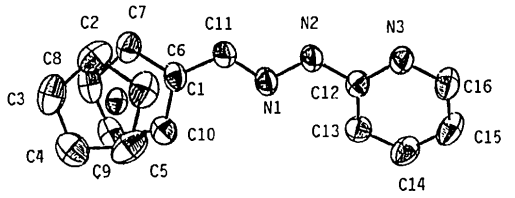

Fig. 1. An *ORTEP* (Johnson, 1965) diagram of the title compound.

were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Computations were carried out on a MicroVAX using *SHELXS*86 (Sheldrick, 1986), and *MolEN* (Enraf–Nonius, 1990). Diagrams were drawn with *ORTEP* (Johnson, 1965).

Discussion. Bond lengths and angles are listed in Table 2. The atomic numbering scheme is shown in Fig. 1. The distance from Fe to the centre of the cyclopentadienyl ring is 1.644 (1) Å for the unsubstituted ring and 1.649 (1) Å for the other. The average Fe—C distance is 2.042 (4) Å.

We have previously reported the structure of $[Fe\{C_5H_4C(CH_3)NNHC_5NH^+\}_2][CH_3COO^-]_2$ (Houlton, Dilworth, Roberts, Silver & Drew, 1990). In this salt, the average C=N distance is 1.289 (19) Å and the average N—N distance is 1.384 (13) Å, which are longer than in (1). However, owing to the large errors reported these differences cannot be considered significant. In comparison with similar compounds (Houlton, Dilworth, Roberts, Silver & Drew, 1990), the N—N bond length in (1) at 1.368 Å is at the shorter end of the observed range, indicating some double-bond character. This is consistent with the trigonal geometry of N(2) indicating a delocalization of the lone pair. This feature explains the observed coordination chemistry of ligands of this type, in that the N(1)- and N(3)-type atoms and not N(2) are involved in ligation (Chiswell & Lions, 1964).

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{Bis[2-(4-ethoxyphenyltelluro)ethyl]amino-*N*,*Te*,*Te'*}chloroplatinum(II) Chloride Trichloromethane Monohydrate

By John E. Drake* and Jane H. E. Bailey

Department of Chemistry and Biochemistry, University of Windsor, Windsor, Ontario, Canada N9B 3P4

and Ajai K. Singh* and Vinod Srivastava

*Department of Chemistry, Indian Institute of Technology, New Delhi-*110 016, *India*

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Abstract. $[PtCl\{(C_{10}H_{13}OTe)_2NH\}]Cl.CHCl_3.H_2O$, $M_r = 972.2$, triclinic, $P\bar{1}$, $a = 10.590\,(7)$, $b = 17.606\,(6)$, $c = 8.493\,(3)$ Å, $\alpha = 90.10\,(3)$, $\beta = 102.22\,(5)$, $\gamma = 106.85\,(4)°$, $V = 1478\,(3)$ Å^3, $Z = 2$, $D_m = 2.20$, $D_x = 2.18$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 7.218$ mm^{-1}, $F(000) = 908$, $T = 297\,(1)$ K, final $R = 0.0429$, $wR = 0.0446$ for 2910 unique observed reflections, $S = 1.38$. The Te atoms are coordinated in a *trans* fashion with the Pt—Te bond lengths of 2.557 (2) and 2.564 (2) Å being

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* To whom correspondence should be addressed.

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slightly shorter than the length previously reported for a *trans* Pt—Te complex of 2.575 (1) Å and considerably longer than the 2.514 (1) Å reported for a Pt—Te bond *trans* to a Pt—Cl bond. The Pt—Cl bond length of 2.306 (4) Å is as to be expected for such a bond *trans* to a Pt—N bond, as is the Pt—N bond length of 2.05 (1) Å.

