromator as described previously (Canty, Chaichit & Gatehouse, 1980). Three standard reflections were measured at 4 h intervals. No decomposition occurred. Data collected using the  $\omega$ -scan technique with a symmetric scan width of  $\pm 0.60^\circ$  in  $\omega$  from the calculated Bragg angle, with an allowance for dispersion, at a scan rate of  $0.05^\circ \text{ s}^{-1}$ . No reflection was sufficiently intense to require the insertion of an attenuation filter. Data were processed using a program written specifically for the PW1100 diffractometer (Hornstra & Stubbe, 1972). Values of *I* and  $\sigma(I)$  were corrected for Lorentz and polarization effects. An absorption correction was applied on the basis of indexed crystal faces, maximum and minimum transmission factors were 0.3397 and 0.2685 respectively. 2601 reflections measured to  $2\theta = 60^\circ$ , 1882 unique reflections [ $I > 3\sigma(I)$ ] used in analysis, index range *h* -8/8, *k* 0/20, *l* 0/12. Pt and I parameters were found from a Patterson synthesis and all non-H atoms were located in subsequent difference Fourier syntheses. Function minimized in full-matrix least-squares refinement was  $\sum w(|F_o| - |F_c|)^2$ , where *w* is the weight  $[\sigma^2(F_o)]^{-1}$ . Pt and I atoms were refined anisotropically, other atoms were refined isotropically (H atoms in their calculated positions refined using a riding model with a single isotropic thermal parameter for all H atoms); 41 variable parameters, final *R* = 0.037 and *wR* = 0.054 (for observed reflections); max.  $\Delta/\sigma$  in final cycle 0.001. Final difference Fourier synthesis had  $\Delta\rho$  within 2.0 and  $-4.0 \text{ e } \text{Å}^{-3}$ , both in the vicinity of the Pt atom. Scattering factors for neutral atoms and corrections for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on the Monash University

Table 1. Atomic parameters and temperature factors with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub> (Å <sup>2</sup> )
Pt	0.5	0.0	0.5	†
I(1)	0.0022 (1)	0.1533 (1)	0.1439 (1)	†
C(1)	0.4794 (11)	0.1852 (5)	0.4830 (7)	0.034 (1)
C(2)	0.5125 (11)	0.1597 (4)	0.6476 (7)	0.032 (1)
C(3)	0.1351 (12)	0.0868 (4)	0.6651 (8)	0.037 (1)
C(4)	0.4745 (11)	0.0484 (5)	0.8316 (7)	0.033 (1)
N(1)	0.5250 (9)	0.1108 (3)	0.3832 (6)	0.027 (1)
N(2)	0.3991 (8)	0.0764 (3)	0.6738 (5)	0.023 (1)

	<i>U</i> <sub>11</sub>	<i>U</i> <sub>22</sub>	<i>U</i> <sub>33</sub>	<i>U</i> <sub>12</sub>	<i>U</i> <sub>13</sub>	<i>U</i> <sub>23</sub>
Pt	0.017 (1)	0.021 (1)	0.016 (1)	0.002 (1)	0.003 (1)	0.001 (1)
I(1)	0.030 (1)	0.041 (1)	0.040 (1)	-0.003 (1)	0.005 (1)	0.006 (1)

† Anisotropic thermal parameters are of the form  $\exp[-2^2(U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^* + \dots)]$ .

Table 2. Interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses

Pt—N(1)	2.045 (5)	C(2)—N(2)	1.499 (8)
Pt—N(2)	2.053 (5)	C(3)—N(2)	1.515 (9)
N(1)—C(1)	1.502 (9)	C(4)—N(2)	1.489 (8)
C(1)—C(2)	1.497 (9)		
N(1)—Pt—N(2)	83.7 (2)	N(1)—C(1)—C(2)	109.2 (6)
Pt—N(1)—C(1)	111.7 (4)	C(1)—C(2)—N(2)	110.7 (5)
Pt—N(2)—C(2)	105.6 (4)	C(2)—N(2)—C(3)	109.5 (5)
Pt—N(2)—C(3)	110.4 (4)	C(2)—N(2)—C(4)	107.5 (5)
Pt—N(2)—C(4)	115.5 (4)	C(3)—N(2)—C(4)	108.3 (5)
N(1)...I	3.594 (5)	N(1)—HN(1)—I	155.0 (3)
N(2)...I	3.611 (5)	N(1)—HN(2)—I	157.5 (3)
1* 2 3 4		1* 2 3 4	
N(1)—C(1)—C(2)—N(2)	-44.1 (6)	Pt—N(1)—C(1)—C(2)	19.7 (6)
C(4)—N(2)—C(2)—C(1)	170.2 (5)	Pt—N(2)—C(2)—C(1)	46.5 (5)
C(3)—N(2)—C(2)—C(1)	-72.4 (6)		

\*The convention is that if a clockwise motion of atom 1 superimposes it on atom 4 then the sign is positive.

VAX11/780 computer system. Programs used: *SHELX76* (Sheldrick, 1976) and *ORFFE* (Busing, Martin & Levy, 1964).

