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Tetraphenylphosphonium Salts of 2-Telluro-5-methylthiophene and its Mercury(II) Metal Complex

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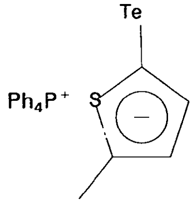
Abstract

Structures of the tetraphenylphosphonium salts of 2-telluro-5-methylthiophene and its mercury(II) metal complex, 2-tris(5-methyl-2-thienyltelluro)mercury(II) have been determined. Both compounds have ionic structures, with no close contacts between ions. The metal complex consists of three 2-telluro-5-methylthiophene ligands bound through the tellurium atoms in a trigonal-planar arrangement about the mercury(II) metal center. Addition of the methyl substituent to the thiophene ring avoids the rotational disorder which can occur upon complexation of the parent ion, 2-tellurothiophene.

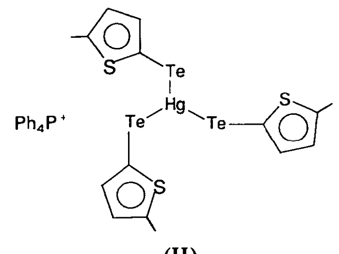
Comment

The complexation chemistry of organothiolate ligands has received considerable attention (Blower & Dilworth, 1987), in part because of their rich structural diversity (Lee, Craig, Ma, Scudder, Bailey & Dance, 1988; Pulla Rao, Dorfman & Holm, 1986; Christou, Hagen & Holm, 1982; Money, Huffman & Christou, 1988). The chemistry of organoselenium and organotellurium compounds, on the other hand, is much less developed (Gysling, 1986). The anion, 2-tellurothiophene, one of the few stable organotellurides (Engman & Cava, 1982), has been used to prepare one of only two known homoleptic metal organotelluride complexes, a tetrameric silver cluster, $Ag_4(TeC_4H_3S)_6^{2-}$ (Zhao, Adcock, Pennington & Kolis, 1990). We were interested in expanding the coordination chemistry of this ion; however, to avoid the formation of disordered complexes as a result of rotation of the thiophene ring about the C—Te bond, as was observed in the silver complex, we have modified the ligand by addition of a methyl substituent at the 5 position.

A solution (2.0 mmol) of the 2-telluro-5-methylthiophene ligand was prepared by dissolving methylthiophene (0.20 ml) in THF (10 ml) at 195 K, followed by addition of *n*-butyllithium (1.6 ml, 3.6 mmol). The solution was allowed to return to room temperature, at which time powdered elemental Te (2.0 mmol) was added. Tetraphenylphosphonium 2-telluro-5-methylthiophene (I) was formed by addition of a stoichiometric amount of tetraphenylphosphonium bromide to the above mixture; the resulting yellow solution was filtered, layered with diethyl ether and stored at 277 K overnight to generate yellow crystals in good yield. The metal complex, tris(2-telluro-5-methylthiophene)mercury(II) was formed by addition of $HgCl_2$ (0.5 mmol) in CH_3CN (10 ml) to a solution of (I) as described above, followed by filtration and layering with diethyl ether to form red crystals of the tetraphenylphosphonium salt (II).



(I)



(II)

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Compound (I) crystallizes as well separated ions, with no solvent molecules or interactions to stabilize the anion. The anion (Fig. 1*a*) is essentially planar [mean deviation of 0.03 (2) Å] with only minor differences in distances and angles as compared to those of the unsubstituted parent ion (Zhao, Adcock, Pennington & Kolis, 1990).

The structure of the metal complex of (II) is very similar to that observed in tris(tellurophenolato)-mercurate(II) which also has been structurally characterized as the tetraphenylphosphonium salt (Behrens, Hoffman & Klar, 1977). The three telluro-methyl-thiophene ligands are bound to the metal through the tellurium atoms in a trigonal-planar arrangement with the ligands in a propeller-like configuration (Fig. 1*b*). No disorder of the functionalized rings was observed. The dihedral angles between thiophene ring planes and the Te_3 plane are 22.0 [S(1)—C(4)], 53.3 [S(2)—C(9)] and 83.5° [S(3)—C(14)] *versus* values of 11.2, 34.6 and 71.2° for the phenyl derivative. The average Hg—Te distance of 2.70 (2) Å is identical for the two compounds as is the average Te—Hg—Te angle (120°), although both compounds exhibit some distortion from an idealized trigonal planar geometry about the metal center. Deviations in the present compound are somewhat larger, with Te—Hg—Te angles in the range 107.8 (1)–130.0 (1)° as opposed to a range of 110.7 (1)–125.2 (1)° for the phenyl derivative. This distortion appears to be the result of intramolecular steric interactions between a Te atom of one ligand and the thiophene ring of an adjacent ligand; in particular, repulsion between the S atom [S(1)] of the ring most nearly coplanar with the metal coordination sphere and a Te atom [Te(3)] seems to be a dominant interaction [S(1)···Te(3) = 3.899 (9) Å]. The observed conformation may be a result of packing effects, as suggested for the phenyl derivative, but other factors cannot be ruled out.