Introduction. This paper is a follow up to a synthetic study in which it was proposed that a compound analysed as $PtCl_2.[\{(C_2H_5OC_6H_4)TeCH_2CH_2\}_2NH]$ might have an unusual five-coordinated platinum(II) structure (Singh, Srivastava & Khandelwal, 1990*a*). Despite the recent interest in multidentate hybrid Te donors (Singh, Srivastava & Khandelwal, 1990*b*; Singh & Srivastava, 1990), only three structures have been reported containing Pt—Te bonds (Gysling & Luss, 1984; Kelly, Slawin, Williams & Woolins, 1990; Singh, Srivastava, Dhingra, Drake & Bailey, 1992). This is the first structure reported of a complex containing a tridentate (*Te*, *N*, *Te*) donor molecule.

Experimental. Preparation was as described recently by Singh, Srivastava & Khandelwal (1990*a*) followed by crystallization by slow evaporation of a $CHCl_3/CH_3CN$ solution to yield pale-yellow crystals; density measured by flotation in CCl_4/CH_3I. A crystal of size 0.48 × 0.52 × 0.45 mm was used for data collection on a Rigaku AFC-GS diffractometer with highly oriented graphite monochromator. Cell parameters were determined from 25 strong reflections ($30.14 < 2\theta < 32.44°$). Data were collected at 297 (1) K using the ω–2θ scan technique to a maximum 2θ value of 50.0°; ω scans had average width at half height of 0.28° with take-off angle of 6.0°; scans of $(1.52 + 0.30\tan\theta)°$ width had rate $32.0°\ \text{min}^{-1}$. Weak reflections [$I < 10.0\sigma(I)$] were rescanned (maximum of two rescans) and counts accumulated to assure good counting statistics. The diameter of the incidence-beam collimator was 0.5 mm and crystal to detector distance was 250.0 mm. Intensities of three representative reflections measured every 150 reflections remained constant throughout data collection indicating crystal and electronic stability (no decay correction was applied). Space group $P\bar{1}$ was used, assumed correct because of successful refinement. 5517 reflections were collected, of which 5209 were unique ($R_{\text{int}} = 0.110$; $4 < 2\theta < 50°$; $h = 0$ to 12, $k = -20$ to 19, $l = -10$ to 9). The linear absorption coefficient for Mo $K\alpha$ radiation was $7.218\ \text{mm}^{-1}$; an empirical absorption correction, based on azimuthal scans of several reflections, was applied with transmission factors 0.55–1.00. Data were corrected for Lorentz and polarization effects. The position of the Pt atom was determined from a sharpened Patterson synthesis (*SHELX*; Sheldrick & Egert, 1986); positions of remaining non-H atoms were determined from subsequent difference Fourier maps. During refinement, all non-H atoms were treated anisotropically minimizing $\sum w(|F_o| - |F_c|)^2$; all H atoms attached to C atoms were included in ideal positions (C—H = 0.95 Å) with isotropic thermal parameters set at 1.2 times that of the C atoms to which they are attached; the H atom on N was located from difference maps and treated isotropically, with positional parameters fixed at the observed values from the penultimate least-squares cycle to ensure convergence; both H atoms of H_2O were not located successfully despite utilization of a 2θ cut-off of 28° after complete refinement. The final cycle of full-matrix least-squares refinement based on 2910 observed reflections [$I > 3.00\sigma(I)$] and 298 variable parameters, converged (largest shift/e.s.d. = 0.0003) with $R = 0.0429$ and $wR = 0.0446$ where $w = 4F_o/\sigma^2(F_o^2)$; the final difference map had no features of chemical significance, and had maximum and minimum peaks at 1.24 and $-1.29\ \text{e}\ \text{Å}^{-3}$, respectively. Neutral-atom scattering factors were taken from Cromer & Waber (1974) and anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964); values of f' and f'' were those of Cromer (1974). All calculations used the *TEXSAN* crystallographic software package (Molecular Structure Corporation, 1985), including *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978).

