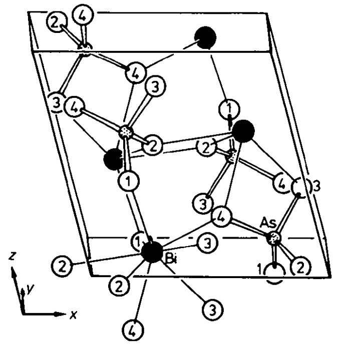
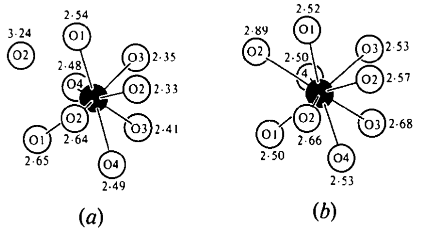


Fig. 1 A view of the structure of α-BiAsO$_4$.



(*a*) (*b*)

Fig. 2. Comparison of the cation coordination in the monazite type compounds (*a*) α-BiAsO$_4$ and (*b*) LaVO$_4$ (Rice & Robinson, 1976). Numbers with two decimal places are cation–oxygen distances (e.s.d.'s < 0·01 Å).

atoms with Bi—O = 2·49 and 2·59 Å was reported for the scheelite type β-BiAsO$_4$ at room temperature (Mooney, 1948). In this compound the inert electron pair of Bi^{3+} may become stereochemically active at low temperatures and cause distortions of the BiO polyhedron accompanied by a phase transformation, as was observed for scheelite type BiVO$_4$ (Sleight *et al.*, 1979).

The AsO$_4$ group of the title compound exhibits usual dimensions with respect to bond lengths but is, with respect to O—As—O angles, strongly distorted. The distance O(3)—O(4), corresponding to an O—As—O angle of 97·3°, is quite short, 2·537 Å; it represents the only edge shared between the AsO$_4$ tetrahedron and the BiO polyhedron. The distortion of the VO$_4$ tetrahedron in monoclinic LaVO$_4$ is similar but smaller.

This work was carried out with instrumentation supplied by the Fonds zur Förderung der wissenschaftlichen Forschung (project 2178). DB gratefully appreciates the support of the Consejo Nacional de Investigaciones Científicas y Técnicas de Argentina and of the Universidad de Buenos Aires.

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Tris[pentafluorotellurato(VI)]boron(III)

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(*Received* 21 *October* 1981; *accepted* 11 *January* 1982)

Abstract. B(OTeF$_5$)$_3$, hexagonal, space group $P6_3/m$, $a = 9{\cdot}218$ (2), $c = 9{\cdot}207$ (2) Å, $U = 677{\cdot}6$ (2) Å^3, $D_c = 3{\cdot}56$ Mg m^{-3} for $Z = 2$; $R_1 = 0{\cdot}031$ for 566 observed reflections. The B(OTeF$_5$)$_3$ molecule has D_{3h} symmetry with average B—O, Te—O and Te—F bond lengths of 1·358 (6), 1·874 (6) and 1·816 (5) Å, respectively. The

0567-7408/82/051561-03$01.00

environments of the B^{III} and Te^{VI} atoms are strictly trigonal planar and approximately octahedral, respectively; the B—O—Te angle is 132·3 (4)°.

Introduction. The pentafluorotellurate(VI) ligand has recently been used to prepare many elements in their highest oxidation states (Lentz & Seppelt, 1978*a*,*b*, 1979; Schroeder & Sladky, 1980) since it has the advantage of both steric size and a high group electronegativity that has been estimated to be 3·87 compared to 4·00 for F (Pauling scale) (Birchall, Meyers, de Waard & Schrobilgen, 1982). A necessary precursor in many of these reactions is the tris[pentafluorotellurato(VI)]boron(III) compound, $B(OTeF_5)_3$. To gain additional structural information on the $-OTeF_5$ ligand and its effect on the B—O distance, we have determined the crystal structure of the compound $B(OTeF_5)_3$.

