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Acta Cryst. (1993). **C49**, 267–270

Structure of Benzo[*a*]phenothiazine Pentachlorotellurate(IV)

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(Received 3 February 1992; accepted 18 May 1992)

Abstract. Bis(benzo[*a*]phenothiaziniumyl) di- μ -chlorobis[tetrachlorotellurate(IV)], (C₁₆H₁₁NS)₂.Te₂Cl₁₀, $M_r = 1108.4$, monoclinic, $P2_1/a$, $a = 10.975$ (2), $b = 19.937$ (3), $c = 8.742$ (4) Å, $\beta = 90.40$ (2)°, $V = 1912.9$ (9) Å³, $Z = 2$, $D_m = 1.93$, $D_x = 1.925$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 2.37$ mm⁻¹, $F(000) = 1068$, $T = 293$ K, $R = 0.052$ for 3724 observed reflections. Benzo[*a*]phenothiazine exists as a nearly planar cation radical. Each Te atom is coordinated by six Cl atoms in a distorted octahedral configuration. Both cation radicals and anions construct separate dimers.

Introduction. The structure determination of the title compound is part of a series of studies on phenothiazines, in which we have been interested in both the electronic and the structural natures of the ionic radical salts of phenothiazine (PT) and/or its derivatives (Uchida, Ito & Kozawa, 1983; Uchida, Seki, Ito, Nakano, Hoshizaki & Kozawa, 1986; Kozawa & Uchida, 1990; Kozawa, Hoshizaki & Uchida, 1991). Although it has long been observed that PT has the

unique feature of possessing multiple stable oxidation states in solution (Billon, 1962; Shine & Mach, 1965), we found that the cation radical state of PT and its derivatives is stable even in the solid state, and that their molecular conformations were flattened compared with the folded conformation of their neutral states. Furthermore, we found that the aggregate manner of PT derivatives in the crystals was usually the same as that of counter metal chloride anions (Kozawa, Hoshizaki & Uchida, 1991), that is, they construct dimeric pairs with dimer anions, tetrameric stacks with tetramer anions and infinite stacks with infinite polyanions. This paper is concerned with the dimeric structure of (B[*a*]PT)₂-Te₂Cl₁₀. Hereafter, B[*a*]PT denotes benzo[*a*]phenothiazine.

Experimental. B[*a*]PT was synthesized from *N*-phenyl-1-naphthylamine and sulfur according to the literature method (Knoevenagel, 1914) and purified by sublimation *in vacuo*. Single crystals of the complex were obtained from a 1,2-dichloroethane solu-

tion of B[a]PT and extra grade $TeCl_4$ in a 1:2 molar ratio. A black needle-shaped single crystal of dimensions $0.45 \times 0.2 \times 0.1$ mm was used for data collection. D_m was measured by flotation in a bromoform/chloroform solution. Rigaku AFC-5 four-circle diffractometer, $\theta/2\theta$ data collection, scan width $(1 + 0.5 \tan \theta)^\circ$ in θ , scan rate 4° min^{-1} in θ , $2\theta_{\text{max}} = 60^\circ$, range of h, k, l -15 to 15, 0 to 27, 0 to 12; standard reflections ($7\bar{2}0, 720, \bar{4}, \bar{1}0, \bar{1}$) monitored every 100 measurements; intensity variations in F 3%; 6080 reflections measured, 5585 unique, $R_{\text{int}} = 0.02$, 3724 observed with $F_o > 3\sigma(F_o)$; lattice parameters determined from 20 ($25.9 < 2\theta < 31.0^\circ$) reflections; correction for Lorentz and polarization effects but not for absorption.

The structure was solved by the heavy-atom method. Te atoms were located from Patterson map, all non-H atoms were found in repeated Fourier syntheses. The structure was refined by block-diagonal least-squares refinement. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma(F_o)^2$. H atoms except for H(13) and H(N) were revealed on a difference Fourier map and the remaining two H atoms were calculated. Parameters refined: atomic coordinates for all atoms, anisotropic temperature factors for non-H atoms, isotropic temperature factor for H atoms was fixed at $B_{\text{iso}} = 4.0 \text{ \AA}^2$. $R = 0.052$, $wR = 0.032$, $S = 2.89$ for 3724 reflections, $(\Delta/\sigma)_{\text{max}} = 0.40$ for non-H atoms, $\Delta\rho_{\text{min}} = -0.8$, $\Delta\rho_{\text{max}} = 0.9 \text{ e \AA}^{-3}$ around Te in the final difference map. Scattering factors and anomalous-dispersion factors for Te, Cl and S from *International Tables for X-ray Crystallography* (1974, Vol. IV). The calculations were carried out on a Panafacom U-1200II computer with the Rigaku *RASA-5P* program package and on a HITAC *M680H* at the Computer Centre of the University of Tokyo with the *UNICS* program system (Sakurai, 1967).

