

Bis(triphenyltellurium) Di- μ -chloro-bis[dichloromercurate(II)], $[\text{Te}(\text{C}_6\text{H}_5)_3]^+[\text{Hg}_2\text{Cl}_6]^-$

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Abstract. $M_r = 1331.74$, triclinic, $P\bar{1}$, $a = 9.090$ (6), $b = 10.224$ (5), $c = 10.820$ (5) Å, $\alpha = 95.54$ (3), $\beta = 92.47$ (3), $\gamma = 99.48$ (3)°, $V = 985.5$ (9) Å³, $Z = 1$, D_m (floatation, benzene/bromofrom mixture) = 2.249, $D_x = 2.244$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 72.1$ cm⁻¹, $F(000) = 612$, $T = 295$ K, final $R = 0.046$ for 3334 reflections. The structure contains trigonal pyramidal Ph_3Te^+ cations and chloro-bridged dimeric Hg_2Cl_6^- anions which consist of two distorted HgCl_4 tetrahedra sharing an edge; interionic $\text{Te}\cdots\text{Cl}$ interactions complete distorted octahedral geometry at Te. $\text{Hg}-\text{Cl} = 2.684$ (2), 2.717 (2) (bridging, b), 2.350 (2) (terminal, t) Å, $\text{Cl}(b)-\text{Hg}-\text{Cl}(b) = 89.6$ (1), $\text{Cl}(b)-\text{Hg}-\text{Cl}(t) = 101.2$ (1), 106.6 (1), $\text{Cl}(t)-\text{Hg}-\text{Cl}(t) = 141.3$ (1), $\text{Hg}-\text{Cl}(b)-\text{Hg} = 90.4$ (1)°; $\text{Te}-\text{C} = 2.11$ (1) Å, $\text{C}-\text{Te}-\text{C} = 94.4$ (2), 95.2 (2), 101.3 (2)°, $\text{Te}\cdots\text{Cl} = 3.337$ (2), 3.489 (2), 3.574 (2) Å.

Introduction. Complexes formed from organotellurium compounds and mercury halides have been known for many years (Irgolic, 1974), and recently several complexes have been obtained from reactions of triphenyltellurium and mercury halides (Khandelwal, Jain & Berry, 1982). The present study shows that a member of this class of complex is bis(triphenyltellurium) di- μ -chloro-bis[dichloromercurate(II)].

Experimental. Colorless crystals (from ethanol), $0.2 \times 0.4 \times 0.24$ mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta \leq 30^\circ$, $\omega-2\theta$ scan, ω -scan width $(0.60 + 0.35\tan\theta)^\circ$ extended by 25% on each side of the peak for background measurement, horizontal aperture $(2.0 + \tan\theta)$ mm, vertical aperture 4 mm, intensity and orientation controls regularly throughout data collection; decay of three standard intensities necessitated application of an anisotropic decay correction; data corrected for L_p and absorption (Coppens, Leiserowitz & Rabinovich, 1965; Busing & Levy, 1967), transmission factors ranged from 0.1457 to 0.3636; cell parameters by least-squares fit to observed $\sin\theta$ values for 24 centered reflections with $15 < \theta < 22^\circ$; 5552 independent reflections, 3334 (60.1%) with $I \geq 3\sigma(I)$, where $\sigma^2(I) = S + 2B + [0.04(S - B)]^2$, $S = \text{scan count}$ and $B = \text{time-averaged background}$. $h = 12-12$, $k = 14-13$, $l = 0-15$. Structure solved by Patterson and Fourier

methods, full-matrix least-squares refinement with anisotropic thermal parameters, H atoms fixed and included for structure-factor calculations but not refined, function minimized $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F)$; $\Delta\rho$ excursions in final difference map $2.0 \text{ e } \text{Å}^{-3}$; $R = 0.046$, $R_w = 0.056$ for 3334 reflections, 208 variables, $R = 0.084$, $R_w = 0.056$ for all data; final refinement included anomalous scattering; mean and max. Δ on the last cycle 0.005σ and 0.027σ , respectively, standard deviation in an observation of unit weight 2.220; atomic scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965), anomalous-scattering factors from Cromer & Liberman (1970). Local adaptations of standard computer programs ORFLS (Busing, Martin & Levy, 1962), ORFFE (Busing, Martin & Levy, 1964), FORDAP (A. Zalkin, unpublished) and PLUTO (Motherwell & Clegg, 1978). Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1.*

Discussion. The structure (Figs. 1 and 2) contains trigonal pyramidal Ph_3Te^+ cations and centrosymmetric chloro-bridged dimeric Hg_2Cl_6^- anions which consist of two distorted HgCl_4 tetrahedra sharing an edge. Pairs of cations interact with each anion through sets of three $\text{Te}\cdots\text{Cl}$ contacts which complete distorted octahedral coordination at Te.

