Acta Cryst. (1989). C45, 2002–2003

Structure of Bis(tetraphenylarsonium) Hexabromotellurate(IV)

By W. Abriel* and A. du Bois

Institut für Anorganische Chemie und SFB 173 *der Universität, Callinstr.* 9, *D*-3000 *Hannover* 1,
Federal Republic of Germany

(*Received* 29 *March* 1989; *accepted* 26 *July* 1989)

Abstract. $[As(C_6H_5)_4]_2[TeBr_6]$, $M_r = 1373·72$, triclinic, $P\bar{1}$, $a = 10·430\,(2)$, $b = 10·673\,(2)$, $c = 12·346\,(3)$ Å, $\alpha = 92·26\,(2)$, $\beta = 100·30\,(2)$, $\gamma = 116·24\,(1)°$, $V = 1201·82$ Å^3, $Z = 1$, $D_x = 1·898$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0·7107$ Å, $\mu = 68·18$ cm^{-1}, $F(000) = 656$, $T = 293$ K, $R = 0·0707$ for 2511 reflexions ($F > 3\sigma_F$). The $TeBr_6^{2-}$ anion shows a rather regular octahedral structure with a mean Te—Br distance of 2·699 (1) Å. This dynamically distorted anion in a centrosymmetric crystal field confirms the symmetry rules for the stereochemistry of the lone-pair electrons in AX_6E systems.

Experimental. Crystals of the title compound were prepared by dissolving $TeBr_4$ and $[As(C_6H_5)_4]Cl.H_2O$ in concentrated hydrobromic acid in a molar ratio of 2:1. According to Abriel & Ehrhardt (1988) the $TeBr_6^{2-}$ salt precipitates exclusively due to the instability of the Te—Cl bond in the presence of Br^-. Extraction with CH_2Cl_2 and subsequent slow evaporation of the solvent yielded pseudo-hexagonal-prismatic single crystals suitable for X-ray single-crystal work. The crystal used for the data collection was placed in a capillary in order to protect it from moisture. A summary of data collection and structure refinement is given in Table 1.

The initial positions of Te, Br and As were obtained from isostructural $[As(C_6H_5)_4]_2[TeCl_6]$ (Milne, Philippot & Maurin, 1984). Subsequent ΔF synthesis yielded the C-atom positions. H atoms were refined with geometrical constraints due to an sp^2-hybridization of the adjacent C atom. Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). All computations were performed with the program system *STRUCSY* from the diffractometer software package (Siemens, 1983). Atomic parameters are

Table 1. *Summary of data collection and structure refinement for* $[As(C_6H_5)_4]_2[TeBr_6]$

Crystal shape	Pseudo-hexagonal-prismatic, faces {001}, {010}, {100}, $(1\bar{1}0)$, $(\bar{1}10)$
Crystal volume (mm^3)	$8·25 \times 10^{-4}$
Diffractometer	Siemens AED2, graphite-monochromated Mo $K\alpha$ radiation
Determination of cell parameters	
number of reflexions used	31
θ range (°)	8·3–13·0
Intensity-data collection	
max. $\sin\theta/\lambda$(Å^{-1})	0·5951
range of h, k, l	$\overline{12},\overline{12},\overline{14}$ to 12,12,8
scan mode	ω
min. scan angle (°)	1·6
max. scan time (s)	36
standard reflexions	$32\bar{3}$, $\bar{3}41$, $\bar{1}\bar{1}5$
intensity variation	< 2%
measured reflexions	7760
unique reflexions	4177
observed reflexions	2511 with $F > 3\sigma_F$
R_{int}	0·079
Numeric absorption correction	
min./max. transmission	0·43/0·78
Structure refinement (on F)	
R, wR $[w = 1/\sigma^2(F_o)]$	0·0707, 0·0398
S	1·93
number of parameters	259
max. final shift/e.s.d.	< 0·001
min./max. final $\Delta\rho$ (e Å^{-3})	$-0·22 \le \Delta \le 0·2$

Table 2. *Positional and equivalent isotropic displacement parameters for atoms in* $[As(C_6H_5)_4]_2[TeBr_6]$

$U_{eq} = 1/3\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{A}_{ij}$, where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.

