La Fig. 1 représente la molécule et les ellipsoïdes de vibration thermique avec une probabilité de 50% tracées avec le programme *ORTEP* (Johnson, 1965). Sur la Fig. 2, sont reportées les distances interatomiques et les angles; les écarts standards sont indiqués entre parenthèses.

Tableau 2. *Coordonnées cristallographiques des atomes d'hydrogène*

	x	y	z
H(1)	0,107 (3)	0,307 (3)	0,366 (11)
H(2)	0,048 (3)	0,308 (3)	0,060 (10)
H(3)	0,151 (3)	0,349 (3)	0,095 (11)
H(4)	0,218 (3)	0,204 (3)	0,121 (11)
H(5)	0,157 (3)	0,211 (4)	−0,180 (12)
H(6)	0,114 (3)	0,118 (3)	0,367 (13)
H(7)	0,054 (3)	0,126 (3)	0,096 (11)
H(8)	0,163 (3)	0,041 (4)	−0,138 (12)
H(9)	0,108 (3)	−0,066 (3)	0,361 (13)
H(10)	0,054 (3)	−0,054 (3)	0,098 (12)
H(11)	0,215 (3)	−0,159 (4)	0,102 (11)
H(12)	0,148 (3)	−0,150 (4)	−0,173 (12)
H(13)	0,039 (3)	−0,228 (3)	0,071 (11)
H(14)	0,102 (3)	−0,220 (4)	0,339 (11)
H(15)	0,137 (3)	−0,301 (3)	0,103 (12)
H(16)	0,298 (3)	0,047 (3)	−0,181 (13)
H(17)	0,436 (3)	0,054 (3)	−0,128 (12)
H(18)	0,414 (3)	−0,034 (3)	0,068 (10)
H(19)	0,414 (3)	0,063 (3)	0,224 (11)

Discussion. Une distance intermoléculaire N–O de 2,817 Å et l'observation d'une bande d'absorption très intense à 3300 cm^{-1} caractéristique de la vibration de valence du groupement N–H lié, montrent l'existence d'une liaison hydrogène N–H···O. Les molécules s'enchaînent d'une maille élémentaire à l'autre selon cette liaison, dont la direction est proche de l'axe c.

Les atomes de la molécule se répartissent en deux plans:

- Le plan P_1 de la chaîne centrale (trois atomes de carbone, atomes d'azote et d'oxygène).
- Le plan P_2 du reste dipropyle (sept atomes de carbone).

Ces deux plans font un angle de 89,6°. Les deux groupements propyles s'orientent symétriquement par rapport au plan P_2 en formant une chaine plane complètement étendue de longueur totale 7,568 Å.

Ces caractéristiques structurales et ce mode d'enchaînement intermoléculaire s'observent dans tous les composés de la série déjà étudiés, quelle que soit leur activité pharmacologique.

Références

Busing, W. R. & Levy, H. A. (1959). *ORFLS*. Report ORNL 59-4-37, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Cohen-Addad, C. (1973). *Acta Cryst.* B29, 157–161.

Cohen-Addad, C., Lajzérowicz, J., Benoit-Guyod, J. L. & Boucherle, A. (1972). *Chim. Ther.* 7, 384–387.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A27, 368–376.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Kurtz, S. K. & Perry, T. T. (1968). *J. Appl. Phys.* 39, 3798–3813.

Troughton, P. G. H. (1969). Ph. D. Thesis, Imperial College, London.

Trichloro-(π-*trans*-pent-2-enylammonium)platinum(II)

By R. Spagna, G. Ughetto and L. Zambonelli

Laboratorio di Strutturistica Chimica 'Giordano Giacomello', C.N.R., Città Universitaria, 00100 Roma, Italy