The Ph_3Te^+ geometry is quite similar to that found in other triphenyltellurium and selenium compounds (Hope, 1966; Einstein, Trotter & Williston, 1967; Lee, Titus & Ziolo, 1976). The three $\text{Te}-\text{C}$ distances do not differ significantly (Table 2), with the mean value of 2.11 (1) Å close to the covalent radius sum of 2.142 Å (Pauling, 1960). The $\text{C}-\text{Te}-\text{C}$ angles are 94.4 (2), 95.2 (2) and 101.3 (2)°, mean 97.0° ; the deviation from threefold symmetry probably results from the asymmetric surroundings of the cation. The angles are consistent with those found for Ph_3TeX ($X = \text{Cl}, \text{Br}$)

* Lists of structure factors, anisotropic thermal parameters, coordinates, bond distances, and angles involving hydrogen atoms, and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39466 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final positional ($\times 10^4$) and isotropic thermal ($\times 10^3, \text{\AA}^2$) parameters with e.s.d.'s in parentheses

	x	y	z	U_{eq}/U_{iso}^*
Hg	1746.5 (5)	-760.7 (3)	-348.1 (3)	83
Te	2474.4 (4)	2588.1 (4)	2370.2 (4)	46
Cl(1)	1412.8 (23)	-1867.2 (20)	-2372.9 (18)	68
Cl(2)	861.4 (24)	1557.8 (19)	-712.5 (19)	68
Cl(3)	3465.1 (27)	-320.2 (22)	1388.0 (21)	78
C(1)	4559 (7)	3533 (7)	1839 (6)	47
C(2)	5321 (8)	4702 (8)	2494 (7)	63
C(3)	6621 (9)	5336 (9)	2071 (8)	70
C(4)	7122 (9)	4868 (9)	955 (9)	68
C(5)	6370 (9)	3715 (8)	276 (8)	69
C(6)	5050 (8)	3028 (7)	726 (7)	56
C(7)	3084 (7)	1941 (6)	4070 (6)	47
C(8)	1922 (8)	1382 (9)	4743 (8)	67
C(9)	2237 (9)	994 (9)	5896 (8)	74
C(10)	3669 (10)	1134 (9)	6350 (8)	77
C(11)	4816 (10)	1640 (11)	5672 (10)	87
C(12)	4518 (8)	2053 (10)	4539 (9)	75
C(13)	1854 (7)	4377 (6)	3170 (6)	47
C(14)	1241 (9)	5146 (9)	2370 (9)	70
C(15)	914 (10)	6367 (10)	2873 (13)	87
C(16)	1196 (10)	6801 (9)	4094 (14)	90
C(17)	1770 (9)	6015 (9)	4880 (9)	74
C(18)	2139 (8)	4832 (8)	4408 (7)	60

$$* U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

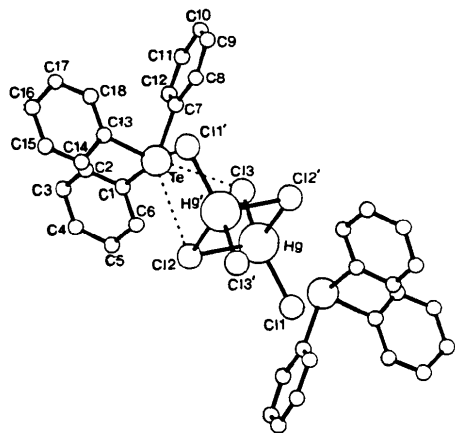
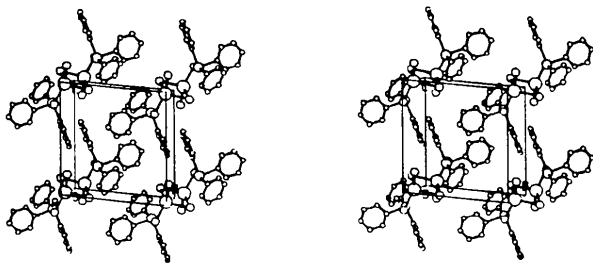


Fig. 1. Perspective drawing of the structure showing the atom numbering scheme.