Discussion. Both B[a]PT and $TeCl_5^-$ form dimers in the crystal and each dimer is centrosymmetric around the crystallographic inversion center. Fig. 1 shows the molecular structure of B[a]PT and dimeric $Te_2Cl_{10}^{2-}$ with the atomic numbering scheme, and the final atomic parameters are listed in Table 1.* The bond lengths and angles of B[a]PT are listed in Table 2. It is estimated that this molecule, existing as a cation radical in this complex crystal, has unit positive charges. This is inferred not only from its molar ratio to the anion but also from its characteristic

cation radical absorption spectrum in 1,2-dichloroethane solution (Kozawa, Hoshizaki & Uchida, 1991). The molecular structure (Table 3) also reveals cation radical characteristics. Namely, values of the averaged S—C and N—C bond lengths of this molecule (1.701 and 1.340 Å, respectively) are similar to those of the phenothiazine cation radical ($PT^{+\cdot}$) (1.71–1.72 and 1.36–1.38 Å, respectively), being shorter than those of neutral B[a]PT and neutral PT (1.762–1.770 and 1.399–1.406 Å, respectively). The C—S—C and C—N—C bond angles and the dihedral angle between aromatic rings on both sides of the thiazine structure (105.4, 125.3 and 175.8°, respectively) are larger than those of neutral B[a]PT and neutral PT (99.6–101.4, 121.5–124.4 and 153.3–158.6°, respectively), and they are close to those of $PT^{+\cdot}$ (104–105.4, 123–128 and 175.8–178.4°, respectively).

The crystal structure of the title compound is shown in Fig. 2. Dimeric pairs of B[a]PT and $Te_2Cl_{10}^{2-}$ stack almost alternately in the crystal lattice. B[a]PT molecules overlap within the dimeric pair of *A* and *B*, but they scarcely overlap between the dimers (*B* and *C*) as shown in Fig. 2. The interplanar distance between the least-squares molecular planes in the dimer is 3.46 Å. This is similar in magnitude to the thickness of neutral aromatic molecules and also to the interplanar distance in infinite $PT^{+\cdot}$ stacks (3.47 Å) in monoclinic $PT-SbCl_4$ (Kozawa & Uchida, 1990). The shortest intermolecular distance in the dimer is 3.484 (6) Å for $S \cdots C(3')$, which is almost equal to the sum of the van der Waals radii of S and C. The bond lengths and angles of dimeric

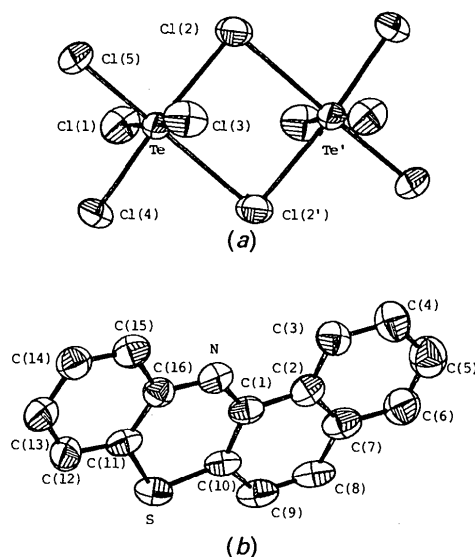


Fig. 1. ORTEP drawing (Johnson, 1976) of the molecular structure of (a) dimeric $Te_2Cl_{10}^{2-}$ and (b) B[a]PT, with the atomic numbering scheme.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55467 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1004]