	Site	x	y	z	U_{eq}(Å^2)
Te	1(h)	0·5000	0·5000	0·5000	0·0296
Br(1)	2(i)	0·4060 (1)	0·6810 (1)	0·5653 (1)	0·0444
Br(2)	2(i)	0·7797 (1)	0·6968 (1)	0·5856 (1)	0·0527
Br(3)	2(i)	0·4830 (1)	0·4082 (1)	0·7008 (1)	0·0486
As	2(i)	0·6821 (1)	0·1108 (1)	0·8212 (1)	0·0314
C(1)	2(i)	0·3098 (9)	0·7529 (8)	1·0688 (7)	0·0274
C(2)	2(i)	−0·5660 (10)	−0·2569 (10)	0·0599 (8)	0·0322
C(3)	2(i)	0·4272 (12)	0·6477 (11)	0·9756 (10)	0·0497
C(4)	2(i)	0·2970 (14)	0·5608 (10)	0·9060 (10)	0·0531
C(5)	2(i)	0·1715 (12)	0·5689 (12)	0·9170 (9)	0·0542
C(6)	2(i)	0·1787 (11)	0·6625 (11)	0·9996 (9)	0·0434
C(7)	2(i)	−0·8252 (9)	−0·2146 (10)	0·2609 (8)	0·0325
C(8)	2(i)	−0·8281 (10)	−0·3322 (10)	0·3007 (8)	0·0367
C(9)	2(i)	−0·9337 (11)	−0·4109 (11)	0·3558 (9)	0·0495
C(10)	2(i)	−1·0403 (11)	−0·3705 (13)	0·3688 (9)	0·0508
C(11)	2(i)	−1·0354 (11)	−0·2521 (12)	0·3338 (9)	0·0504
C(12)	2(i)	−0·9259 (10)	−0·1706 (10)	0·2796 (9)	0·0407
C(13)	2(i)	0·5130 (10)	0·9862 (10)	0·2659 (9)	0·0384
C(14)	2(i)	0·5463 (11)	0·9385 (11)	0·3657 (10)	0·0466
C(15)	2(i)	0·6924 (13)	0·9984 (12)	0·4221 (10)	0·0527
C(16)	2(i)	0·7988 (12)	1·1057 (12)	0·3831 (11)	0·0498
C(17)	2(i)	0·7632 (10)	1·1512 (11)	0·2851 (11)	0·0488
C(18)	2(i)	0·6208 (11)	1·0913 (10)	0·2270 (9)	0·0395
C(19)	2(i)	0·2656 (9)	1·0187 (10)	1·1058 (8)	0·0327
C(20)	2(i)	0·2742 (10)	1·1357 (11)	1·1672 (9)	0·0405
C(21)	2(i)	0·2300 (11)	1·2261 (12)	1·1171 (11)	0·0481
C(22)	2(i)	0·1757 (11)	1·2019 (12)	1·0042 (12)	0·0551
C(23)	2(i)	0·1711 (11)	1·0879 (13)	0·9417 (10)	0·0497
C(24)	2(i)	0·2159 (10)	0·9966 (11)	0·9921 (8)	0·0406

---

* Author to whom correspondence should be addressed. Present address: Institut für Anorganische Chemie der Universität, PO Box 397, D-8400 Regensburg, Federal Republic of Germany.

0108-2701/89/122002-02$03.00

Table 3. *Bond lengths* (Å) *and bond angles* (°)

