

Fig. 2. Environnement de l'atome d'oxygène (premiers et second voisins). Les valeurs des angles interatomiques sont soulignées.

que sans molécules d'eau, l'édifice cristallin ne peut exister.

Si l'on essaie par ailleurs de comparer nos résultats à ceux obtenus sur les fluorostannates alcalins, l'originalité du composé étudié apparaît clairement. Deux d'entre eux seulement cristallisent sous forme hydratée: le sel de potassium $K_2SnF_6 \cdot H_2O$ et celui de lithium $Li_2SnF_6 \cdot 2H_2O$ dont la formule est identique à celle du sel qui nous occupe, avec une structure basée sur l'ion SnF_6^- et sur l'octaèdre $LiF_4(OH_2)_2$

(Marseglia & Brown, 1973); tous les autres sels (Na, Cs...) cristallisent sous forme anhydre.

Les points de similitude sont en fait très rares. Hormis l'ion SnF_6^{2-} , très comparable dans tous les sels, l'absence de molécules d'eau d'hydratation et le fait évident que l'ion triméthylammonium n'est pas un ion alcalin rend toute comparaison impossible.

Retenons simplement l'identité de formule: $M_2SnF_6 \cdot xH_2O$ pour placer ce composé dans la série et notons son originalité: réseau maintenu stable grâce à la présence des molécules d'eau créant des liaisons hydrogènes avec les fluors des ions SnF_6^{2-} ou avec l'azote du triméthylammonium.

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Structure of *trans*-Diiodobis(methyl phenyl telluride)platinum(II)

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Abstract. $[PtI_2(C_7H_8Te)_2]$, $M_r = 888.37$, monoclinic, $I2/a$, $a = 23.807$ (6), $b = 9.548$ (1), $c = 24.192$ (6) Å, $\beta = 136.48$ (2)°, $V = 3786.7$ Å³, $Z = 8$, $D_m = 3.04$ (2), $D_x = 3.116$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 137.2$ cm⁻¹, $F(000) = 3104$, ambient temperature, $R = 0.041$ for 2653 observed reflections [$F > 2\sigma(F)$]. The structure consists of discrete *trans* square-planar molecules [Pt—I 2.604 (1), 2.607 (1); Pt—Te 2.578 (1), 2.586 (1) Å].

Introduction. The coordination chemistry of Te donor ligands has only recently attracted serious study (Gysling, 1982, 1986). In a detailed study

(Kemmitt & Levason, 1990) of the ¹²⁵Te NMR properties of complexes of two ditelluroether ligands, $PhTe(CH_2)_3TePh$ and $o-C_6H_4(TeMe)_2$, the complexes of the analogous monodentate $PhMeTe$ were prepared. Although many Pt and Pd complexes of type $M(R_2Te)_2X_2$ ($M = Pd$ or Pt , $X =$ halogen) exist in both *cis* and *trans* forms in solution (Gysling, 1982), for $Pt(PhMeTe)_2I_2$ only a single geometric isomer was evident from the NMR spectrum, and hence a structural study was undertaken to establish the isomer present in the solid.

Experimental. A dark brown air-stable crystal (0.5 × 0.25 × 0.2 mm), obtained from $CH_2Cl_2-Et_2O$ by vapour diffusion, was mounted in a thin-walled glass

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Table 1. Atomic coordinates and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Pt(1)	0.26473 (2)	0.46961 (5)	0.07865 (2)	37.9 (1)
I(1)	0.16866 (5)	0.25568 (9)	0.02490 (5)	57.2 (5)
I(2)	0.36353 (5)	0.68235 (9)	0.14044 (5)	56.4 (4)
Te(1)	0.13821 (5)	0.61957 (9)	0.01082 (5)	56.5 (4)
Te(2)	0.39783 (5)	0.32424 (9)	0.17329 (5)	51.3 (3)
C(1)	0.1453 (7)	0.6068 (12)	0.1044 (6)	50 (5)
C(2)	0.2152 (7)	0.5707 (15)	0.1804 (6)	65 (7)
C(3)	0.2201 (9)	0.5759 (14)	0.2421 (8)	69 (7)
C(4)	0.1508 (10)	0.6144 (14)	0.2226 (8)	74 (8)
C(5)	0.0818 (12)	0.6443 (18)	0.1455 (12)	107 (12)
C(6)	0.0782 (10)	0.6429 (18)	0.0853 (9)	93 (10)
C(7)	0.1669 (11)	0.8343 (17)	0.0314 (9)	92 (10)
C(8)	0.4136 (7)	0.3443 (13)	0.2732 (6)	53 (6)
C(9)	0.4588 (8)	0.4541 (17)	0.3237 (7)	76 (8)
C(10)	0.4616 (9)	0.4714 (18)	0.3829 (8)	96 (10)
C(11)	0.4267 (8)	0.3909 (17)	0.3936 (7)	72 (8)
C(12)	0.3793 (10)	0.2826 (18)	0.3415 (9)	94 (10)
C(13)	0.3749 (10)	0.2593 (17)	0.2793 (8)	84 (9)
C(14)	0.3693 (9)	0.1077 (15)	0.1552 (8)	74 (7)

