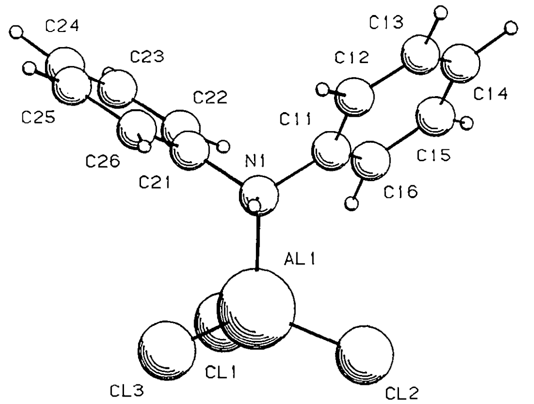

Fig. 1. View of diphenylamine–trichloroaluminium(III) (1/1).

lengths and angles.* A view (Davies, 1983) of the molecular unit is shown in Fig. 1.

Related literature. The structural features of $Ph_2NH.AlCl_3$ are consistent with those of $Me_3N.AlCl_3$ (Grant, Killean & Lawrence, 1969) and $(Me_2NH)_2.AlCl_3$ (Ahmed, Schwarz, Weidlein & Hess, 1977).

We thank the Natural Sciences and Engineering Research Council of Canada (NB and TSC), the Donors of the Petroleum Research Fund of the American Chemical Society (NB) and the Killam Foundation (BWR and BB) for financial support.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles and hydrogen positions, and a view of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53657 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Ahmed, A., Schwarz, W., Weidlein, J. & Hess, H. (1977). *Z. Anorg. Allg. Chem.* **434**, 207–216.
Cameron, T. S. & Cordes, R. E. (1979). *Acta Cryst.* **B35**, 748–750.
Davies, E. K. (1983). *The CHEMGRAF Suite*. Chemical Crystallography Laboratory, Oxford, England.
Grant, D. F., Killean, R. C. G. & Lawrence, J. L. (1969). *Acta Cryst.* **B25**, 377–379.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86*. *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Kruger & R. Goddard, pp. 175–189. Oxford Univ. Press.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1991). C**47**, 1067–1069

Structure of DL-Dichloro(*trans*-1,2-diamino-*trans*-3,6-cyclohexanediol)platinum(II) Monohydrate, $[PtCl_2(C_6H_{14}N_2O_2)].H_2O$

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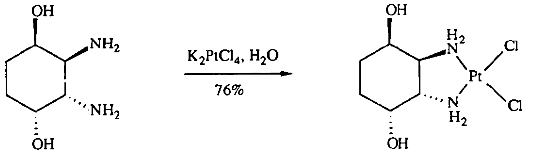
(*Received* 29 *March* 1990; *accepted* 18 *September* 1990)

Abstract. $M_r = 430{\cdot}19$, monoclinic, $P2_1/c$, $a = 10{\cdot}919\,(7)$, $b = 10{\cdot}520\,(2)$, $c = 9{\cdot}789\,(4)$ Å, $\beta = 103{\cdot}26\,(4)^\circ$, $V = 1094{\cdot}5$ Å^3, $Z = 4$, $D_x = 2{\cdot}610$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Cu } K\alpha) = 13{\cdot}43$ mm^{-1}, $F(000) = 808$, $T = 293$ K, $R = 0{\cdot}0310$, $wR = 0{\cdot}0326$ for 1655 observed reflections. The title *trans*-(diaminocyclohexane)platinum complex is of biological interest as an antitumor agent. We resorted to X-ray analysis to establish the configuration of the substituents. A water molecule is hydrogen bonded to one of the hydroxyls.

Experimental. The reaction of *trans*-1,2-diamino-*trans*-3,6-dihydroxycyclohexane (1) with potassium tetrachloroplatinate(II) in water gave the platinum complex (2) in 76% yield (Hanessian & Wang, 1990) which was subjected to X-ray analysis.