(1) Anion			
Te—Br(1)	2·679 (1)	Br(1)—Te—Br(2)	89·32 (2)
Te—Br(2)	2·710 (1)	Br(1)—Te—Br(2)	90·68 (2)
Te—Br(3)	2·708 (1)	Br(1)—Te—Br(3)	92·44 (3)
		Br(1)—Te—Br(3)	87·56 (3)
Br(1)—Br(2)	3·788 (1)	Br(2)—Te—Br(3)	90·38 (3)
Br(1)—Br(2)	3·833 (1)	Br(2)—Te—Br(3)	89·62 (3)
Br(1)—Br(3)	3·727 (1)		
Br(1)—Br(3)	3·889 (1)		
Br(2)—Br(3)	3·818 (1)		
Br(2)—Br(3)	3·844 (2)		
(2) Cation			
As—C(1)	1·914 (7)	C(1)—As—C(7)	105·7 (3)
As—C(7)	1·910 (7)	C(1)—As—C(13)	108·6 (3)
As—C(13)	1·907 (7)	C(1)—As—C(19)	108·7 (4)
As—C(19)	1·900 (8)	C(7)—As—C(13)	114·2 (4)
		C(7)—As—C(19)	109·7 (2)
		C(13)—As—C(19)	109·9 (3)
C(1)—C(2)	1·368 (6)	C(7)—C(8)	1·356 (12)
C(2)—C(3)	1·397 (13)	C(8)—C(9)	1·370 (11)
C(3)—C(4)	1·360 (13)	C(9)—C(10)	1·389 (7)
C(4)—C(5)	1·379 (9)	C(10)—C(11)	1·334 (14)
C(5)—C(6)	1·367 (14)	C(11)—C(12)	1·396 (11)
C(6)—C(1)	1·372 (11)	C(12)—C(7)	1·375 (6)
C(13)—C(14)	1·397 (15)	C(19)—C(20)	1·394 (12)
C(14)—C(15)	1·393 (11)	C(20)—C(21)	1·365 (12)
C(15)—C(16)	1·373 (13)	C(21)—C(22)	1·375 (19)
C(16)—C(17)	1·372 (17)	C(22)—C(23)	1·393 (15)
C(17)—C(18)	1·368 (11)	C(23)—C(24)	1·379 (12)
C(18)—C(13)	1·369 (11)	C(24)—C(19)	1·379 (14)

given in Table 2,* bond lengths and some important bond angles in Table 3. Atom labeling is given in Fig. 1. The maximal deviation of C atoms from the common ring plane is 0·02 [C(1)–C(6)], 0·022 [C(7)–C(12)], 0·016 [C(13)–C(18)] and 0·015 Å [C(19)–C(24)].

Related literature. Symmetry rules for the stereochemistry of the lone-pair electrons in AX_6E systems are discussed by Abriel (1986), Abriel & Zehnder (1987) and Abriel (1987). The last paper gives basic structural data for all compounds containing AX_6^{2-} ions (A = Se, Te; X = Cl, Br, I) known to date.

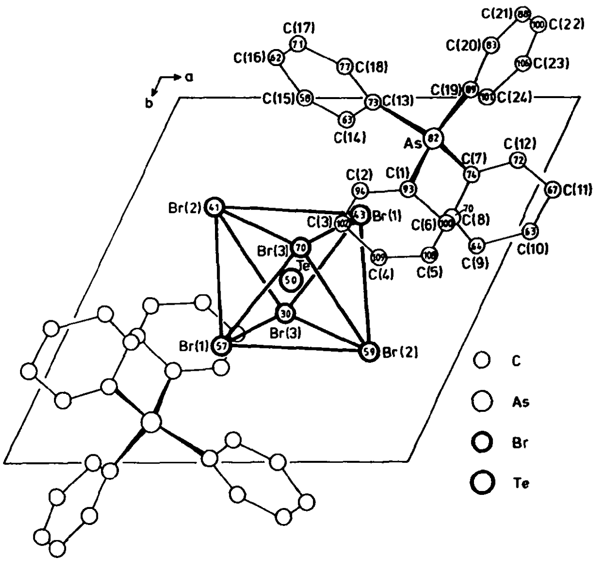

Fig. 1. Projection of the crystal structure of $[As(C_6H_5)_4]_2[TeBr_6]$ on (001). Heights of atoms from the *ab* plane are indicated as percentages of the *c* length. Two complete cations are shown.

Extended symmetry rules considering SbX_6^{3-} and BiX_6^{3-} species are given by du Bois & Abriel (1988).

Financial support by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie is gratefully acknowledged.

References

Abriel, W. (1986). *Acta Cryst.* **B42**, 449–453.
Abriel, W. (1987). *Z. Naturforsch. Teil B*, **42**, 415–420.
Abriel, W. & Ehrhardt, H. (1988). *Z. Naturforsch. Teil B*, **43**, 557–560.
Abriel, W. & Zehnder, E.-J. (1987). *Z. Naturforsch. Teil B*, **42**, 1273–1281.
Bois, A. du & Abriel, W. (1988). *Z. Naturforsch. Teil B*, **43**, 1003–1009.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Milne, J. B., Philippot, E. & Maurin, M. (1984). *Rev. Chim. Minér.* **21**, 749–757.
Siemens (1983). *STRUCSY*. Diffractometer software description for AED2. Siemens, Karlsruhe, Federal Republic of Germany.

---

* Lists of anisotropic displacement parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52145 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.