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Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

D—H···*A*	H···*A*	*D*···*A*	*D*–H···*A*
O(4)–H(13)···O(2^{i})	1·57 (8)	2·53 (2)	161 (8)
N^{+}—H(1)···O(1^{ii})	1·76 (12)	2·69 (2)	171 (8)

Notation: (i) $1-x,\bar{y},\bar{z}$; (ii) $x,1+y,1+z$.

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Ditellurium(IV) Trioxosulphate

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Abstract. $Te_2O_3SO_4$, $P2_1nm$, orthorhombic, $a=4{\cdot}654$ (1), $b=6{\cdot}936$ (1), $c=8{\cdot}879$ (2) Å, $Z=2$, $V=286{\cdot}6$ (1) Å^3, $M=399{\cdot}26$, $D_x=4{\cdot}63$ g cm^{-3}. The S–O distances are 1·47 (1)–1·49 (1) Å. The Te^{IV} coordination is threefold pyramidal with Te–O distances of 1·89 (1), 1·91 (1) and 2·00 (1) Å. In addition there are three longer Te–O bonds of 2·26 (1), 2·63 (1) and 2·84 (1) Å. The TeO_3 and SO_4 units are connected to form sheets.

Introduction. The preparation of $Te_2O_3SO_4$ has been described by Hubková, Loub & Syneček (1966), who found that $Te_2O_3SO_4$ belonged to space group $P2_1nm$. Since there were, however, uncertainties about the proposed structure, a complete redetermination has been performed. Single crystals were prepared and provided by Dr Moret, Montpellier. The cell dimensions were refined (Lindqvist & Wengelin, 1967) from 48* lines measured on a Guinier powder photograph, taken with KCl as an internal standard ($a_{KCl}=6{\cdot}2919$ Å at 20°C; Hambling, 1953).

Intensities were collected on a two-circle Pailred diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. The ω-scan procedure was used with a scan speed of 2·5° min^{-1} and the background was measured for 24 s at each end of the scan interval ($\Delta\omega=3$–6°). The layers $0kl$–$8kl$ were registered out to $2\theta=100°$ and 1050 unique reflexions for which $\sigma(I)/I<0{\cdot}3$ were used in the structure analysis. The intensities were corrected for absorption (program *DATAP*2; Coppens, Leiserowitz & Rabinovich, 1965). A drawing of the crystal used for the data collection is shown in Fig. 1. μ(Mo $K\alpha$) = 177·6 cm^{-1} (*International Tables for X-ray Crystallography*, 1962).

The structure was solved from Patterson and electron density calculations (program *DRF*; A. Zalkin, Berkeley) and the space group confirmed to be $P2_1nm$ in accordance with the systematic absences $h+l=2n+1$ for the $h0l$ reflexions. The structural parameters were refined with the block-diagonal approximation (program *BLOCK*; O. Lindgren, Göteborg), initially with isotropic temperature factors and separate scale factors for each layer ($R=0{\cdot}052$) and subsequently with anisotropic temperature factors and an overall scale factor ($R=0{\cdot}047$). The positional parameters obtained from

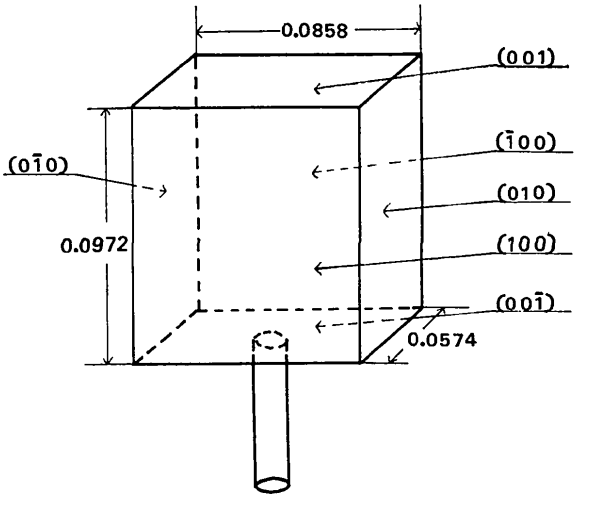

Fig. 1. The crystal used for data collection. The distances are given in mm.