Abstract. PtCl$_3$(C$_5$H$_{12}$N), monoclinic, $P2_1/c$, $a=$ 11·215(3), $b=8·729(2)$, $c=10·259(3)$ Å, $\beta=90·0(1)°$, $D_m=2·56(1)$ g cm^{-3}, $Z=4$, $D_c=2·563$ g cm^{-3}. The zwitterionic complex was prepared by method (1) of Denning & Venanzi, *J. Chem. Soc.* (1963), pp. 3241–3247. The ligand *trans*-pent-2-enylamine hydrochloride was prepared from *trans*-bromopent-2-ene [Bouis (1928). *Ann. Chim.* 9, 402–465] by the Gabriel phthalimide method [Sheenan & Bolhofer (1950). *J. Amer. Chem. Soc.* 72, 2786–2788]. The *N*-pent-2-enyl-*N'*-phenylthiourea derivative, recrystallized from dilute alcohol, melted at 101·5–102·5°C. (Found: C, 65·62; H, 7·41; N, 12·93; S, 14·74%. Calc. for C$_{12}$H$_{16}$N$_2$S: C, 65·41; H, 7·31; N, 12·70; S, 14·55%).

Introduction. Cell constants were determined by a least-squares refinement of 35 2θ values measured on $h0l$ and $0kl$ Weissenberg photographs collected at ~ 23°C, using Cu Kα radiation ($\lambda=1·5418$ Å). Systematic absences were observed for $h0l$ reflexions with l odd and

Table 1. *Positional and thermal parameters for the non-hydrogen atoms* ($\times 10^4$)

Estimated standard deviations are given in parentheses. Coefficients b_{ij} are defined by the expression

$$T = \exp\left[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)\right].$$

	x	y	z	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
Pt	3053 (0)	536 (1)	222 (0)	52 (0)	12 (1)	−14 (1)	114 (1)	12 (1)	71 (1)
Cl(1)	4347 (3)	2607 (4)	339 (4)	81 (3)	−37 (7)	−16 (5)	152 (6)	−18 (8)	110 (4)
Cl(2)	3714 (3)	−234 (5)	2258 (4)	104 (4)	34 (8)	−44 (6)	169 (6)	31 (8)	98 (4)
Cl(3)	1920 (4)	−1674 (5)	143 (4)	111 (4)	−99 (9)	−73 (8)	183 (7)	94 (10)	159 (5)
N	3796 (12)	−1151 (16)	−2783 (15)	107 (14)	52 (29)	−48 (27)	145 (22)	−123 (35)	189 (21)
C(1)	2675 (13)	−274 (18)	−2685 (15)	83 (13)	−18 (29)	−68 (24)	141 (24)	1 (33)	114 (17)
C(2)	2774 (12)	1097 (18)	−1742 (14)	74 (12)	33 (28)	−17 (21)	154 (24)	−21 (31)	96 (15)
C(3)	1759 (12)	1610 (19)	−1079 (13)	77 (12)	59 (30)	−19 (21)	170 (25)	24 (32)	89 (14)
C(4)	1455 (13)	3203 (20)	−762 (15)	81 (13)	79 (32)	−9 (24)	180 (27)	0 (37)	122 (18)
C(5)	630 (15)	3855 (24)	−1812 (20)	77 (14)	85 (40)	−27 (32)	254 (39)	102 (54)	207 (28)

$0k0$ reflexions with k odd. The intensities were obtained from a crystal of approximate dimensions $0.05 \times 0.10 \times 0.12$ mm mounted on a Siemens AED single-crystal diffractometer (Mo $K\alpha$, Zr-filtered, θ–2θ scan, scintillation counter with pulse height discrimination). The unique b axis of the crystal was coincident with the polar φ axis of the goniostat. 1951 independent reflexions were measured in a hemisphere up to 2θ (Mo $K\alpha$) = 50°; 1560 of these, with $F_o^2 > 3\sigma(F_o^2)$ were used for the analysis. A correction for the shape anisotropy [μ(Mo $K\alpha$) = 154·1 cm^{-1}] was applied according to the method described in the literature (Furnas, 1957; North, Phillips & Matthews, 1968) using a procedure similar to that previously described (Spagna & Zambonelli, 1971). For this correction the variation in intensity of the conveniently intense axial reflexions (020, 040, 080 and 0,10,0) was measured as a function of φ. Lorentz and polarization corrections were then applied.