Clear, colourless, moisture-sensitive crystals of this compound are hexagonal prisms and rhombs and were prepared as previously described (Sladky, Kropshofer & Leitzke, 1973). The crystals have a high vapour pressure at room temperature and in mounting the crystals in Lindemann capillaries in a dry-box, some surface loss was experienced. However, this had the advantage of rounding off the crystals' edges such that the sealed crystals approximated to spheres or cylinders. One approximately spherical crystal of radius 0·11 mm was selected and accurate unit-cell dimensions were obtained by least-squares fit of 2θ, ω and χ for 15 high-angle reflections measured on a Syntex $P2_1$ diffractometer using Mo $K\bar{\alpha}$ radiation (λ = 0·71069 Å).* Data were collected using θ–2θ scans over a 2θ scan range (K_{α_1} − 0·85°) to (K_{α_2} + 0·85°) and variable scan rates of 3–29·3° min^{-1} which were dependent on the intensity of a prescan. Due to sublimation/decomposition of the crystal, there was a gradual decrease in the intensities of the three standard reflections which were monitored after every 37 reflections collected. The crystal was also recentred several times during the data collection. All reflections in the quadrants (h,$\pm k$,l) with $2\theta \leq 55°$ were collected. Lorentz, polarization and spherical absorption corrections (μ = 6·85 mm^{-1} and hence μR = 0·75) were applied to all the data (1668 reflections) after the measured intensities had been rescaled for crystal deterioration. Subsequent averaging resulted in a data set of 641 independent reflections. The agreement index for this averaging procedure was somewhat high (0·05) due to the large rescale factors required for some of the data (up to 25%).

Table 1. *Final atomic positional and thermal parameters with e.s.d.'s in parentheses*

	x	y	z	U/U_{eq}* (Å^2)
Te	0·3555 (1)	0·3569 (1)	0·2500	0·0336 (3)
O	0·4218 (8)	0·5850 (7)	0·2500	0·0387 (33)
F(1)	0·2986 (9)	0·1371 (7)	0·2500	0·0638 (42)
F(2)	0·2006 (5)	0·3096 (5)	0·3902 (5)	0·0569 (26)
F(3)	0·5124 (5)	0·3968 (5)	0·3878 (5)	0·0550 (26)
B	0·3333	0·6667	0·2500	0·0419 (41)

* $U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Te(1)–F(1)	1·822 (6) [1·847]	Te–O	1·874 (6) [1·883]
Te(1)–F(2)	1·809 (4) [1·831] (×2)	B–O	1·358 (6)
Te(1)–F(3)	1·818 (4) [1·834] (×2)	B···Te	2·963 (1)
B–O–Te	132·3 (4)		
F(1)–Te–F(2)	88·8 (2) (×2)	F(2)–Te–F(3)	90·2 (2) (×2)
F(1)–Te–F(3)	89·0 (2) (×2)	F(2)–Te–F(2)'	91·2 (2)
F(1)–Te–O	178·0 (3)	F(2)–Te–O	92·6 (2)

Primed atoms are related to the corresponding unprimed atoms by reflection through the mirror plane at $y = \frac{1}{4}$. Values in square brackets are interatomic distances corrected for thermal motion assuming a riding model.

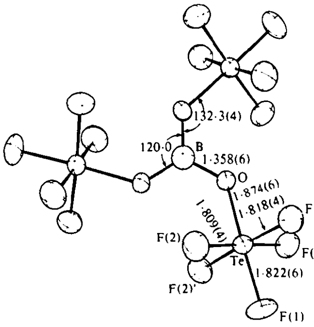

Fig. 1. Labelling of atoms in $B(OTeF_5)_3$ with some distances (Å) and angles (°).

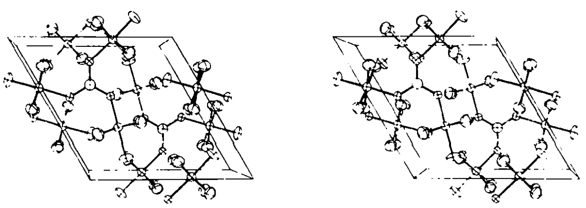

Fig. 2. Stereoscopic view of the $B(OTeF_5)_3$ unit cell down **c**.