Table 2. Selected bond lengths (\AA) and angles ($^\circ$)

Pt(1)—I(1)	2.604 (1)	Pt(1)—Te(1)	2.578 (1)
Pt(1)—I(2)	2.607 (1)	Pt(1)—Te(2)	2.586 (1)
Te(1)—C(1)	2.15 (1)	Te(2)—C(8)	2.17 (1)
Te(1)—C(7)	2.10 (2)	Te(2)—C(14)	2.12 (2)
C—C (minimum)	1.29 (2)	C—C (maximum)	1.45 (2)
C—C (mean)	1.37 (4)		
I(1)—Pt(1)—I(2)	176.6 (1)	I(1)—Pt(1)—Te(2)	95.1 (1)
Te(1)—Pt(1)—Te(2)	167.8 (1)	I(2)—Pt(1)—Te(1)	94.8 (1)
I(1)—Pt(1)—Te(1)	85.4 (1)	I(2)—Pt(1)—Te(2)	83.9 (1)
Pt(1)—Te(1)—C(1)	97.7 (3)	Pt(1)—Te(2)—C(8)	92.9 (3)
Pt(1)—Te(1)—C(7)	111.2 (5)	Pt(1)—Te(2)—C(14)	109.7 (4)
C(1)—Te(1)—C(7)	92.4 (5)	C(8)—Te(2)—C(14)	95.0 (5)
Te(1)—C(1)—C(2)	121.7 (9)	Te(2)—C(8)—C(9)	117.1 (10)
Te(1)—C(1)—C(6)	117.2 (9)	Te(2)—C(8)—C(13)	120.2 (10)
C—C—C (minimum)	116 (1)	C—C—C (maximum)	125 (2)

capillary. Preliminary photographic examination was performed. Density was measured by flotation ($\text{CH}_2\text{I}_2\text{—C}_2\text{H}_4\text{Br}_2$). An Enraf-Nonius CAD-4 diffractometer equipped with graphite-monochromated Mo $K\alpha$ radiation was used to obtain unit-cell dimensions from 25 reflections ($8.7 < \theta < 15.0^\circ$) and subsequently record 3766 reflections ($\omega\text{—}2\theta$ scan; $1.5 < \theta < 25^\circ$; $h\text{—}28\text{—}28$, $k\text{—}0\text{—}11$, $l\text{—}0\text{—}28$). Three standard reflections showed no change with time and an empirical ψ -scan absorption correction based on three reflections was applied (transmission: minimum 59.6, maximum 99.9%). Lorentz and polarization corrections were applied. Of 3317 unique reflections ($R_{int} = 0.007$), 2653 with $F > 2\sigma(F)$ were used in the refinement. The normalized structure factors favoured the (non-standard) space group $I2/a$ (No. 15) rather than Ia , and this was confirmed by the subsequent analysis. Structure solution was by direct methods and refinement by *SHELX76* (Sheldrick, 1976) using full-matrix least squares on F . Some of the H atoms appeared on a difference electron-density synthesis ($R = 0.042$) and they were all introduced into the model in calculated positions ($\text{C—H} = 0.95 \text{\AA}$). Final $R = 0.041$ and $wR = 0.038$ for 173

parameters, anisotropic (Pt, Te, I, C) and isotropic (H) atoms, and $w = [\sigma^2(F) + 0.0001F^2]^{-1}$; $S = 1.82$. Maximum $|\Delta/\sigma|$ in the final cycle was 0.1; $\Delta\rho$ for the final model was between 1.76 and -1.11 e \AA^{-3} . Neutral-atom scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) (Pt, Te and I) and *SHELX76* (C, H). Fig. 1 was drawn using *ORTEPII* (Johnson, 1976) and all calculations were carried out on an IBM 3090 computer.