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* ($Å^2 \times 10^2$)

$$B_{\text{eq}} = (8\pi^2/3)\textstyle\sum_{i=1}^{3}\sum_{j=1}^{3} U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Pt	0.48391 (7)	0.67752 (4)	0.43593 (7)	2.72 (2)
Te(1)	0.6772 (1)	0.69534 (6)	0.2897 (1)	3.19 (4)
Te(2)	0.2790 (1)	0.66579 (6)	0.5602 (1)	3.78 (5)
Cl(1)	0.6316 (5)	0.7447 (2)	0.6671 (4)	4.6 (2)
Cl(2)	0.3158 (5)	0.4960 (3)	0.6681 (5)	5.5 (2)
Cl(3)	0.0023 (5)	0.3735 (3)	1.2087 (6)	6.4 (2)
Cl(4)	0.0430 (5)	0.2535 (3)	1.0139 (7)	7.2 (3)
Cl(5)	0.1573 (5)	0.4166 (3)	0.9651 (5)	4.9 (2)
O(1)	0.676 (1)	1.0334 (6)	0.055 (1)	4.7 (5)
O(2)	0.159 (1)	0.9780 (7)	0.299 (1)	5.6 (6)
O(3)	0.369 (1)	0.4617 (6)	0.325 (1)	4.7 (5)
N	0.356 (1)	0.6162 (6)	0.230 (1)	2.9 (5)
C(1)	0.402 (2)	0.6385 (8)	0.076 (2)	3.5 (6)
C(2)	0.536 (2)	0.629 (1)	0.080 (2)	4.1 (7)
C(3)	0.205 (1)	0.6140 (9)	0.206 (2)	3.7 (6)
C(4)	0.151 (2)	0.589 (1)	0.357 (2)	4.6 (7)
C(5)	0.680 (1)	0.8108 (8)	0.214 (2)	2.9 (5)
C(6)	0.683 (1)	0.830 (1)	0.052 (2)	3.6 (6)
C(7)	0.683 (2)	0.906 (1)	0.009 (2)	3.8 (6)
C(8)	0.677 (1)	0.9617 (9)	0.117 (2)	3.4 (6)
C(9)	0.673 (2)	0.9434 (8)	0.277 (2)	3.7 (6)
C(10)	0.672 (1)	0.8693 (8)	0.325 (2)	3.0 (5)
C(11)	0.668 (2)	1.095 (1)	0.154 (2)	5.8 (8)
C(12)	0.675 (2)	1.167 (1)	0.059 (2)	6.3 (9)
C(13)	0.234 (2)	0.7696 (9)	0.467 (2)	3.6 (6)
C(14)	0.329 (2)	0.831 (1)	0.420 (2)	5.9 (9)
C(15)	0.299 (2)	0.899 (1)	0.367 (2)	6 (1)
C(16)	0.177 (2)	0.907 (1)	0.354 (2)	4.0 (7)
C(17)	0.078 (2)	0.848 (1)	0.404 (2)	4.9 (8)
C(18)	0.109 (2)	0.778 (1)	0.460 (2)	4.5 (7)
C(19)	0.036 (2)	0.988 (1)	0.274 (2)	7 (1)
C(20)	0.040 (2)	1.069 (1)	0.219 (3)	6.3 (9)
C(21)	0.117 (2)	0.350 (1)	1.109 (2)	4.5 (7)