Fig. 2. Stereoview of the unit cell along the a axis.Table 2. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

Hg—Cl(1)	2.350 (2)	Hg...Hg'	3.831 (1)
Hg—Cl(2)	2.684 (2)	Cl(2)...Cl(2')	3.807 (4)
Hg—Cl(2')	2.717 (2)		
Hg—Cl(3)	2.350 (2)	Te...Cl(1')	3.489 (2)
C(1)—C(2)	1.389 (10)	Te...Cl(2)	3.574 (2)
C(1)—C(6)	1.385 (10)	Te...Cl(3)	3.337 (2)
C(2)—C(3)	1.371 (11)	Te—C(1)	2.110 (7)
C(3)—C(4)	1.379 (12)	Te—C(7)	2.097 (6)
C(4)—C(5)	1.386 (12)	Te—C(13)	2.119 (6)
C(5)—C(6)	1.417 (11)		
C(7)—C(8)	1.386 (11)	C(13)—C(14)	1.382 (10)
C(7)—C(12)	1.360 (10)	C(13)—C(18)	1.374 (10)
C(8)—C(9)	1.378 (12)	C(14)—C(15)	1.396 (14)
C(9)—C(10)	1.352 (13)	C(15)—C(16)	1.352 (17)
C(10)—C(11)	1.362 (13)	C(16)—C(17)	1.368 (16)
C(11)—C(12)	1.366 (13)	C(17)—C(18)	1.366 (11)
Cl(1)—Hg—Cl(2)	101.23 (7)	Hg—Cl(2)—Hg'	90.37 (6)
Cl(1)—Hg—Cl(3)	141.34 (8)	Cl(2)—Hg—Hg'	45.16 (5)
Cl(2)—Hg—Cl(3)	106.59 (7)	Cl(2')—Hg—Hg'	44.47 (4)
Cl(2)—Hg—Cl(2')	89.63 (6)		
Te—C(1)—C(2)	121.6 (5)	Cl(1')...Te...Cl(2)	69.28 (5)
Te—C(1)—C(6)	116.9 (5)	Cl(1')...Te...Cl(3)	104.18 (5)
C(2)—C(1)—C(6)	121.1 (7)	Cl(2)...Te...Cl(3)	71.47 (5)
C(1)—C(2)—C(3)	119.9 (7)	C(1)—Te—C(7)	101.3 (2)
C(2)—C(3)—C(4)	120.2 (7)	C(1)—Te—C(13)	94.4 (2)
C(3)—C(4)—C(5)	120.9 (7)	C(7)—Te—C(13)	95.2 (2)
C(4)—C(5)—C(6)	119.2 (7)		
C(1)—C(6)—C(5)	118.6 (7)	C(7)—C(12)—C(11)	120.5 (8)
Te—C(7)—C(8)	116.2 (5)	Te—C(13)—C(14)	117.0 (5)
Te—C(7)—C(12)	124.3 (6)	Te—C(13)—C(18)	122.8 (5)
C(8)—C(7)—C(12)	119.5 (7)	C(14)—C(13)—C(18)	120.0 (7)
C(7)—C(8)—C(9)	119.3 (7)	C(13)—C(14)—C(15)	117.6 (9)
C(8)—C(9)—C(10)	120.1 (7)	C(14)—C(15)—C(16)	121.8 (9)
C(9)—C(10)—C(11)	120.7 (8)	C(15)—C(16)—C(17)	119.9 (8)
C(10)—C(11)—C(12)	119.8 (8)	C(16)—C(17)—C(18)	119.5 (9)
		C(13)—C(18)—C(17)	121.1 (8)

(Lee, Titus & Ziolo, 1976; Titus, Lee & Ziolo, 1976), and are smaller than those in corresponding selenium species (Hope, 1966; Lee & Titus, 1976). The phenyl rings are planar [maximum deviation 0.020 (8) \AA] and have a propeller arrangement, with dihedral angles between the ring planes of 95.1 (3), 100.0 (3), and 115.7 (3) $^\circ$. The C—C bond distances, 1.35 (1)–1.42 (1) \AA , and angles at C, 116 (1)–124 (1) $^\circ$, are normal, with the outer bonds probably affected by thermal libration errors.