The structure was solved by the Patterson and Fourier methods and refined by least-squares calculations. The full-matrix least-squares refinement converged at $R = 0.060$ with isotropic temperature factors. The positions of the hydrogen atoms were idealized in a manner similar to that previously described (Spagna & Zambonelli, 1972) and the contribution of the hydrogen atoms
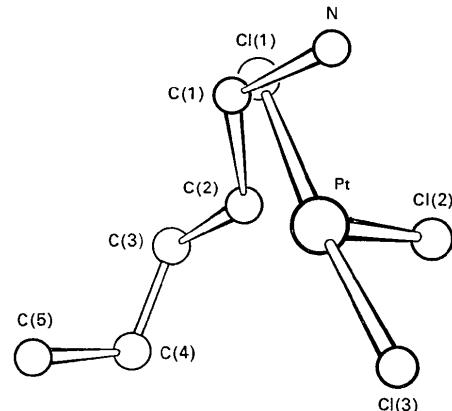
was included in the structure-factor calculation. The final R index is 0.040 ($R_w = 0.047$). The function minimized during the refinement was $\sum w(|F_o| - |F_c|)^2$ with weights $w = 4F_o^2/\sigma(F_o^2)$.

Atomic form factors given by Cromer & Mann (1968) for platinum, chlorine, nitrogen and carbon, and by Hanson, Herman, Lea & Skillman (1964) for hydrogen were used. The effects of anomalous dispersion were included in the calculated structure factors; the values of $\Delta f'$ and $\Delta f''$ for Pt and Cl were those computed by Cromer (1965).

In Table 1 the positional and thermal parameters for the non-hydrogen atoms are reported; the idealized positions of the hydrogen atoms are given in Table 2. Bond lengths and angles are given in Table 3. A list of structure amplitudes is obtainable.*

Table 2. *Idealized coordinates* ($\times 10^3$) *and isotropic temperature factors for the hydrogen atoms*

	Bonded to	x	y	z	B (Å^2)
H(1)	N	369	−202	−340	4·3
H(2)	N	445	−47	−311	4·3
H(3)	N	402	−156	−190	4·3
H(4)	C(1)	246	12	−357	4·6
H(5)	C(1)	203	−97	−237	4·6
H(6)	C(2)	334	186	−212	4·3
H(7)	C(3)	95	113	−109	4·1
H(8)	C(4)	220	383	−72	4·9
H(9)	C(4)	104	324	10	4·9
H(10)	C(5)	43	494	−159	6·2
H(11)	C(5)	104	382	−268	6·2
H(12)	C(5)	−12	323	−185	6·2

Discussion. Trichloro-(*trans*-pent-2-enylammonium)platinum(II) is a zwitterionic platinum-olefin complex. A perspective view of the molecule is shown in Fig. 1. Platinum is four-coordinated, in a square-planar configuration, by the three chlorine atoms and the olefinic double bond of the pentenylammonium cation. The best plane ($-0.749x + 0.524y + 0.406z + 2.288 = 0$, in

Fig. 1. Trichloro-(π-*trans*-pent-2-enylammonium)platinum(II). A perspective view of the molecule.

Table 3. *Bond lengths and angles in trichloro-(trans-pent-2-enylammonium)platinum*(II)

Estimated standard deviations are given in parentheses. MP defines the midpoint of the olefinic double bond.

Pt——Cl(1)	2·321 (4) Å	Cl(1)–Pt——Cl(2)	88·8 (1)°
Pt——Cl(2)	2·316 (4)	Cl(1)–Pt——Cl(3)	174·5 (1)
Pt——Cl(3)	2·312 (5)	Cl(1)–Pt——MP	92·3
Pt——MP	2·02	Cl(2)–Pt——Cl(3)	88·0 (2)
Pt——C(2)	2·097 (14)	Cl(2)–Pt——MP	171·3
Pt——C(3)	2·183 (14)	Cl(3)–Pt——MP	91·5
N——C(1)	1·476 (20)	C(2)–Pt——C(3)	38·1 (5)
C(1)–C(2)	1·543 (21)	N——C(1)–C(2)	112·6 (12)
C(2)–C(3)	1·400 (20)	C(1)–C(2)–C(3)	119·6 (13)
C(3)–C(4)	1·468 (23)	C(2)–C(3)–C(4)	126·8 (14)
C(4)–C(5)	1·530 (25)	C(3)–C(4)–C(5)	109·7 (14)