Table 1. Fractional coordinates and equivalent isotropic thermal parameters for non-H atoms with *e.s.d.*'s in parentheses
$$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Te	0.01938 (3)	0.09661 (2)	0.42976 (5)	3.32 (1)
Cl(1)	0.0248 (2)	0.13637 (9)	0.7042 (2)	5.67 (6)
Cl(2)	-0.1761 (1)	0.01710 (9)	0.4859 (2)	5.40 (6)
Cl(3)	0.0012 (2)	0.0653 (1)	0.1572 (2)	5.96 (6)
Cl(4)	0.2028 (1)	0.15815 (9)	0.3725 (2)	5.72 (6)
Cl(5)	-0.1050 (1)	0.18990 (8)	0.3788 (2)	5.34 (5)
S	0.7514 (1)	0.0537 (1)	0.8962 (2)	5.17 (6)
N	0.5181 (4)	-0.0066 (3)	0.7594 (5)	4.2 (1)
C(1)	0.5779 (5)	-0.0441 (3)	0.8608 (6)	3.8 (2)
C(2)	0.5300 (5)	-0.1101 (3)	0.8971 (6)	3.9 (2)
C(3)	0.4237 (5)	-0.1353 (3)	0.8298 (7)	4.5 (2)
C(4)	0.3842 (6)	-0.1982 (3)	0.8648 (8)	5.8 (2)
C(5)	0.4490 (6)	-0.2391 (3)	0.9685 (9)	6.3 (3)
C(6)	0.5517 (6)	0.2140 (3)	1.0390 (8)	5.5 (2)
C(7)	0.5938 (5)	-0.1497 (3)	1.0068 (7)	4.6 (2)
C(8)	0.6986 (5)	-0.1220 (3)	1.0805 (7)	5.0 (2)
C(9)	0.7428 (5)	-0.0622 (3)	1.0500 (7)	4.9 (2)
C(10)	0.6868 (5)	-0.0209 (3)	0.9387 (7)	3.9 (2)
C(11)	0.6565 (5)	0.0900 (3)	0.7639 (6)	4.0 (2)
C(12)	0.6851 (5)	0.1527 (3)	0.7069 (8)	4.9 (2)
C(13)	0.6106 (6)	0.1828 (3)	0.6039 (8)	5.4 (2)
C(14)	0.5037 (6)	0.1493 (3)	0.5543 (8)	5.2 (2)
C(15)	0.4742 (5)	0.0880 (3)	0.6051 (7)	4.6 (2)
C(16)	0.5516 (5)	0.0554 (3)	0.7137 (7)	4.1 (2)

Table 2. Bond lengths (\AA) and angles ($^\circ$) of $[\text{Te}_2\text{Cl}_{10}]^{2-}$ and benzo[a]phenothiazine

