

TiO courtes pointent alternativement de part et d'autre du plan de la couche (Fig. 1). L'empilement de ces couches ménage de larges cavités dans lesquelles viennent s'insérer les ions césium.

La coordinence des atomes de césium est très déformée. Pour l'atome Cs(1), on relève onze distances Cs—O inférieures à 4 Å qui s'étagent entre 2,998 et 3,762 Å. Pour l'atome Cs(2), les neuf distances Cs—O inférieures à 4 Å se distribuent entre 2,985 et 3,552 Å. Ces distances sont toutes égales ou supérieures à la somme des rayons ioniques des ions Cs<sup>+</sup> et O<sup>2-</sup>. Les distances qui impliquent la présence de l'oxygène apical de la pyramide TiO<sub>5</sub> valent notamment 3,407 Å pour Cs(1)—O(6) et 3,211 Å pour Cs(2)—O(6) et rendent de ce fait parfaitement plausible l'existence d'une liaison multiple TiO. Les distances les plus courtes entre deux ions césium Cs(1)—Cs(2) sont de 4,449 Å.

Comme dans les matériaux de structure type KTiO(PO<sub>4</sub>), les ions alcalins, bien que les plus lourds, possèdent les facteurs d'agitation thermique les plus élevés, mais pour Cs<sub>2</sub>TiO(P<sub>2</sub>O<sub>7</sub>), les valeurs des déplacements atomiques moyens le long des axes des ellipsoïdes sont assez bien groupées [0,149, 0,158 et 0,172 Å pour Cs(1); 0,138, 0,166 et 0,181 Å pour Cs(2)]. L'inspection des distances interatomiques des oxygènes des polyèdres du césium ne permet pas cependant de prévoir avec certitude l'existence possible de conductivité ionique. Ce type structural vient d'être récemment décrit par Lii & Wang (1989) qui ont résolu la structure de Rb<sub>2</sub>VO(P<sub>2</sub>O<sub>7</sub>) avec laquelle le composé Cs<sub>2</sub>TiO(P<sub>2</sub>O<sub>7</sub>) est isotype.

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## Structure of 2-Carboxypyridinium Trichloro(dimethyl sulfoxide)platinate(II)

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**Abstract.** C<sub>6</sub>H<sub>6</sub>NO<sub>2</sub><sup>+</sup>[PtCl<sub>3</sub>(C<sub>2</sub>H<sub>6</sub>OS)]<sup>-</sup>, [H<sub>2</sub>pic]-[PtCl<sub>3</sub>(C<sub>2</sub>H<sub>6</sub>OS)], H<sub>2</sub>pic = 2-carboxypyridinium, *M*, = 503.70, orthorhombic, *Pbca*, *a* = 9.994 (2), *b* = 21.268 (3), *c* = 13.200 (3) Å, *V* = 2805.7 (9) Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 2.385 Mg m<sup>-3</sup>,  $\bar{\lambda}$ (Mo *K*α) = 0.71069 Å,  $\mu$  = 10.821 mm<sup>-1</sup>, room temperature, *F*(000) = 1888, *R*

= 0.031 for 1447 independent observed reflections. The structure consists of [PtCl<sub>3</sub>(Me<sub>2</sub>SO)]<sup>-</sup> complex anions and protonated 2-carboxypyridinium cations linked through a hydrogen bond involving the protonated pyridinium N atom and a Cl atom of the platinum complex.

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**Introduction.** Recently we reported that *trans*-(*N,S*)-[Pt(pic)(Me<sub>2</sub>SO)Cl] (pic = 2-pyridinecarboxylato), in the presence of Cl<sup>-</sup>, isomerizes to *cis*-(*N,S*)[Pt(pic)(Me<sub>2</sub>SO)Cl] (Annibale, Cattalini, Canovese, Pitteri, Tiripicchio, Tiripicchio Camellini & Tobe, 1986). By reacting the *trans* isomer with an excess of HCl, in ethanol solution, the title compound (1) was obtained and recrystallized by slow evaporation of an ethanol-acetic acid (1:1 v/v) solution. In contrast, the *cis* isomer reacts with HCl to give [Pt(pic)Cl<sub>2</sub>]<sup>-</sup> and Me<sub>2</sub>SO.