(1) (2)

Crystal of $C_6H_{14}Cl_2N_2O_2Pt.H_2O$, bounded by {100}, {010}, {001} and with dimensions $0{\cdot}04 \times 0{\cdot}15 \times 0{\cdot}19$ mm, was obtained from water. Unit-cell dimensions from 25 well centered reflections in the

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Table 1. *Final atomic coordinates* ($\times 10^5$ *for* Pt; $\times 10^4$ *for* Cl, O, N *and* C) *with e.s.d.'s in parentheses and* U_{eq} *values* ($Å^2 \times 10^3$)

$$U_{eq} = (1/3)\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}
Pt	101506 (4)	39138 (3)	62857 (4)	27
Cl(1)	12174 (3)	4721 (3)	7139 (3)	42
Cl(2)	10740 (3)	2672 (2)	4609 (3)	40
O(3)	5853 (7)	3896 (7)	5212 (7)	45
O(6)	8054 (8)	4462 (6)	9766 (6)	42
W	3565 (13)	2570 (10)	4430 (14)	111
N(1)	9399 (9)	4929 (7)	7691 (8)	33
N(2)	8376 (8)	3262 (7)	5674 (7)	31
C(1)	8013 (9)	4780 (8)	7353 (9)	28
C(2)	7737 (10)	3422 (9)	6868 (9)	29
C(3)	6337 (10)	3174 (9)	6448 (9)	34
C(4)	5714 (11)	3506 (9)	7647 (10)	42
C(5)	5997 (11)	4879 (9)	8136 (10)	41
C(6)	7391 (10)	5156 (9)	8530 (9)	34

Table 2. *Interatomic distances* (Å), *bond angles* (°), *torsion angles* (°) *and hydrogen-bond geometry* (Å, °) *with e.s.d.'s in parentheses*

Pt—Cl(1)	2·335 (3)	C(2)—C(3)	1·512 (15)
Pt—Cl(2)	2·302 (3)	C(3)—C(4)	1·526 (14)
Pt—N(1)	2·055 (8)	C(4)—C(5)	1·531 (14)
Pt—N(2)	2·013 (9)	C(5)—C(6)	1·510 (16)
N(1)—C(1)	1·481 (14)	C(6)—C(1)	1·518 (14)
N(2)—C(2)	1·502 (12)	C(3)—O(3)	1·424 (12)
C(1)—C(2)	1·514 (12)	C(6)—O(6)	1·456 (11)

Cl(1)—Pt—Cl(2)	93·73 (9)	N(2)—C(2)—C(3)	113·0 (8)
Cl(1)—Pt—N(1)	93·55 (24)	C(1)—C(2)—C(3)	111·4 (8)
Cl(1)—Pt—N(2)	175·99 (23)	C(2)—C(3)—C(4)	110·7 (8)
Cl(2)—Pt—N(1)	172·72 (24)	C(3)—C(4)—C(5)	111·3 (9)
Cl(2)—Pt—N(2)	90·12 (23)	C(4)—C(5)—C(6)	112·4 (9)
N(1)—Pt—N(2)	82·6 (3)	C(5)—C(6)—C(1)	110·9 (8)
Pt—N(1)—C(1)	109·9 (6)	C(6)—C(1)—C(2)	113·1 (8)
Pt—N(2)—C(2)	108·5 (6)	C(1)—C(6)—O(6)	106·0 (8)
N(1)—C(1)—C(2)	106·8 (7)	C(5)—C(6)—O(6)	113·3 (8)
N(1)—C(1)—C(6)	114·5 (8)	C(2)—C(3)—O(3)	107·1 (8)
N(2)—C(2)—C(1)	104·8 (7)	C(4)—C(3)—O(3)	112·8 (8)

C(1)—C(2)—C(3)—C(4)	−55·5 (10)	N(1)—C(1)—C(2)—N(2)	−55·7 (9)
C(2)—C(3)—C(4)—C(5)	55·4 (11)	N(1)—C(1)—C(6)—C(5)	−175·6 (8)
C(3)—C(4)—C(5)—C(6)	−54·6 (11)	N(1)—C(1)—C(2)—C(3)	−178·2 (8)
C(4)—C(5)—C(6)—C(1)	52·5 (11)	N(2)—C(2)—C(1)—C(6)	177·4 (7)
C(5)—C(6)—C(1)—C(2)	−52·9 (11)	N(2)—C(2)—C(3)—C(4)	−173·1 (8)
C(6)—C(1)—C(2)—C(3)	54·9 (11)	O(3)—C(3)—C(4)—C(5)	−64·5 (11)
O(6)—C(6)—C(5)—C(4)	−66·6 (11)	O(3)—C(3)—C(2)—N(2)	−49·8 (10)
O(6)—C(6)—C(1)—N(1)	−52·3 (10)	O(3)—C(3)—C(2)—C(1)	67·8 (10)
O(6)—C(6)—C(1)—C(2)	70·5 (10)		