Three interionic Te...Cl interactions complete distorted octahedral coordination at Te. The Te...Cl distances of 3.337 (2), 3.489 (2) and 3.574 (2) \AA (Table 2) are considerably shorter than the van der Waals distance of 4.0 \AA (Pauling, 1960; Bondi, 1964) and indicate significant bonding interaction; the Cl...Te...Cl angles differ considerably from 90 $^\circ$ (Table 2).

The centrosymmetric $\text{Hg}_2\text{Cl}_6^{2-}$ anion contains two bent Cl(1)—Hg—Cl(3) units bridged by two Cl(2) atoms, with distorted tetrahedral coordination for Hg and a four-membered Hg_2Cl_2 ring. The bridging Hg—Cl(2) bonds are longer than the terminal Hg—Cl(1,3) and slightly asymmetric. One other feature is a significant asymmetry in the Hg thermal vibration; $U_{11} = 0.1325 (4) \text{\AA}^2$ corresponds to a r.m.s. displacement of 0.36 \AA . Deviations from $P\bar{1}$ symmetry seem

unlikely, since no Friedel pair differences were observed, and the parameter probably corresponds to a real vibration in a direction approximately bisecting the large Cl(1)–Hg–Cl(3) angle of 141°. The overall geometry and dimensions of the anion are similar to those found previously for $\text{Hg}_2\text{X}_6^{2-}$ species (Beurskens, Bosman & Cras, 1972; Brotherton, Epstein, White & Willis, 1974; von Brodersen, Pezzeri & Thiele, 1983).

Distances between the $[\text{Ph}_3\text{Te}]_2[\text{Hg}_2\text{Cl}_6]$ units (Figs. 1 and 2) correspond to normal van der Waals interactions.

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Structure of $[N,N'$ -Bis(β -carbamoylethyl)-2-hydroxytrimethylenediamine]copper(II) Perchlorate, $[\text{Cu}(\text{C}_9\text{H}_{20}\text{N}_4\text{O}_3)](\text{ClO}_4)_2^\dagger$

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Abstract. $M_r = 494.73$, monoclinic, $P2_1/c$, $Z = 4$, $a = 13.997$ (7), $b = 10.656$ (9), $c = 14.254$ (12) Å, $V = 1783.2$ (3) Å³, $D_x = 1.85$, $D_m = 1.81$ (3) Mg m⁻³ (by flotation in CCl_4 -hexane), Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 2.98$ mm⁻¹, $F(000) = 1012$, $T = 296$ (4) K, $R(F) = 0.076$ for 2195 observed reflections. The coordination geometry around the Cu atom is a tetragonally distorted octahedron comprising two O atoms from the two amide groups and two N atoms from the two amine groups in an equatorial plane, and the two O atoms of the perchlorate ions above and below this plane at

distances of 2.448 (11) and 2.641 (11) Å. This complex has the *RS* configuration for the chiral amine N centres. The two perchlorate ions are situated on opposite sides of the equatorial plane; they form intra- and intermolecular hydrogen bonds with the multidentate ligand and neighbouring molecules.

Introduction. In a previous paper we have reported the crystal structure of a copper(II) complex with *N,N'*-bis(β -carbamoylethyl)ethylenediamine, $[\text{Cu}(\text{NO}_3)\text{-(bcen)}(\text{H}_2\text{O})]\text{NO}_3 \cdot \text{H}_2\text{O}$ (Lee, Lu, Liu, Chung & Lee, 1984). As an extension, the crystal and molecular structure of the title compound, $[\text{Cu}(\text{bchtn})](\text{ClO}_4)_2$ (Chen, 1982), are described here.

* (6-Hydroxy-4,8-diazaundecanediamide)copper(II) perchlorate.
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