**Experimental.** A prismatic yellow crystal of (1) of approximate dimensions 0.18 × 0.27 × 0.30 mm, Siemens AED diffractometer, Nb-filtered Mo K $\alpha$  radiation, 25 reflections (10 <  $\theta$  < 15°) for accurate unit-cell parameters;  $\theta/2\theta$  scan mode, 4937 reflections with 3 <  $\theta$  < 27°, -12 <  $h$  < 12, 0 <  $k$  < 21, 0 <  $l$  < 16, 1447 independent reflections with  $I > 2\sigma(I)$  considered observed, no significant intensity deterioration of the standard reflection (270), Lp corrections. Corrections for absorption effects were applied empirically (maximum and minimum transmission factors 1.188 and 0.902, respectively; Walker & Stuart, 1983; Ugozzoli, 1987). Patterson and Fourier methods; full-matrix least-squares refinement on  $F_o$ , anisotropic thermal parameters for non-H atoms; all the H atoms were clearly localized in the final  $\Delta F$  map and introduced in the final structure-factor calculation, but without refinement of their positions and assuming a common  $U_{iso}$  of 0.08 Å<sup>2</sup> (SHELX76; Sheldrick, 1976). Final  $R = 0.031$ ,  $wR = 0.044$ ;  $w = K[\sigma^2(F_o) + gF_o^2]^{-1}$  with  $K = 0.835$  and  $g = 0.005$  at convergence;  $(\Delta/\sigma)_{max} = 0.65$ , final  $\Delta\rho$  values < |0.8| e Å<sup>-3</sup>; atomic scattering factors (anomalous dispersion of Pt and S atoms) from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–101, 149–150); calculations performed on the CRAY X-MP-12 computer of the Centro di Calcolo Elettronico Interuniversitario dell'Italia Nord Ori-

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters for the non-H atoms, with e.s.d.'s in parentheses

	$B_{eq} = (8\pi^2/3) \text{ trace } U.$			
	x	y	z	$B_{eq}(\text{Å}^2)$
Pt	1403 (1)	4153 (1)	5028 (1)	2.03 (1)
Cl(1)	-163 (3)	3593 (2)	5914 (3)	3.93 (9)
Cl(2)	2611 (3)	4235 (1)	6500 (2)	3.42 (8)
Cl(3)	3066 (3)	4689 (2)	4228 (3)	4.47 (10)
S	264 (3)	4086 (1)	3614 (2)	2.36 (7)
O(1)	617 (9)	3540 (4)	2949 (6)	3.38 (24)
O(2)	3898 (8)	2006 (4)	8201 (6)	3.10 (22)
O(3)	3118 (10)	2546 (5)	6871 (7)	5.18 (31)
N	1590 (10)	3253 (5)	8095 (8)	3.05 (30)
C(1)	-1492 (10)	4049 (6)	3797 (10)	3.22 (37)
C(2)	406 (14)	4777 (6)	2865 (10)	3.69 (35)
C(3)	3118 (13)	2419 (6)	7774 (9)	3.29 (35)
C(4)	2200 (10)	2750 (5)	8485 (9)	2.40 (29)
C(5)	2000 (11)	2548 (6)	9456 (9)	3.07 (32)
C(6)	1114 (14)	2925 (7)	10033 (10)	4.05 (36)
C(7)	478 (14)	3457 (7)	9614 (12)	4.05 (39)
C(8)	737 (13)	3609 (6)	8633 (11)	3.93 (38)

Table 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses in [H<sub>2</sub>pic][PtCl<sub>3</sub>(Me<sub>2</sub>SO)]