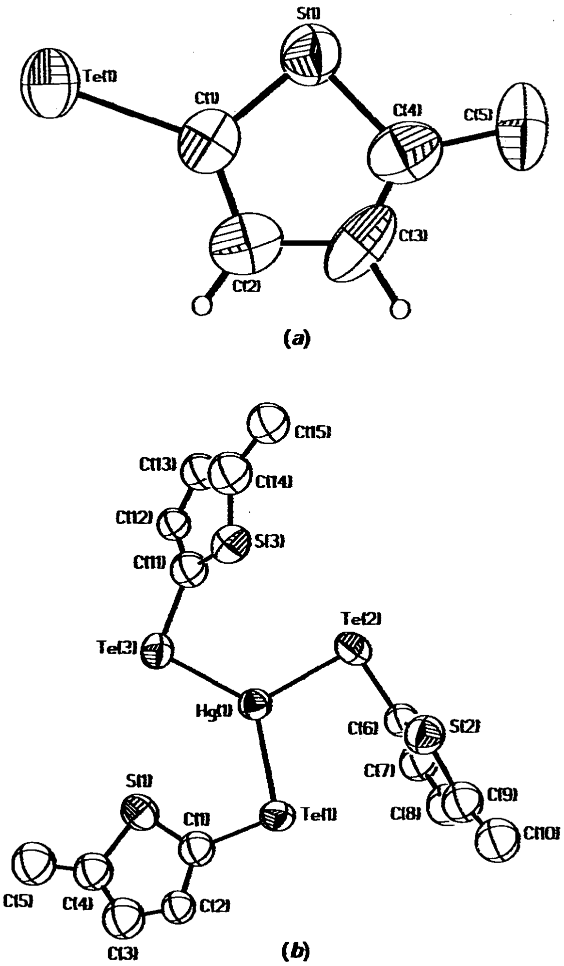

Fig. 1. Views of (*a*) (I) and (*b*) (II) showing the labeling of non-H atoms. Thermal ellipsoids are shown at 50% probability levels.

Experimental

Compound (I)

Crystal data

$C_{24}H_{20}P^+.C_5H_5STe^-$
$M_r = 564.17$
Monoclinic
$P2_1/c$
$a = 10.612$ (4) Å
$b = 13.902$ (7) Å
$c = 17.898$ (6) Å
$\beta = 104.62$ (3)°
$V = 2555$ (2) Å^3
$Z = 4$
$D_x = 1.47$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 35 reflections
$\theta = 16$–29°
$\mu = 1.32$ mm^{-1}
$T = 294$ K
Plate
0.35 × 0.29 × 0.11 mm
Pale orange

Data collection

Nicolet *R3m/V* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{min} = 0.54$, $T_{max} = 1.00$
3734 measured reflections
3344 independent reflections
1858 observed reflections [$I > 3\sigma(I)$]

$R_{int} = 0.028$
$\theta_{max} = 22.5°$
$h = -12 \rightarrow 0$
$k = -15 \rightarrow 0$
$l = -20 \rightarrow 20$
3 standard reflections monitored every 100 reflections
intensity variation: ±2%

Refinement

Refinement on F
Final $R = 0.0486$
$wR = 0.0552$
$S = 1.53$
1858 reflections
290 parameters
$w = 1/[\sigma^2(F)+0.000255F^2]$

$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.78$ e Å^{-3}
Atomic scattering factors from Cromer & Waber (1974)

Compound (II)

Crystal data

$[C_{24}H_{20}P][Hg(C_5H_5STe)_3]$
M_r = 1214.25
Orthorhombic
$P2_12_12_1$
a = 9.632 (3) Å
b = 17.067 (4) Å
c = 25.040 (5) Å
V = 4116 (2) Å^3
Z = 4
D_x = 1.96 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 37 reflections
θ = 22–30°
μ = 6.04 mm^{-1}
T = 294 K
Parallelepiped
0.30 × 0.25 × 0.19 mm
Red

Data collection

Nicolet *R3m/V* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min}$ = 0.62, $T_{\max}$ = 1.00
3054 measured reflections
3054 independent reflections
1858 observed reflections [$I > 3\sigma(I)$]