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

Pt—Te(1)	2.557 (2)	Pt—Te(2)	2.564 (2)
Pt—Cl(1)	2.306 (4)	Pt—N	2.05 (1)
Te(1)—C(2)	2.15 (2)	Te(2)—C(4)	2.14 (2)
Te(1)—C(5)	2.13 (1)	Te(2)—C(13)	2.13 (1)
N—C(1)	1.51 (2)	N—C(3)	1.55 (2)
C(1)—C(2)	1.46 (2)	C(3)—C(4)	1.53 (2)
C(5)—C(6)	1.42 (2)	C(13)—C(14)	1.36 (2)
C(5)—C(10)	1.42 (2)	C(13)—C(18)	1.36 (2)
C(6)—C(7)	1.38 (2)	C(14)—C(15)	1.37 (2)
C(7)—C(8)	1.37 (2)	C(15)—C(16)	1.33 (2)
C(8)—C(9)	1.40 (2)	C(16)—C(17)	1.40 (2)
C(9)—C(10)	1.37 (2)	C(17)—C(18)	1.42 (2)
C(11)—C(12)	1.50 (2)	C(19)—C(20)	1.49 (2)
O(1)—C(8)	1.37 (2)	O(2)—C(16)	1.38 (2)
O(1)—C(11)	1.41 (2)	O(2)—C(19)	1.35 (2)
N—H(N)	1.0 (1)	C(21)—Cl(3)	1.76 (2)
C(21)—Cl(4)	1.76 (1)	C(21)—Cl(5)	1.73 (2)
Te(1)···Cl′(2)$^-$	3.408 (4)	Te(2)···Cl(2)$^-$	3.235 (4)
Cl(2)$^-$···O(3)	3.17 (1)	Cl(2)$^-$···O′(3)	3.19 (1)
O(3)···N	2.87 (1)	O(3)···C″(21)	3.04 (2)

Te(1)—Pt—Te(2)	174.55 (4)	Cl(1)—Pt—N	178.6 (3)
Te(1)—Pt—Cl(1)	91.4 (1)	Te(2)—Pt—Cl(1)	91.7 (1)
Te(1)—Pt—N	87.7 (3)	Te(2)—Pt—N	89.2 (3)
Pt—Te(1)—C(2)	89.8 (4)	Pt—Te(2)—C(4)	89.8 (4)
Pt—Te(1)—C(5)	96.0 (4)	Pt—Te(2)—C(13)	97.0 (4)
C(2)—Te(1)—C(5)	96.8 (5)	C(4)—Te(2)—C(13)	94.1 (6)
Te(1)—C(2)—C(1)	109 (1)	Te(2)—C(4)—C(3)	109 (1)
N—C(1)—C(2)	112 (1)	N—C(3)—C(4)	111 (1)
Te(1)—C(5)—C(6)	121 (1)	Te(2)—C(13)—C(14)	123 (1)
Te(1)—C(5)—C(10)	120 (1)	Te(2)—C(13)—C(18)	119 (1)
C(6)—C(5)—C(10)	119 (1)	C(14)—C(13)—C(18)	118 (1)
C(5)—C(6)—C(7)	119 (1)	C(13)—C(14)—C(15)	121 (2)
C(6)—C(7)—C(8)	122 (1)	C(14)—C(15)—C(16)	122 (2)
C(7)—C(8)—C(9)	120 (1)	C(15)—C(16)—C(17)	119 (2)
C(8)—C(9)—C(10)	120 (1)	C(16)—C(17)—C(18)	118 (1)
C(9)—C(10)—C(5)	120 (1)	C(17)—C(18)—C(13)	121 (2)
C(8)—O(1)—C(11)	120 (1)	C(16)—O(2)—C(19)	119 (1)
O(1)—C(11)—C(12)	108 (2)	O(2)—C(19)—C(20)	111 (2)
C(1)—N—C(3)	109 (1)	C(3)—N—H(N)	105
C(1)—N—H(N)	98	Pt—N—H(N)	112
C(1)—N···O(3)	113.6 (8)	Pt—N···O(3)	95.8 (4)
N—H(N)···O(3)	153	C(3)—N···O(3)	106.4 (8)
Cl(2)$^-$···O(3)···N	91.1 (4)	Cl′(2)$^-$···O(3)···N	93.3 (4)
Cl(2)$^-$···O(3)···H(N)	100	Cl′(2)···O(3)···H(N)	93
Cl(3)—C(21)—Cl(4)	108.9 (9)	Cl(4)—C(21)—Cl(5)	108.9 (9)
Cl(3)—C(21)—Cl(5)	111.1 (9)		

Symmetry code: (′) $1 - x$, $1 - y$, $1 - z$; (″) x, y, $z - 1$.