A—H···*B*	*A*—*B*	H—*B*	*A*—H—*B*
O(3)—HO(3)···O(3^{i})	2·947 (11)	2·364 (8)	125·9 (5)
O(3)—HO(3)···*W*	2·810 (16)	2·256 (13)	122·7 (6)
O(6)—HO(6)···Cl(1^{ii})	3·213 (7)	2·414 (3)	157·2 (5)
N(2)—HN(2A)···O(6^{iii})	2·997 (10)	2·102 (6)	173·3 (5)

C(3)—O(3)···O(3^{i})	129·8 (6)	C(6)—O(6)···Cl(1^{ii})	121·2 (5)
C(3)—O(3)···*W*	95·8 (6)	C(2)—N(2)···O(6^{iii})	107·1 (5)

Symmetry operations: (i) $1 - x, 1 - y, 1 - z$; (ii) $2 - x, 1 - y, 2 - z$; (iii) $x, \frac{1}{2} - y, -\frac{1}{2} + z$.

range $9·5 \leq \theta \leq 11·2°$. Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo $K\alpha$ radiation, ω–2θ scan, $2\theta_{max} = 50·0°$. Orientation monitored every 200 measurements, intensity checked every hour using seven standard reflections with the largest intensity fluctuation 0·6%. 4058 measured reflections, 1918 ($-12 \leq h \leq 12$, $0 \leq k \leq 12$, $0 \leq l \leq 11$) of which were retained after averaging of the equivalent reflections to Laue symmetry $2/m$ (with R averaging 0·034), of which 1656 with $I \geq 3·0\sigma(I)$ were retained for the structure determination and refinement. Lp correction, absorption correction (transmission range 0·03–0·4).

The structure was solved using direct methods (*MULTAN*80)* and difference Fourier calculations (*SHELX*76). Full-matrix least-squares refinement based on F, all non-H atoms anisotropic, H coordinates were found on difference Fourier synthesis and/or calculated, then fixed at idealized positions ($d_{C—H} = 1·08$, $d_{N—H} = 0·90$, $d_{O—H} = 0·85$ Å). U_{iso} for H atoms previously refined, fixed in the last cycles: $U_{C—H}$, $U_{N—H} = 0·05$ and $U_{O—H} = 0·09$ Å^2. The function minimized was $\sum w(|F_o| - |F_c|)^2$. In the last refinement cycle one reflection (100) with high residuals was given zero weight because of suspected errors in intensity measurements. Final $R = 0·0310$, $wR = 0·0326$ [weights derived from the counting statistics, $w = 1/\sigma^2(F)$] and $S = 4·194$ for 152 parameters. Maximum $(\Delta/\sigma) = 0·12$, average $(\Delta/\sigma) = 0·01$. The residual electron density fluctuation on final difference Fourier synthesis was below $\pm 0·3$ e Å^{-3}, with a few peaks in the range $\pm 0·4$–$0·7$ e Å^{-3} near Pt and Cl.

The scattering curves for the non-H atoms were taken from Cromer & Mann (1968) and those for the H atoms from Stewart, Davidson & Simpson (1965). Real and imaginary parts of anomalous dispersion of Pt and Cl were from Cromer & Liberman (1970).

The final coordinates for non-H atoms are given in Table 1, bond distances, bond angles and torsion angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

Related literature. The antitumor activity of platinum complexes has prompted many researchers to investigate in this area. Similar diamino platinum complexes have been synthesized and X-ray structures have been resolved (Witiak, Rotella, Filppi & Gal-

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* The programs used here are modified versions of *NRC*-2, data reduction, *NRC*-10, bond distances and angles and *NRC*-22, mean planes (Ahmed, Hall, Pippy & Huber, 1973); *MULTAN*80, multisolution program (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); *SHELX*76, program for crystal structure determination (Sheldrick, 1976); and *ORTEP* (Johnson, 1965).

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and distances to least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53664 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

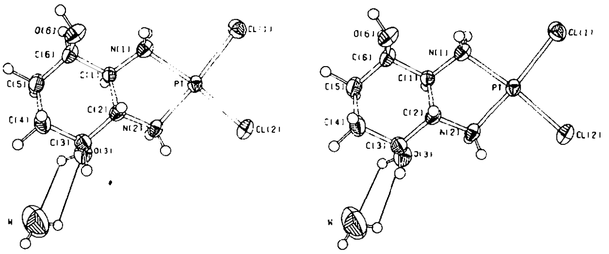

Fig. 1. Stereopair showing the molecular conformation and the atomic numbering of $C_6H_{14}Cl_2N_2O_2Pt.H_2O$. The ellipsoids correspond to 50% probability, except for the H atoms which are shown as spheres of arbitrary size.

lucci, 1987); see also Okude, Ichida, Miyamoto & Sasaki (1989).