$\theta_{\max}$ = 22.5°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 27$
3 standard reflections monitored every 100 reflections
intensity variation: ±1%

Refinement

Refinement on F
Final R = 0.0530
wR = 0.0566
S = 1.27
1858 reflections
229 parameters
$w = 1/[\sigma^2(F)+0.0005F^2]$

$(\Delta/\sigma)_{\max}$ = 0.06
$\Delta\rho_{\max}$ = 1.56 e Å^{-3}
$\Delta\rho_{\min}$ = −0.81 e Å^{-3}
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Compound (I)				
Te(1)	0.7290 (1)	0.0831 (1)	0.6349 (1)	0.066 (1)
S(1)	0.9517 (3)	0.1697 (2)	0.7851 (2)	0.066 (1)
P(1)	0.6283 (3)	0.5099 (2)	0.6806 (1)	0.041 (1)
C(1)	0.7898 (12)	0.1606 (7)	0.7408 (6)	0.056 (5)
C(2)	0.7176 (14)	0.2102 (8)	0.7817 (7)	0.064 (5)
C(3)	0.8037 (18)	0.2575 (8)	0.8475 (7)	0.073 (7)
C(4)	0.9254 (16)	0.2429 (9)	0.8581 (7)	0.072 (7)
C(5)	1.0384 (15)	0.2853 (10)	0.9188 (7)	0.091 (7)
C(11)	0.4875 (10)	0.4349 (7)	0.6600 (5)	0.041 (4)
C(12)	0.4920 (12)	0.3432 (8)	0.6306 (5)	0.053 (5)
C(13)	0.3808 (15)	0.2852 (9)	0.6159 (6)	0.066 (6)
C(14)	0.2692 (14)	0.3175 (9)	0.6323 (6)	0.067 (6)
C(15)	0.2624 (12)	0.4087 (9)	0.6628 (6)	0.059 (5)
C(16)	0.3751 (12)	0.4661 (8)	0.6768 (5)	0.049 (4)
C(21)	0.5842 (10)	0.6287 (7)	0.6453 (5)	0.041 (4)
C(22)	0.4787 (11)	0.6431 (7)	0.5816 (5)	0.051 (4)
C(23)	0.4563 (13)	0.7362 (9)	0.5496 (6)	0.065 (5)
C(24)	0.5374 (15)	0.8100 (9)	0.5821 (7)	0.071 (6)
C(25)	0.6363 (15)	0.7967 (7)	0.6455 (7)	0.075 (6)
C(26)	0.6618 (12)	0.7061 (8)	0.6775 (6)	0.057 (5)
C(31)	0.7443 (11)	0.4650 (7)	0.6314 (6)	0.043 (3)
C(32)	0.8159 (12)	0.3822 (8)	0.6567 (7)	0.058 (5)
C(33)	0.8935 (13)	0.3426 (8)	0.6152 (7)	0.069 (6)
C(34)	0.9066 (13)	0.3888 (10)	0.5482 (7)	0.069 (6)
C(35)	0.8388 (12)	0.4722 (10)	0.5241 (6)	0.064 (5)
C(36)	0.7602 (11)	0.5109 (8)	0.5646 (5)	0.050 (4)
C(41)	0.7030 (11)	0.5145 (7)	0.7824 (5)	0.041 (4)
C(42)	0.6243 (12)	0.5083 (7)	0.8346 (5)	0.052 (4)
C(43)	0.6858 (14)	0.5177 (8)	0.9136 (6)	0.062 (5)
C(44)	0.8132 (13)	0.5317 (8)	0.9391 (6)	0.062 (5)
C(45)	0.8906 (11)	0.5377 (8)	0.8884 (6)	0.058 (5)
C(46)	0.8360 (12)	0.5283 (8)	0.8099 (6)	0.054 (5)
Compound (II)				
Hg(1)	0.7338 (1)	0.5727 (1)	0.8521 (1)	0.062 (1)
Te(1)	0.9325 (3)	0.5393 (1)	0.9250 (1)	0.069 (1)
Te(2)	0.7734 (3)	0.7223 (1)	0.8189 (1)	0.077 (1)
Te(3)	0.5250 (3)	0.4841 (1)	0.8157 (1)	0.079 (1)