Te—Cl(1)	2.527 (2)	Te—Cl(4)	2.413 (2)
Te—Cl(2)	2.715 (2)	Te—Cl(5)	2.349 (2)
Te—Cl(3)	2.470 (2)	Te—Cl(2')	2.937 (2)
S—C(10)	1.690 (6)	C(6)—C(7)	1.392 (9)
S—C(11)	1.712 (6)	C(7)—C(8)	1.426 (9)
N—C(1)	1.329 (8)	C(8)—C(9)	1.315 (9)
N—C(16)	1.351 (8)	C(9)—C(10)	1.412 (9)
C(1)—C(2)	1.453 (8)	C(11)—C(12)	1.383 (9)
C(1)—C(10)	1.447 (8)	C(11)—C(16)	1.410 (8)
C(2)—C(3)	1.396 (8)	C(12)—C(13)	1.352 (10)
C(2)—C(7)	1.423 (8)	C(13)—C(14)	1.415 (10)
C(3)—C(4)	1.362 (9)	C(14)—C(15)	1.341 (9)
C(4)—C(5)	1.408 (10)	C(15)—C(16)	1.426 (9)
C(5)—C(6)	1.375 (10)		
Cl(1)—Te—Cl(2)	91.43 (6)	Cl(2)—Te—Cl(2')	88.05 (6)
Cl(1)—Te—Cl(3)	175.13 (6)	Cl(3)—Te—Cl(4)	89.44 (7)
Cl(1)—Te—Cl(4)	91.34 (7)	Cl(3)—Te—Cl(5)	88.52 (7)
Cl(1)—Te—Cl(5)	86.64 (6)	Cl(3)—Te—Cl(2')	95.18 (6)
Cl(1)—Te—Cl(2')	89.65 (6)	Cl(4)—Te—Cl(5)	92.46 (7)
Cl(2)—Te—Cl(3)	88.18 (6)	Cl(4)—Te—Cl(2')	87.51 (6)
Cl(2)—Te—Cl(4)	174.76 (7)	Cl(5)—Te—Cl(2')	176.29 (6)
Cl(2)—Te—Cl(5)	92.14 (6)	Te—Cl(2)—Te'	91.95 (6)
C(10)—S—C(11)	105.4 (3)	C(7)—C(8)—C(9)	123.8 (6)
C(1)—N—C(16)	125.3 (5)	C(8)—C(9)—C(10)	120.6 (6)
N—C(1)—C(2)	118.5 (5)	S—C(10)—C(1)	121.6 (4)
N—C(1)—C(10)	122.4 (5)	S—C(10)—C(9)	118.9 (5)
C(2)—C(1)—C(10)	119.0 (5)	C(1)—C(10)—C(9)	119.5 (5)
C(1)—C(2)—C(3)	122.4 (5)	S—C(11)—C(12)	119.2 (5)
C(1)—C(2)—C(7)	118.2 (5)	S—C(11)—C(16)	119.7 (5)
C(3)—C(2)—C(7)	119.3 (5)	C(12)—C(11)—C(16)	121.1 (6)
C(2)—C(3)—C(4)	120.2 (6)	C(11)—C(12)—C(13)	120.2 (6)
C(3)—C(4)—C(5)	121.2 (6)	C(12)—C(13)—C(14)	119.4 (6)
C(4)—C(5)—C(6)	119.1 (7)	C(13)—C(14)—C(15)	122.0 (6)
C(5)—C(6)—C(7)	121.1 (6)	C(14)—C(15)—C(16)	119.5 (6)
C(2)—C(7)—C(6)	119.0 (6)	N—C(16)—C(15)	125.4 (5)
C(2)—C(7)—C(8)	118.8 (6)	N—C(16)—C(11)	116.9 (5)
C(6)—C(7)—C(8)	122.2 (6)	C(11)—C(16)—C(15)	117.7 (5)

Symmetry code: (i) $-x, -y, 1-z$.Table 3. Comparison of some averaged bond lengths (\AA) and angles ($^\circ$) of B[a]PT and PT

	S—C	N—C	C—S—C	C—N—C	Dihedral angle	Ref.
B[a]PT	1.769	1.404	101.4	122.0	156.0	(a)
B[a]PT—TeCl ₅	1.701	1.340	105.4	125.3	175.8	
PT (orthorhombic)	1.762	1.399	100.9	124.4	158.5	(b)
PT (monoclinic)	1.770	1.406	99.6	121.5	153.3	(c)
PT*—SbCl ₆	1.71	1.38	104	127	175.8	(d)
PT*—SbCl ₆ (triclinic)	1.72	1.36	104.6	128	178.4	(e)
PT*—SbCl ₆ (monoclinic)	1.72	1.37	105.4	123	176.2	(e)

References: (a) Yoshida, Matsuzawa, Kozawa & Uchida (1993); (b) McDowell (1976); (c) Bell, Blount, Briscoe & Freeman (1968); (d) Uchida, Ito & Kozawa (1983); (e) Kozawa & Uchida (1990).

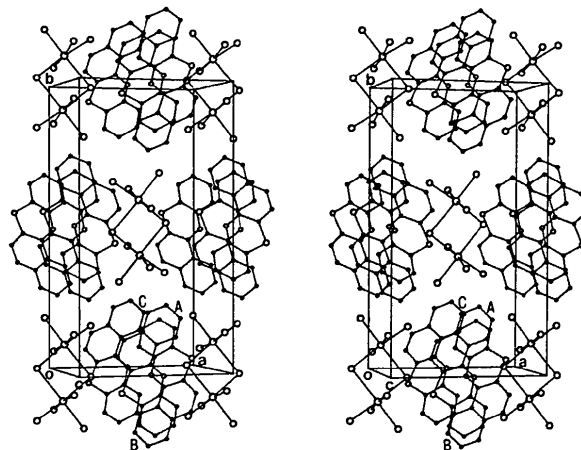


Fig. 2. Packing diagram of molecules in the unit cell with labelling of B[a]PT molecules.