Pt—Cl(1)	2.288 (4)	C(3)—O(2)	1.303 (15)
Pt—Cl(2)	2.294 (3)	C(3)—O(3)	1.222 (15)
Pt—Cl(3)	2.275 (4)	C(3)—C(4)	1.489 (17)
Pt—S	2.191 (3)	C(4)—N	1.335 (15)
S—O(1)	1.498 (9)	C(4)—C(5)	1.367 (17)
S—C(1)	1.773 (10)	C(5)—C(6)	1.417 (18)
S—C(2)	1.777 (13)	C(6)—C(7)	1.411 (21)
		C(7)—C(8)	1.360 (21)
		C(8)—N	1.343 (17)
Cl(1)—Pt—Cl(2)	88.1 (1)	O(2)—C(3)—O(3)	124.8 (12)
Cl(2)—Pt—Cl(3)	88.3 (1)	C(4)—C(3)—O(2)	114.5 (10)
Cl(3)—Pt—S	91.0 (1)	C(4)—C(3)—O(3)	120.7 (11)
Cl(1)—Pt—S	92.7 (1)	N—C(4)—C(5)	123.2 (10)
Pt—S—O(1)	115.3 (4)	C(4)—C(5)—C(6)	114.7 (11)
Pt—S—C(1)	113.6 (4)	C(5)—C(6)—C(7)	121.7 (12)
Pt—S—C(2)	112.3 (5)	C(6)—C(7)—C(8)	118.6 (13)
O(1)—S—C(1)	106.2 (5)	C(7)—C(8)—N	119.4 (12)
O(1)—S—C(2)	107.2 (6)	C(4)—N—C(8)	122.5 (11)
C(1)—S—C(2)	101.0 (6)		

entale, Casalecchio (Bologna), with financial support from the University of Parma.

**Discussion.** Final atomic coordinates for the non-H atoms are listed in Table 1 and relevant distances and angles in Table 2.\*

The structure consists of [PtCl<sub>3</sub>(Me<sub>2</sub>SO)]<sup>-</sup> anions and 2-carboxypyridinium cations held together by an N—H...Cl hydrogen bond (Fig. 1) [N...Cl(2) = 3.136 (11) Å, N—H(9)—Cl(2) = 167°]. The coordination of the Pt atom is roughly square planar and involves three Cl atoms and the S atom from the Me<sub>2</sub>SO ligand. Pt, Cl(1), Cl(2), Cl(3) and S are

\* 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 53381 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

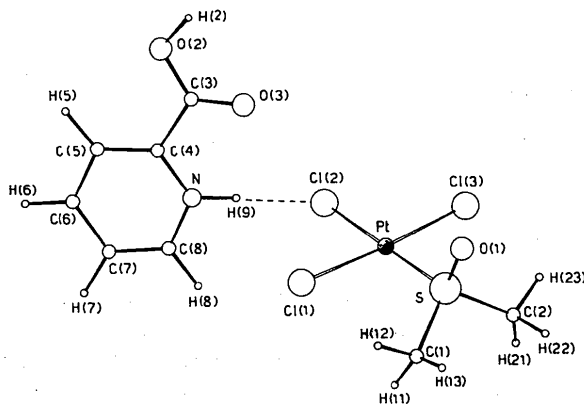


Fig. 1. View of the structure of [H<sub>2</sub>pic][PtCl<sub>3</sub>(Me<sub>2</sub>SO)] with the atomic numbering scheme.