The structure was solved using conventional Patterson and Fourier methods. Least-squares refinement minimizing $\sum w||F_o| - |F_c||^2$ with all atoms except B having anisotropic thermal parameters has converged (no shift/error > 20%) to final agreement indices R_1 = 0·0311 for 566 observed reflections with $F > 6·0\,\sigma(F)$ and R_1 = 0·0365 for 624 data with $F > 2·0\,\sigma(F)$. Unit

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* Recently Kropshofer, Heitzke, Peringer & Sladky (1981) reported the cell parameters of $B(OTeF_5)_3$ as a = 9·20 (1) Å, c = 9·19 (1) Å, space group $P6_3/m$.

weights were used throughout and a final difference Fourier map was featureless with a maximum peak of 1·0 e Å^{-3} and a minimum trough of −1·7 e Å^{-3}. Computing was carried out using the XRAY system (1972) and *SHELX* (Sheldrick, 1976) on a CDC 6400 computer. The final atomic coordinates and temperature factors are included in Table 1 and bond lengths and angles are given in Table 2. Views of the molecule and the crystal packing are given in Figs. 1 and 2 respectively.*

Discussion. The crystal structure contains isolated $B(OTeF_5)_3$ molecules, with the central B atom of each molecule on the rotary inversion axes and the Te, O and F atoms *trans* to O in the mirror planes. The molecule has, therefore, $\bar{6}m2$ or D_{3h} symmetry and the environment of the B^{III} atom is strictly trigonal planar. Bond angles around Te are close to 90 and 180° so that the Te^{VI} atom is approximately octahedrally coordinated by the O and five F atoms (Fig. 1).

The B—O distance in $B(OTeF_5)_3$ is 1·358 (6) Å, which is slightly shorter than most of the B—O distances in the trigonal planar units observed in borates where distances range from 1·352–1·412 Å.

However, the values of the B—O bond lengths in the compounds $B(OMe)_3$ and $O(BMe_2)_2$ have been determined by electron diffraction as 1·367 (4) and 1·359 (4) Å respectively (Gundersen, 1976; Gundersen & Vahrenkamp, 1976) and are virtually the same length as the present distance. This might be considered somewhat surprising in view of the expected electronegativity of the $OTeF_5$ ligand. However, the B—O distances in $B(OTeF_5)_3$ as well as $B(OMe)_3$ and $O(BMe_2)_2$ are all significantly shorter than the Schomaker–Stevenson modified B—O single-bond length of 1·43 Å, indicating that some significant multiple bonding exists for the B—O bonds in all three compounds. A distance of 1·25 (2) Å has been quoted for a linear O—B—O grouping (Calvo & Faggiani, 1974).

Bond lengths in the $OTeF_5$ ligand of $B(OTeF_5)_3$ are comparable to those observed in other compounds containing this ligand. Thus, the observed Te—O distance of 1·874 (6) Å is very similar in length to the analogous bond lengths in $F_2Te(OTeF_5)_4$ [1·879 (5) Å] (Lentz, Pritzkow & Seppelt, 1978) and $Te(OTeF_5)_6$ [1·890–1·898 (3) Å] (Pritzkow & Seppelt, 1977). Similarly, the B—O—Te angle in $B(OTeF_5)_3$ of 132·3 (4)° is a few degrees smaller than the Te—O—Te angles of 139·4 (3) and 139·0 (2)° in $F_2Te(OTeF_5)_4$ and $Te(OTeF_5)_6$ respectively. Previous arguments have based this large angle at O on $Te(M)\cdots Te$ repulsion [the $Te\cdots Te$ contact distances are 3·518 and 3·555 Å in $F_2Te(OTeF_5)_4$ and $Te(OTeF_5)_6$ respectively]. In the present compound the $B\cdots Te$ distance is 2·963 (1) Å.

We thank the Natural Sciences and Engineering Research Council of Canada for support of this work.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36657 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.