We would like to thank Le Ministère de l'Education du Québec, NSERC and the National Cancer Institute (Canada) for financial support.

References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1973). *NRC Crystallographic Programs for the IBM* 360 *System.* Accession Nos. 133–137. *J. Appl. Cryst.* **6**, 309–346.
Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
Hanessian, S. & Wang, J. (1990). In preparation.
Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
Okude, K., Ichida, H., Miyamoto, T. K. & Sasaki, Y. (1989). *Chem. Lett.* pp. 119–120.
Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
Witiak, D. T., Rotella, D. P., Filppi, J. A. & Gallucci, J. (1987). *J. Med. Chem.* **30**, 1327–1336.

Acta Cryst. (1991). C47, 1069–1072

Structures of Trimethylphosphonium *trans*-Tetrachlorobis(trimethylphosphine)chromate(III), *mer*-Trichlorobis(dimethylphenylphosphine)(dimethylphenylphosphine oxide)chromium(III) and *mer*-Trichlorotris(trimethylphosphine oxide)molybdenum(III) Dichloromethane Solvate

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(*Received* 2 *April* 1990; *accepted* 22 *October* 1990)

Abstract. (I) $[C_3H_{10}P][CrCl_4(C_3H_9P)_2]$, $M_r = 423{\cdot}05$, monoclinic, $C2/c$, $a = 15{\cdot}334$ (4), $b = 10{\cdot}202$ (2), $c = 13{\cdot}145$ (2) Å, $\beta = 92{\cdot}49$ (1)°, $V = 2054{\cdot}4$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}368$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 12{\cdot}8$ cm^{-1}, $F(000) = 876$, $T = 292$ K, $R = 0{\cdot}043$ for 1348 ($F_o^2 > 3\sigma F_o^2$) reflections. (II) $[CrCl_3(C_8H_{11}OP)(C_8H_{11}P)_2]$, $M_r = 588{\cdot}81$, orthorhombic, $Pnma$, $a = 23{\cdot}234$ (4), $b = 13{\cdot}677$ (2), $c = 9{\cdot}064$ (1) Å, $V = 2880{\cdot}3$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}358$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 8{\cdot}5$ cm^{-1}, $F(000) = 1220$, $T = 292$ K, $R = 0{\cdot}047$ for 1246 ($F_o^2 > 3\sigma F_o^2$) reflections. (III) $[MoCl_3(C_3H_9OP)_3].CH_2Cl_2$, $M_r = 563{\cdot}47$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}402$ (2), $b = 12{\cdot}298$ (3), $c = 21{\cdot}607$ (6) Å, $V = 2498{\cdot}1$ (7) Å^3, $Z = 4$, $D_x = 1{\cdot}498$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 12{\cdot}5$ cm^{-1}, $F(000) = 1140$, $T = 292$ K, $R = 0{\cdot}041$ for 2176 ($F_o^2 > 3\sigma F_o^2$) reflections. $\bar{1}$ symmetry is imposed on the anion in (1). All bond angles at Cr are close to 90°. Twofold symmetry is imposed on the cation $[PMe_3H]^+$ which leads to disorder between one methyl group and the H atom bonded on P. Cr—Cl distances are 2·327 (1)and 2·353 (1) Å; Cr—P is 2·468 (1) Å. Mirror symmetry is imposed on (II); the Cr atom, three Cl atoms and the phenylphosphine oxide groups of the dimethylphenylphosphine oxide ligand are in the mirror plane. The Cr—Cl distances range from 2·319 (3) to 2·346 (3) Å and Cr—P is 2·489 (2) Å. Cr—O is 1·962 (6) Å. The angles between the *cis*-nonequivalent atoms range from 85·40 (5)° for Cl(3)—Cr—P(2) to 94·56 (5)° for P(2)—Cr—O(1). Cr—O(1)—P(1) is 154·6 (4)° and O(1)—P(1) is 1·501 (7) Å. For (III), the Mo—Cl distances range from 2·432 (3) to 2·441 (3) Å,

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0108-2701/91/051069-04$03.00