S(1)	0.7727 (12)	0.3666 (5)	0.9092 (3)	0.078 (4)
S(2)	0.8447 (11)	0.7931 (5)	0.9384 (4)	0.080 (4)
S(3)	0.3748 (12)	0.6579 (6)	0.7957 (4)	0.093 (4)
P(1)	0.1120 (9)	0.0358 (5)	0.8721 (3)	0.053 (3)
C(1)	0.8711 (36)	0.4273 (19)	0.9477 (13)	0.073 (9)
C(2)	0.9041 (35)	0.3857 (17)	0.9962 (12)	0.063 (9)
C(3)	0.8567 (45)	0.3127 (22)	1.0001 (15)	0.099 (13)
C(4)	0.7798 (43)	0.2878 (20)	0.9564 (13)	0.083 (11)
C(5)	0.7037 (48)	0.2139 (24)	0.9449 (15)	0.112 (14)
C(6)	0.9071 (34)	0.7502 (18)	0.8823 (12)	0.063 (9)
C(7)	1.0580 (41)	0.7339 (21)	0.8836 (15)	0.086 (11)
C(8)	1.0976 (46)	0.7586 (23)	0.9360 (16)	0.101 (13)
C(9)	1.0099 (43)	0.7969 (21)	0.9716 (15)	0.088 (12)
C(10)	1.0125 (43)	0.8344 (22)	1.0285 (15)	0.098 (13)
C(11)	0.4319 (38)	0.5705 (21)	0.7695 (13)	0.078 (10)
C(12)	0.3780 (34)	0.5633 (18)	0.7181 (12)	0.065 (9)
C(13)	0.3132 (37)	0.6306 (20)	0.6959 (13)	0.082 (11)
C(14)	0.2941 (47)	0.6864 (23)	0.7365 (15)	0.104 (13)
C(15)	0.2268 (47)	0.7697 (20)	0.7325 (13)	0.092 (11)
C(16)	0.1830 (31)	0.1369 (17)	0.8653 (12)	0.056 (8)
C(17)	0.3127 (37)	0.1444 (20)	0.8452 (14)	0.077 (11)
C(18)	0.3582 (38)	0.2251 (20)	0.8374 (12)	0.075 (10)
C(19)	0.2747 (35)	0.2865 (17)	0.8527 (12)	0.066 (8)
C(20)	0.1425 (36)	0.2753 (18)	0.8791 (12)	0.070 (10)
C(21)	0.0893 (36)	0.1935 (18)	0.8844 (13)	0.068 (9)
C(22)	−0.0139 (30)	0.0323 (16)	0.9276 (11)	0.051 (8)
C(23)	−0.1399 (34)	−0.0017 (17)	0.9198 (12)	0.064 (9)
C(24)	−0.2362 (43)	−0.0019 (19)	0.9658 (12)	0.077 (9)
C(25)	−0.1871 (36)	0.0236 (19)	1.0130 (13)	0.076 (10)
C(26)	−0.0516 (38)	0.0584 (19)	1.0225 (13)	0.076 (10)
C(27)	0.0391 (38)	0.0635 (20)	0.9792 (13)	0.075 (10)
C(28)	0.2544 (32)	−0.0332 (14)	0.8808 (10)	0.048 (7)
C(29)	0.3114 (35)	−0.0580 (19)	0.8326 (12)	0.075 (11)
C(30)	0.4324 (39)	−0.1106 (19)	0.8380 (13)	0.078 (10)
C(31)	0.4684 (37)	−0.1412 (19)	0.8852 (14)	0.077 (10)
C(32)	0.4057 (39)	−0.1160 (20)	0.9330 (14)	0.082 (11)
C(33)	0.2958 (32)	−0.0613 (17)	0.9320 (12)	0.065 (9)
C(34)	0.0292 (28)	0.0142 (14)	0.8102 (10)	0.043 (7)
C(35)	0.0121 (30)	0.0692 (17)	0.7713 (11)	0.052 (8)
C(36)	−0.0606 (34)	0.0456 (17)	0.7234 (12)	0.064 (9)
C(37)	−0.0982 (38)	−0.0341 (20)	0.7146 (13)	0.078 (10)
C(38)	−0.0833 (37)	−0.0858 (19)	0.7565 (13)	0.074 (10)
C(39)	−0.0162 (33)	−0.0678 (18)	0.8035 (12)	0.069 (9)