**Discussion.** Final fractional coordinates and thermal parameters are given in Table 1,\* interatomic distances and selected angles are in Table 2. The numbering scheme used is given in Fig. 1.

The ligand (dmen) in the centrosymmetric square-planar cation [Pt(dmen)<sub>2</sub>]<sup>2+</sup> has a bite angle of  $83.7(2)^\circ$  and Pt—N distances of 2.052(5) and 2.045(5) Å compared with those [84.0(5)°, 2.039(14) and 2.067(12) Å] of dichloro(*N,N*-dimethyl-1,2-ethanediamine)platinum(II), [Pt(dmen)Cl<sub>2</sub>], reported by Melanson, de la Chevrotière & Rochon (1987). The torsion angle for the

\* Lists of structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53743 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

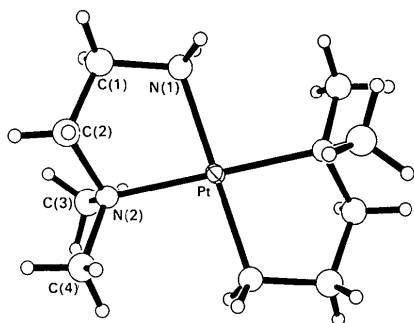


Fig. 1. Structure of  $[\text{Pt}(\text{dmen})_2]\text{I}_2$ , showing the numbering used.

ligand backbone for  $[\text{Pt}(\text{dmen})_2]^{2+}$  is  $-44.1(6)^\circ$  (for the ligand coordinates as given) compared with that calculated for  $[\text{Pt}(\text{dmen})\text{Cl}_2]$ ,  $+54.9(8)^\circ$ . However, it should be noted that as the absolute configuration for  $[\text{Pt}(\text{dmen})\text{Cl}_2]$  was not determined (it crystallizes in space group  $P2_1$ ) the sign of the torsion angle for this compound has little significance.

The deviations of the C atoms from the ligand plane through Pt, N(1) and N(2) are C(1)  $-0.110(6)$ , C(2)  $-0.660(6)$ , C(3)  $1.420(7)$  and C(4)  $-0.756(6)$  Å. They indicate that the ligands in this complex have the asymmetric skew conformation ( $\lambda$  and  $\delta$ , as required by the centre of symmetry) following the nomenclature of Hawkins (1971), and that C(3) tends to be axial with C(4) tending to be equatorial. The ligand in  $[\text{Pt}(\text{dmen})\text{Cl}_2]$  (Melanson *et al.*, 1987) has the symmetric skew conformation, with

the deviations of the C atoms from the Pt, N(1) and N(2) plane being C(1)  $0.38(2)$ , C(2)  $-0.31(2)$ , C(3)  $1.35(2)$  and C(4)  $-1.05(2)$  Å, with C(3) and C(4) approximately equally above and below the Pt-N plane.

The shortest intermolecular distance in  $[\text{Pt}(\text{dmen})_2]^{2+}$ , that between N(1) and the I ion [ $3.594(5)$  Å], and the angles subtended at HN(1) and HN(2) by N(1) and the I ion [ $155.0(3)$  and  $157.5(3)^\circ$  respectively], indicate possible hydrogen bonding.

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#### References

- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.
- CANTY, A. J., CHAICHIT, N. & GATEHOUSE, B. M. (1980). *Acta Cryst.* B36, 786–789.
- HAWKINS, C. (1971). *Absolute Configuration of Metal Complexes*. Interscience Monograph on Chemistry. New York: John Wiley.
- HORNSTRA, J. & STUBBE, B. (1972). *PW1100*. Data-processing program. Philips Research Laboratories, Eindhoven, The Netherlands.
- MELANSON, R., DE LA CHEVROTIÈRE, C. & ROCHON, F. D. (1987). *Acta Cryst.* C43, 57–59.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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## [Bis(diphenylphosphino)methane-*P,P'*](chloro)(pentafluorophenyl)platinum(II): $\text{PtCl}(\text{dppm})\text{C}_6\text{F}_5$

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**Abstract.**  $\text{C}_{31}\text{H}_{22}\text{ClF}_5\text{P}_2\text{Pt}$ ,  $M_r = 782.0$ , triclinic,  $P\bar{1}$ ,  $a = 10.119(1)$ ,  $b = 15.676(2)$ ,  $c = 9.934(2)$  Å,  $\alpha = 105.64(5)$ ,  $\beta = 112.36(1)$ ,  $\gamma = 84.57(1)^\circ$ ,  $V = 1403(2)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.851$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 52.67$  cm<sup>-1</sup>,  $F(000) = 756$ ,  $T = 293(1)$  K,  $R = 0.022$  for 4107 observed reflections. In

the monomeric complex, the approximately square-planar geometry about the Pt atom is defined by a Cl atom [Pt—Cl  $2.360(1)$  Å], a C atom of the pentafluoroaryl group [Pt—C  $2.078(4)$  Å] and two P atoms derived from a chelating  $\text{Ph}_2\text{PCH}_2\text{PPh}_2$  ligand [Pt—P  $2.224(1)$  and  $2.295(1)$  Å; P—Pt—P  $73.9(1)^\circ$ ]

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