* Lists of observed and calculated $\sin^2\theta_{hkl}$ and d_{hkl} values, and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31788 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

the two refinements were the same within the standard deviations. The final parameters are given in Table 1. Scattering factors for Te (Cromer & Waber, 1965), S and O (Doyle & Turner, 1968) were corrected for the real part of the anomalous scattering (Cromer, 1965). Weights $w=20{\cdot}0+F_o+0{\cdot}004F_o^2$ were used. No extinction correction was performed, since only a few of the strongest reflexions appeared to be affected.

Discussion. Crystals of tellurates(IV) are usually prepared by hydrothermal syntheses or from melts. Action of conc. HNO_3 or H_2SO_4 on Te metal can also be used, *e.g.* TeO_2 (Lindqvist, 1968), $Te_2O_4.HNO_3$ (Swink & Carpenter, 1966) and $Te_2O_3SO_4$ (Hubková, Loub & Syneček, 1966). Both TeO_2 and $Te_2O_4.HNO_3$ have three-dimensional Te–O net structures with four-coordinated Te^{IV} atoms.

Bond distances and angles are given in Table 2. $Te_2O_3SO_4$ has a layer structure (Figs. 2 and 3) in which the SO_4 tetrahedron is strongly connected to the layers *via* two Te–O bonds of 2·26 (1) Å. The layers are held together by van der Waals contacts. A similar structural arrangement has been found in $Te_2O_3HPO_4$ (Mayer, 1975).

Table 1. *Atomic parameters in* $Te_2O_3SO_4$

Positional parameters are given as fractions of the lattice translation. The general point position in $P2_1nm$ is (x,y,z), $(x,y,-z)$, $(\frac{1}{2}+x,-y,\frac{1}{2}-z)$, $(\frac{1}{2}+x,-y,\frac{1}{2}+z)$. The isotropic temperature factor, B, is given in Å^2 and the anisotropic temperature factors correspond to the expression $T=\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+U_{12}hka^*b^*+U_{13}hla^*c^*+U_{23}klb^*c^*)\times 10^{-4}]$. Estimated standard deviations are given in parentheses.

	x	y	z	B	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te	0†	0·1783 (1)	0·1991 (1)	0·458 (5)	57 (1)	67 (1)	49 (1)	13 (10)	−7 (9)	−5 (3)
S	0·4236 (9)	0·4809 (5)	0	0·57 (4)	69 (9)	72 (11)	72 (11)	−24 (21)	0	0
O(1)	0·657 (3)	0·338 (2)	0	1·1 (2)	115 (38)	205 (51)	126 (45)	268 (89)	0	0
O(2)	0·248 (2)	0·447 (1)	0·138 (1)	0·8 (1)	133 (28)	105 (26)	96 (27)	−17 (50)	133 (50)	−21 (49)
O(3)	0·088 (3)	0·091 (2)	0	0·6 (1)	106 (36)	70 (33)	50 (32)	−28 (66)	0	0
O(4)	0·635 (2)	0·091 (1)	0·263 (1)	1·1 (1)	84 (25)	116 (29)	257 (44)	−36 (57)	67 (68)	55 (66)
O(5)	0·029 (3)	0·318 (2)	0·5	1·3 (2)	152 (48)	175 (38)	174 (45)	328 (116)	0	0

† Fixed to specify origin along 2_1.

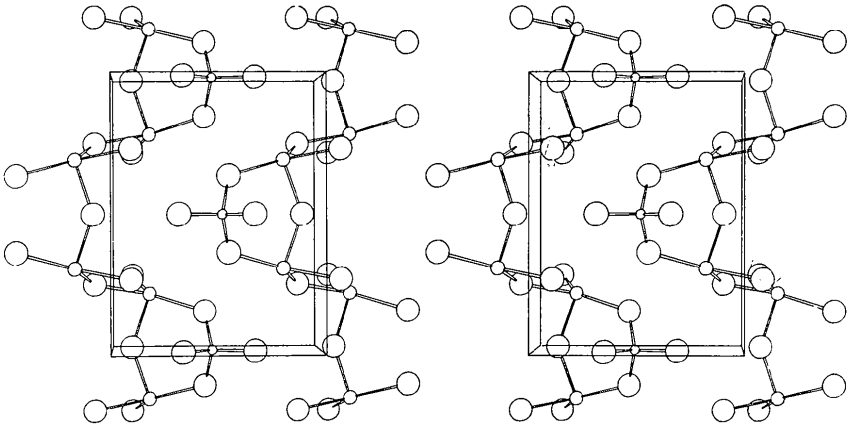

Fig. 2. A stereoscopic picture of the structure projected along the *c* axis (*ORTEP*; Johnson, 1965).