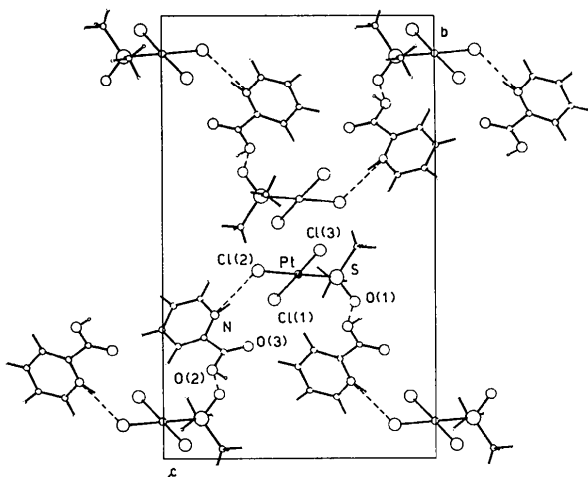


Fig. 2. Projection along [100] of the packing of  $[\text{H}_2\text{pic}][\text{PtCl}_3(\text{Me}_2\text{SO})]$ .

displaced from the mean plane passing through them by  $-0.003$  (2),  $0.029$  (4),  $-0.009$  (2),  $0.030$  (4) and  $-0.008$  (2) Å, respectively. The structural parameters of  $[\text{PtCl}_3(\text{Me}_2\text{SO})]^-$  are quite comparable to those of the same complex in the structure of the potassium salt (2) (Melanson, Hubert & Rochon, 1976) in which the values of the Pt—Cl bonds were

2.302 (6), 2.318 (5) and 2.296 (6) Å, and the Pt—S bond 2.193 (5) Å.

In contrast, some differences in the IR spectra of (1) and (2) have been found. (i) The stretching S—O falls at a lower frequency in (1) with respect to (2) [ $\nu_{\text{S=O}}$ :  $1060\text{ cm}^{-1}$  (1),  $1100\text{ cm}^{-1}$  (2)]; (ii) The stretching Pt—Cl (Cl *trans* to sulfoxide) decreases from  $309\text{ cm}^{-1}$  (1) to  $290\text{ cm}^{-1}$  (2), probably due to the weakness of the bond determined by the hydrogen bond with the 2-carboxypyridinium cation.

The packing of the structure, shown in Fig. 2, is determined by an intermolecular hydrogen bond involving the —OH group of the pyridine-2-carboxylic acid moiety and the O atom of the dimethyl sulfoxide moiety  $[\text{O}(2)\cdots\text{O}(1)]$  ( $x + \frac{1}{2}, \frac{1}{2} - y, 1 - z$ ) = 2.570 (12) Å,  $[\text{O}(2)\cdots\text{H}(2)\cdots\text{O}(1)]$  ( $x + \frac{1}{2}, \frac{1}{2} - y, 1 - z$ ) = 163°.

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## Structure of Dichlorobis(*p*-phenoxyphenyl)tellurium(IV)

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**Abstract.**  $[\text{TeCl}_2(\text{C}_{12}\text{H}_9\text{O})_2]$ ,  $M_r = 536.9$ , monoclinic,  $P2_1/c$ ,  $a = 8.061$  (5),  $b = 21.694$  (5),  $c = 12.606$  (7) Å,  $\beta = 101.5$  (2)°,  $V = 2160$  (2) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.652\text{ cm}^{-3}$ ,  $\text{Mo K}\alpha$ ,  $\lambda = 0.701069$  Å,  $\mu = 16.45\text{ cm}^{-1}$ ,  $F(000) = 1056$ , room temperature,  $R = 0.028$ ,  $wR = 0.030$  for 2626 observed reflections [ $I \geq 2.5\sigma(I)$ ]. The Te atom is in a pseudo trigonal-

bipyramidal configuration with the lone pair in one of the equatorial positions. Distances Te—Cl = 2.506 (1), 2.510 (1) Å; Te—C = 2.107 (3), 2.129 (4) Å; Cl—Te—Cl = 177.21 (1)°; Cl—Te—C = 90.7 (1), 89.2 (1), 88.6 (1), 88.7 (1)°; C—Te—C = 98.7 (1)°.

**Introduction.** Diorganyl ditellurides and diorganyl tellurides constitute an important class of starting

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