Discussion. Atomic coordinates for non-H atoms are listed in Table 1 and bond lengths and angles appear in Table 2.* The structure consists of discrete square-planar *trans* molecules (Fig. 1). The Pt—I distances can be compared with, for example, Pt—I in *trans*-[Pt(PMe₃)₂I₂] [2.599 (2) \AA (Hitchcock, Jacobson & Pidcock, 1977)], and the Pt—Te (*trans* Te) distances can be compared with the values found in the [Pt(*o*-C₆H₄(PPh₂)(TePh))₂]²⁺ cation [2.575 \AA (Gysling & Luss, 1984)] and in *trans*-[Pd(SCN)₂{Te[(CH₂)₃-SiMe₃]₂}]₂ [2.606 \AA (Gysling, Luss & Smith, 1979)]. In the present example, the methyl phenyl telluride ligands with the stereochemically active lone pair adopt the *RR* (and *SS*) configuration. Solution NMR studies (Kemmitt & Levason, 1990) of Pt(PhMeTe)₂X₂ showed that in CH₂Cl₂ solution for X = Cl, the *cis* isomer was more abundant (*ca* 10:1), while for the bromide the reverse was true (*ca* 1:3).

* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positions and complete geometric details have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55310 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0295]

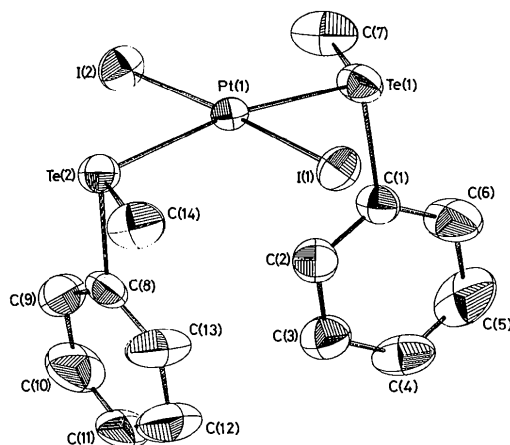


Fig. 1. View of one molecule of *trans*-[Pt(PhMeTe)₂I₂] showing the atom-numbering scheme. H atoms have been excluded and the thermal ellipsoid surfaces have been drawn at the 45% probability level.

The observation of only a single form of the iodide in solution is consistent with increasing preference for a *trans* geometry as the halogen co-ligands become heavier. Under high resolution the ^{125}Te and ^{195}Pt NMR resonances show small splittings which are attributable to *RR/SS* and *RS/SR* enantiomers, which will interconvert by pyramidal inversion at Te. As shown by the present X-ray study, the *trans* structure is also favoured in the solid state.

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Structure of Copper(II) Salicylates: Bis(3-pyridylmethanol)bis(salicylato)copper(II)

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Abstract. $[\text{Cu}(\text{C}_7\text{H}_5\text{O}_3)_2(\text{C}_6\text{H}_7\text{NO})_2]$, $M_r = 556.03$, orthorhombic, *Pbca*, $a = 12.947$ (1), $b = 9.401$ (3), $c = 20.005$ (3) Å, $V = 2435.16$ Å³, $Z = 4$, $D_m = 1.50$, $D_x = 1.52$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 16.949$ cm⁻¹, $F(000) = 1148$, $T = 293$ K, final $R = 0.044$ for 1122 observed reflections. The crystal structure consists of molecules bonded together into layers parallel to (100). The coordination polyhedron of Cu^{II} is a distorted octahedron built up by four short bonds in the equatorial plane [two Cu—N and two Cu—O bonds with bond lengths 1.944 (4) and 2.029 (4) Å] and two longer apical Cu—O bonds [2.622 (4) Å]. The O atoms forming apical bonds belong to the different molecules of 3-pyridylmethanol.

Introduction. Reports of complexes formed between copper(II) and salicylate ligands have given rise to several problems. For example, the ligand shows

monodentate (Pajunen & Pajunen, 1982; Hanic & Michalov, 1960) or bidentate (Ablov, Kiosse, Dimitrova, Malinovski & Popovich, 1974; Muhonen & Hämäläinen, 1978; Hoang, Valach, Dunaj-Jurčo & Melník, 1992) carboxylate coordination and also bidentate coordination through the carboxylic and phenolic O atoms (Jagner, Hazell & Larsen, 1976; Pajunen & Pajunen, 1982). In order to investigate further the structural properties of the salicylate ligand, we have examined the crystal structure of bis(3-pyridylmethanol)bis(salicylato)copper(II).

Experimental. Blue monocrystals of $\text{Cu}(\text{sal})_2 \cdot (3\text{-pyMe})_2$ (sal = salicylate, 3-pyMe = 3-pyridylmethanol) were prepared as described by Melník, Macášková & Mrozinski (1988). A prismatic crystal with dimensions $0.25 \times 0.20 \times 0.20$ mm (D_m by flotation in $\text{CHBr}_3/\text{CH}_3\text{OH}$) was used for data collection on a Syntex P_2 diffractometer, with