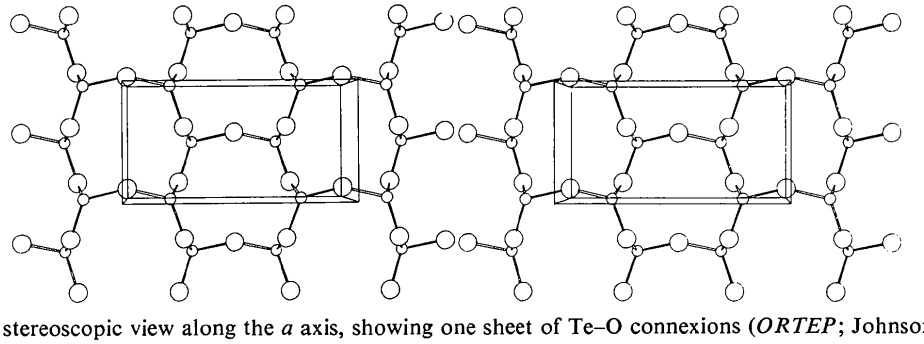

Fig. 3. A stereoscopic view along the *a* axis, showing one sheet of Te–O connexions (*ORTEP*; Johnson, 1965).

Table 2. *Distances and angles in* $Te_2O_3SO_4$

Te—O(4)	1·89 (1)	O(1)–Te–O(2)	78·5 (4)
–O(3)	1·914 (5)	O(1)———O(3)	69·1 (4)
–O′(4)	2·00 (1)	O(1)———O(4)	78·1 (4)
–O(2)	2·26 (1)	O(1)———O′(4)	134·3 (4)
–O(1)	2·63 (1)	O(1)———O(5)	121·1 (4)
–O(5)	2·844 (5)	O(2)———O(3)	86·0 (4)
–O(4)	3·07 (1)	O(2)———O(4)	142·8 (4)
S——O(1)	1·47 (1)	O(2)———O′(4)	130·5 (4)
–O(5)	1·48 (1)	O(2)———O(5)	85·5 (3)
–O(2) (2×)	1·49 (1)	O(3)———O(4)	111·6 (5)
		O(3)———O′(4)	77·9 (5)
O(1)–S–O(2)	107·3 (5)	O(3)———O(5)	164·9 (5)
O(2)–S–O(2)	110·5 (8)	O(4)———O′(4)	86·1 (3)
O(1)–S–O(5)	113·0 (8)	O(4)———O(5)	82·5 (4)
O(2)–S–O(5)	109·3 (5)	O′(4)———O(5)	98·4 (4)

The coordination of Te^{IV} is intermediate between three- and fourfold. Te^{IV} coordination can usually be described in terms of a trigonal bipyramid in which the two shortest Te^{IV}–O bonds are equatorial and the two next shortest are axial while the third equatorial position is occupied by a free electron pair (covalent type; Lindqvist, 1973). However, in $Te_2O_3SO_4$ three of the strongly coordinated O atoms lie in a plane [O(2), O(4) and O(4′)] while the fourth [O(3)] is perpendicular to this plane (Fig. 4). In addition there are two weak Te–O interactions (2·63 and 2·84 Å). The coordination may also be described in terms of an ionic model (Johansson & Lindqvist, 1976).

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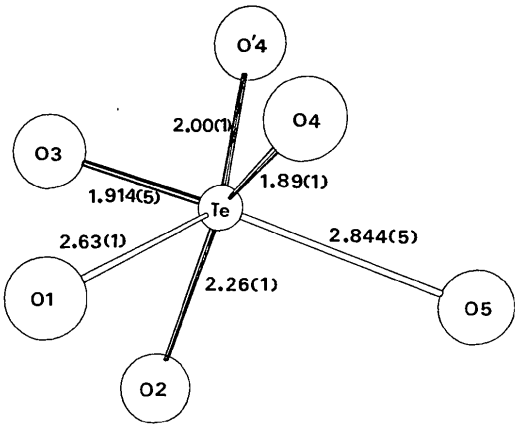

Fig. 4. The oxygen coordination of tellurium(IV).

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