Discussion. The final atomic coordinates for non-H atoms are given in Table 1 and important distances and angles in Table 2.*

The crystal structure analysis of [PtCl{Te($C_6H_4OC_2H_5$)CH_2CH_2}$_2$NH]$^+$.Cl$^-$ indicates that the unique tridentate (*Te*, *N*, *Te*) donor gives an ionic square-planar platinum complex ion rather than the unusual five-coordinate complex predicted earlier. The square-planar arrangement can be seen clearly in the *ORTEP*II diagram (Fig. 1), while the presence of $CHCl_3$ and H_2O in the crystal lattice can be seen in the unit-cell diagram (Fig. 2). The Pt—Te bond lengths of 2.557 (2) and 2.564 (2) Å are slightly shorter than those reported for [Pt{PhTe(*o*-$PPh_2C_6H_4$)}$_2$][Pt(SCN)$_4$].2DMF where the Pt—Te bonds are 2.575 (1) Å and very similar to those reported for [Pt(TeSN_2H)(PMe$_2$Ph)$_2$][BF$_4$], where the Pt—Te bond *trans* to a Pt—P bond is 2.553 (1) Å. By contrast the Pt—Te bond is much shorter, 2.514 (1) Å, in *cis*-PtCl$_2$[Te($C_6H_4OC_2H_5$)CH_2CH_2-(CH_3)S] where it is *trans* to a Cl atom. Thus, the *trans* influence of the Te atom appears to be very similar to that of phosphine. The Cl and N atoms are mutually *trans* and the bond lengths of 2.306 (4) and 2.05 (1) Å, respectively, for Pt—Cl and Pt—N are as expected. The bond angles about each Te atom range from 89.8 (4) to 97.0 (4)° compared to 96.7 (2) to 102.3 (1)° for the [Pt{PhTe(*o*-$PPh_2C_6H_4$)}]$^{2+}$ cation, suggesting less steric hindrance about Te in the (*Te*, *N*, *Te*) donor. The P—Pt—Te angle in the [Pt{PhTe(*o*-$PPh_2C_6H_4$)}$_2$]$^{2+}$ cation is given as 88.50 (3)° which is the same as the average, 88.5 (8)°, of the comparable N—Pt—Te angles in the [PtCl{Te($C_6H_4OC_2H_5$)CH_2CH_2}$_2$NH]$^+$ cation. Thus, this represents another example of Pt favoring a square-planar environment over the five-coordination required for a neutral complex.

The closest contact of the Cl$^-$ ion, designated as Cl(2)$^-$, to an atom in the cation is 3.235 (4) Å from Te(2) in the same asymmetric unit. The packing in the crystal is such that it is also only 3.408 (4) Å from Te(1) in an adjacent cation. Thus, the two cations are arranged with the square planes facing each other and with Cl$^-$ ions essentially bridging two Te atoms. The association of the Cl ion with Te is emphasized by the fact that these Te···Cl distances are similar to those found in Et$_3$TeCl, which has a

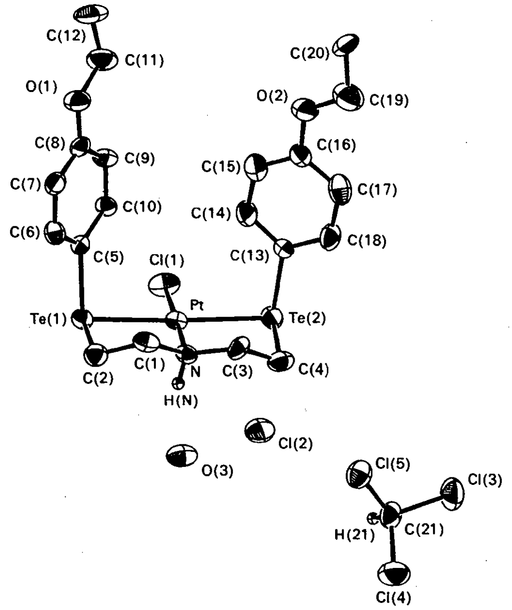

Fig. 1. *ORTEP*II (Johnson, 1976) plot of [PtCl{Te-($C_6H_4OC_2H_5$)CH_2CH_2}$_2$NH]$^+$.Cl$^-$.$CHCl_3$.H_2O. The atoms are drawn with 40% probability ellipsoids. H atoms are omitted for clarity except for those attached to N and the C atom of the $CHCl_3$ group.