$\text{Te}_2\text{Cl}_{10}^{2-}$ are listed in Table 2. This dimer is composed of two TeCl_5^- ions sharing the edge of a hexacoordinated octahedron (Fig. 1) and has four terminal Te—Cl bonds [2.349 (2)—2.527 (2) \AA ; average 2.440 \AA] and two bridging Te—Cl—Te' bonds [2.715 (2) and 2.937 (2) \AA]. A structure similar to this dimeric anion, $\text{Te}_2\text{Cl}_{10}^{2-}$, has been reported in $[(\text{C}_6\text{H}_5)_4\text{P}]_2\text{Te}_2\text{Br}_{10}^{2-}$ (Krebs & Büscher, 1980) in which there are two bridging Br atoms having significantly different Te—Br lengths.

The dimeric structure of $\text{Te}_2\text{Cl}_{10}^{2-}$ can be described in another way. Although neutral TeCl_4 in the gas phase has a monomeric trigonal bipyramidal structure (Stevenson & Schomaker, 1940), it forms a cubane-like tetramer ($\text{Te}_4\text{Cl}_{16}$) in the solid state (Buss & Krebs, 1971). Each Te atom, coordinated by six Cl atoms, is located at an apex of a cube in the tetramer. Successive removal of TeCl_3^+ from the tetramer leads firstly to the $\text{Te}_3\text{Cl}_{13}^-$ ion (Krebs & Paulat, 1979) and then to the $\text{Te}_2\text{Cl}_{10}^{2-}$ ion of the present crystal.

The terminal Te—Cl bond lengths become gradually longer as one moves from the neutral tetramer, $\text{Te}_4\text{Cl}_{16}$ (average 2.311 \AA), to the trimeric monovalent anion, $\text{Te}_3\text{Cl}_{13}^-$ (average 2.342 \AA), to the

present divalent anion, the negative charge also increasing.

The authors wish to thank Mr K. Kato and Mr Y. Hosomi for their technical assistance.

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Acta Cryst. (1993). **C49**, 270–273

Structure of 3-Methoxy-6 α ,17 β -dihydroxyestra-1,3,5(10)-trien-7-one Oxime

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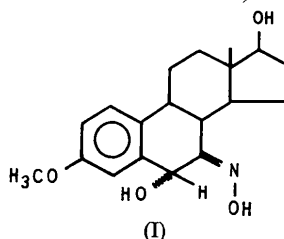
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(Received 18 November 1991; accepted 6 March 1992)

Abstract. C₁₉H₂₅NO₄, $M_r = 331.41$, monoclinic, $P2_1$, $a = 14.950$ (2), $b = 9.248$ (1), $c = 12.597$ (1) Å, $\beta = 100.98$ (1)°, $V = 1709.7$ (5) Å³, $Z = 4$, $D_x = 1.283$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.53$ cm⁻¹, $F(000) = 708$, $T = 293$ (2) K, $R = 0.046$ for 2400 observed reflections. The asymmetric unit contains two molecules, which differ in the conformation of the *B* rings and in the orientations of the C(3) methoxy groups and C(7)=N—OH moieties. The molecules are held together by a three-dimensional network of hydrogen bonds.

Introduction. So far, preparation of *B*-seco estrone derivatives has been almost exclusively achieved by total syntheses (Jhingran, Gupta, Ray, Agarwal, Singh & Anand, 1983, 1986). In our recent research, directed towards a partial synthesis of some novel *B*-seco estrone derivatives (as potential anti-estrogens), the title compound (I) was prepared as a crucial intermediate. The compound was further subjected to the Beckmann fragmentation reaction, in

accordance with our previous work (Miljković & Petrović, 1977), in order to bring about the formation of the corresponding *B*-seco derivative. However, as numerous attempts were unsuccessful, it became obvious that it was necessary to determine the exact geometry of the 7-oximino function (*syn* or *anti*) to overcome synthetic difficulties.



Experimental. A crystal of dimensions $0.50 \times 0.36 \times 0.22$ mm was mounted on a CAD-4 diffractometer equipped with a graphite monochromator. Cell constants were refined by a least-squares fit for 20 centred reflections in the range $16.3 < \theta < 19.4^\circ$. Intensities of 2663 reflections were measured of

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