terms of monoclinic coordinates) was calculated through the platinum and the chlorine atoms. The midpoint of the olefinic double bond is 0·42 Å out of this principal plane, and the double bond makes an angle of 76·8° with it. The large deviation of the double bond from the direction perpendicular to the principal plane is mostly due to a tilt around the Pt–olefin bond: in fact the plane through Pt, C(2) and C(3) makes a dihedral angle of 78·6° with the principal plane. The dimensions and the geometry around the metal atom in trichloro-(*trans*-pent-2-enylammonium)platinum(II) are similar to those observed in other platinum (II)–olefin complexes (Spagna & Zambonelli, 1971 and 1972, and references quoted therein). No *trans*-bond-lengthening effect is apparent in the present case. The ligand pentenylammonium cation has lost the pure *trans* configuration around its double bond: in Table 4 the internal rotation angles of the pentenylammonium cation are reported.

Table 4. *Internal rotation angles of the trans-pent-2-enylammonium cation coordinated to the platinum*(II)

The convention of Klyne & Prelog (1960) is adopted. Estimated standard deviations are given in parentheses.

N——C(1)–C(2)–C(3)	− 151·1 (14)°
C(1)–C(2)–C(3)–C(4)	− 143·7 (15)
C(2)–C(3)–C(4)–C(5)	95·1 (18)

The optically active complex (Paiaro & Panunzi, 1964) is present in the crystal in the racemic form, the C(2)(*R*):C(3)(*R*) and C(2)(*S*):C(3)(*S*) configurations (Cahn, Ingold & Prelog, 1956) being related by the glide plane: the coordinates in the Tables and the drawing in the figure refer to the molecule with C(2)(*S*):C(3)(*S*) configuration.

Probably the hydrogen atoms of the $-NH_3^+$ group take part in a N–H···Cl hydrogen bond network: in fact short contacts between the nitrogen atom and the chlorine atoms are observed (Table 5).

Table 5. *Intermolecular contacts between nitrogen and chlorine atoms shorter than the sum of the ionic radii* (3·62 Å)

N···Cl(2^i)		3·09 Å
N···Cl(2^{ii})		3·16
N···Cl(1^i)		3·50
N···Cl(1^{iii})		3·52
N···Cl(3^{ii})		3·54
i	$1-x,$	$-y, \quad -z$
ii	$x, \quad -\frac{1}{2}-y,$	$\frac{1}{2}+z$
iii	$1-x,$	$\frac{1}{2}+y, \quad -\frac{1}{2}-z$

The technical part of this work was performed by A. Tamburini.

References

Bouis, M. (1928). *Ann. Chim.* **9**, 402–465.

Cahn, C. K., Ingold, C. K. & Prelog, V. (1956). *Experientia*, **12**, 81–94.

Cromer, D. T. (1965). *Acta Cryst.* **18**, 17–23.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321.

Denning, R. G. & Venanzi, L. M. (1963). *J. Chem. Soc.* pp. 3241–3247.

Furnas, T. C. (1957). *Single-Crystal Orienter Instruction Manual.* Milwaukee: General Electric Company.

Hanson, H. P., Herman, F., Lea, J. D. & Skillman, S. (1964). *Acta Cryst.* **17**, 1040–1044.

Klyne, W. & Prelog, V. (1960). *Experientia*, **16**, 521–523.

North, A. C. T., Phillips, D. C. & Matthews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Paiaro, G. & Panunzi, A. (1964). *J. Amer. Chem. Soc.* **86**, 5148–5152.

Sheenan, J. C. & Bolhofer, W. A. (1950). *J. Amer. Chem. Soc.* **72**, 2786–2788.

Spagna, R. & Zambonelli, L. (1971). *J. Chem. Soc.* (*A*). pp. 2544–2549.

Spagna, R. & Zambonelli, L. (1972). *Acta Cryst.* B**28**, 2760–2765.