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* 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 55643 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1001]

distorted cubane-type structure, in which four Et_3Te^+ groups are linked together by four Cl^- ions at a Te···Cl distance of 3.448 (4) Å (Chadha & Drake, 1986). These contacts are relatively short for secondary interactions and are considerably less than the sum of the van der Waals radii of 3.81 Å. The analogy extends even further because in Et_3TeCl, two angles are subtended involving C—Te···Cl of 80.2 (6) and 165.3 (6)° which are remarkably similar to those found in this Pt complex of 79.2 (4) and 167.9 (4)°.

The environment about the O atom of the water molecule, designated as O(3), is of interest although unfortunately it was not possible to locate the H atoms. The O atoms, O(3), are located approximately equidistant between two $Cl(2)^-$ ions of two adjacent independent units at 3.17 (1) and 3.19 (1) Å respectively, subtending an angle of 111.9 (4)° close to tetrahedral. The distances are slightly longer than the O···Cl distances of 2.91–2.95 Å in $HCl.H_2O$ and similar systems (Wells, 1975). The O(3)···N distance of 2.871 (1) Å suggests the presence of a relatively strong hydrogen bond, comparable to that suggested for NH_4OCCH (Nahringbauer, 1968) and $CO(NH_2)$ (Worsham, Levy & Peterson, 1957). The located H(N) atom gives an O(3)—H(N) distance of 1.96 Å and an N—H(N)···O(3) angle of 153°. There are no other distances less than 3.0 Å involving the O(3) atom, other than O(3)···H″(21) which is 2.4 Å. The resulting distance to the C atom of this $CHCl_3$ molecule in the lattice, O(3)—C″(21), is 3.04 (2) Å, just outside the range normally associated with C—H···O hydrogen bonding. The C—Cl bond lengths in the $CHCl_3$ molecule are within the range expected, 1.73 (2)–1.76 (2) Å, as are the angles of 108.9 (9)–111.1 (9)°.

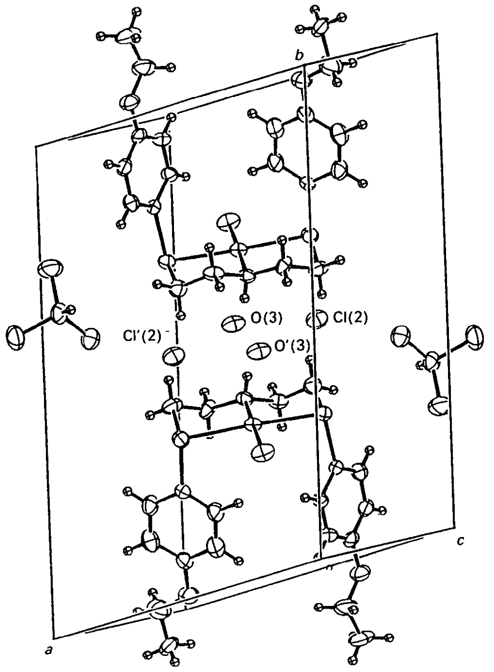

Fig. 2. Unit-cell packing of $[PtCl\{Te(C_6H_4OC_2H_5)CH_2CH_2\}_2NH]^+.Cl^-.CHCl_3.H_2O$. The O atoms of the water molecules are labelled O(3), and the Cl ions are labelled $Cl(2)^-$, for clarity.

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