Table 2. *Geometric parameters* (Å, °)

	(I)	(II)
Hg(1)—Te(1)		2.706 (3)
Hg(1)—Te(2)		2.712 (3)
Hg(1)—Te(3)		2.677 (3)
Te(1)—C(1)	2.13 (1)	2.08 (3)
Te(2)—C(5)		2.10 (3)
Te(3)—C(11)		2.08 (4)
S(1)—C(1)	1.71 (1)	1.70 (3)
S(1)—C(4)	1.73 (1)	1.79 (4)
S(2)—C(6)		1.70 (3)
S(2)—C(9)		1.80 (4)
S(3)—C(11)		1.72 (4)
S(3)—C(14)		1.74 (4)
C(1)—C(2)	1.37 (2)	1.44 (4)
C(2)—C(3)	1.45 (2)	1.33 (5)
C(3)—C(4)	1.27 (2)	1.39 (5)
C(4)—C(5)	1.52 (2)	1.49 (6)
C(6)—C(7)		1.48 (5)
C(7)—C(8)		1.43 (5)
C(8)—C(9)		1.39 (6)
C(9)—C(10)		1.56 (5)
C(11)—C(12)		1.39 (4)

C(12)—C(13)		1.42 (5)
C(13)—C(14)		1.40 (5)
C(14)—C(15)		1.57 (5)
P—C	1.79 (1)	1.83 (4)
C—C	1.38 (2)	1.42 (5)
Te(1)—Hg(1)—Te(2)		107.8 (1)
Te(1)—Hg(1)—Te(3)		130.0 (1)
Te(2)—Hg(1)—Te(3)		122.2 (1)
Hg(1)—Te(1)—C(1)		100.2 (9)
Hg(1)—Te(2)—C(6)		93.9 (9)
Hg(1)—Te(3)—C(11)		96 (1)
C(1)—S(1)—C(4)	93.4 (7)	94 (2)
C(6)—S(2)—C(9)		95 (2)
C(11)—S(3)—C(14)		93 (2)
Te(1)—C(1)—S(1)	119.6 (7)	124 (2)
Te(1)—C(1)—C(2)	130.0 (9)	128 (2)
S(1)—C(1)—C(2)	110.3 (8)	108 (2)
C(1)—C(2)—C(3)	110 (1)	116 (3)
C(2)—C(3)—C(4)	117 (1)	114 (3)
S(1)—C(4)—C(3)	109 (1)	108 (3)
S(1)—C(4)—C(5)		119 (3)
C(3)—C(4)—C(5)		132 (3)
Te(2)—C(6)—S(2)		120 (2)
Te(2)—C(6)—C(7)		125 (2)
S(2)—C(6)—C(7)		114 (2)
C(6)—C(7)—C(8)		103 (3)
C(7)—C(8)—C(9)	124 (4)	
S(2)—C(9)—C(8)	103 (3)	
S(2)—C(9)—C(10)		117 (3)
C(8)—C(9)—C(10)		140 (4)
Te(3)—C(11)—S(3)		123 (2)
Te(3)—C(11)—C(12)		128 (2)
S(3)—C(11)—C(12)		108 (2)
C(11)—C(12)—C(13)		117 (3)
C(12)—C(13)—C(14)		109 (3)
S(3)—C(14)—C(13)		112 (3)
S(3)—C(14)—C(15)		120 (3)
C(13)—C(14)—C(15)		128 (3)
C—P—C	109 (1)	109 (2)
P—C—C	120.2 (9)	117 (4)
C—C—C	120 (1)	120 (4)

Structure solution: direct methods. Structure refinement: full-matrix least-squares on F. As (II) crystallizes in a chiral space group, refinement was carried out for both enantiomeric conformations; the reported coordinates correspond to the model which gave lower residuals (residuals for the incorrect model are: $R = 0.0768$, $wR = 0.0840$, $S = 1.89$). The refinement of (I) included positional and anisotropic thermal parameters for all non-H atoms; H atoms other than those of the methyl C atom (which were not located) were included in idealized positions with a group isotropic thermal parameter [$U_{\rm iso} = 0.072$ (8) Å^2]. The refinement of (II) included positional parameters and anisotropic thermal parameters for the non-H atoms other than C, which were refined isotropically; H atoms were not included. Computer programs: *SHELXTL* (Sheldrick, 1985). Corrections: Lorentz and polarization, real and imaginary anomalous dispersion (Cromer, 1974).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71113 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1024]

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Structures of Bis(di-2-pyridylamine)dinitratocopper(II) and Bis(acetato)bis(di-2-pyridylamine)copper(II) Monohydrate

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Abstract

Both complexes consist of neutral mononuclear units, the copper(II) environment being distorted 4+2 octahedral for (I) and distorted square pyramidal for (II). Compound (I) is centrosymmetric. Its equatorial plane comprises the four N atoms of two di-2-pyridylamine (dpyam) ligands with the axial positions filled by two O atoms of two unidentate nitrate anions. The equatorial plane in (II) is composed of the two N atoms of dpyam and two O atoms of two acetate groups, with the apical position occupied by another O atom of one of the acetate