

Ditellurium(IV) Trioxide Sulphate*

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Abstract. $(\text{Te}_2\text{O}_3)\text{SO}_4$, orthorhombic, $P2_1mn$ (No. 31); $a=4.676$ (2), $b=8.911$ (3), $c=6.879$ (4) Å; $Z=2$, $D_m=4.61$ g cm⁻³. The structure consists of $\infty^2\text{Te}_2\text{O}_3$ layers parallel to (001). Layers are connected through sulphate anions which are bound more closely to one of the adjacent layers.

Introduction. $(\text{Te}_2\text{O}_3)\text{SO}_4$ was synthesized by heating orthotelluric acid and 96% sulphuric acid in a sealed ampoule (Hubková, Loub & Syneček, 1966); its structural arrangement was published in the same paper. The intensities were estimated visually from equi-inclination Weissenberg films for two crystals, $0kl$ to $5kl$ [$(\sin \theta)/\lambda=1.38$ Å⁻¹] and $hk0$ to $hk5$ [$(\sin \theta)/\lambda=0.64$ Å⁻¹], and were found to exhibit systematic absences in $hk0$ for $h+k=2n+1$. Corrections were made for Lp, absorption of the Mo $K\alpha$ radiation ($\mu=109$

cm⁻¹, $\mu R=1.84$ and 1.73 respectively) and for the separation of the Mo $K\alpha_{1,2}$ doublet. The set was reduced from 1525 measured reflexions to 613 independent reflexions. The scattering factors of Cromer & Waber (1965) were used. The Patterson map yielded three Te-Te and two Te-S vectors. The remaining atoms were located by Fourier synthesis and refined by least squares to a final $R(=\sum||F_o|-|F_c||/\sum|F_o|)$ of 0.115. The final positional and isotropic thermal parameters are listed in Table 1. The anisotropic temperature parameters did not refine satisfactorily and were mostly non-positive definite. Interatomic distances and angles are presented in Table 2; the projection of the structure along [100] is depicted in Fig. 1.† The cell parameters were refined from a set of d values from a powder pattern (Cu $K\alpha$ radiation) with the program of Burnham (1962). The data reduction was performed on a Hewlett-Packard 9830 calculator with our own programs; the Patterson and electron density maps were computed on an IBM 370/135 computer with *FOURIER* (Weiss, 1973). The least-squares refinement was carried out on a Tesla 200 computer with *ORFLS* (Busing, Martin & Levy, 1962).

* *Editorial note:* - This analysis is an independent determination of a structure already reported [Johansson & Lindqvist, *Acta Cryst.* (1976), B32, 2720-2722]. It has been included as an additional example of the agreement obtained from diffractometer and visual intensities.

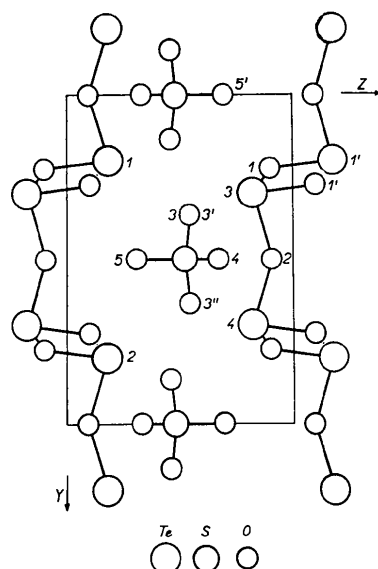


Fig. 1. The projection of the structure along [100]. Primed atoms are outside the limits of the unit cell

Table 1. Fractional atomic coordinates ($\times 10^3$) and isotropic thermal parameters (Å²) with *e.s.d.*'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
Te	750	198.8 (1)	178.7 (2)	0.319 (14)
S	327 (4)	0	477 (1)	0.44 (10)
O(1)	630 (12)	223 (3)	903 (4)	1.2 (4)
O(2)	810 (12)	0	94 (4)	0.6 (3)
O(3)	509 (9)	134 (2)	456 (3)	0.6 (2)
O(4)	90 (18)	0	328 (8)	1.9 (7)
O(5)	210 (17)	0	680 (6)	1.4 (5)

Discussion. The $\infty^2\text{Te}_2\text{O}_3$ layers are composed of -Te-O- screw chains directed along [100]. Each Te atom in a particular chain is coupled with a Te atom of the nearest chain through an O(2) bridge. The CN of Te with respect to O is 3+1, the O atoms being

† A list of structure factors has been deposited with the British Library Division as Supplementary Publication No. SUP 31872 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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

Te(1)–Te(2)	5.368 (2)	Te(3)–O(1) (b)	1.99 (5)
Te(1)–Te(3)	5.083 (3)	–O(2) (a)	1.89 (1)
Te(3)–Te(4)	3.543 (2)	–O(3) (d)	2.29 (3)
Te(3)–Te(1')	3.513 (2)	–O(4)	2.59 (6)
S—O(3')	1.47 (3)	–O(1') (c)	1.99 (3)
–O(4)	1.51 (7)	–O(5')	2.86 (1)
–O(5)	1.50 (5)		

Te(3)–O(1)–Te(1')	124 (1)
Te(3)–O(2)–Te(4)	140 (1)
O(1)–Te(3)–O(2) (ab)	96 (2)
O(2)–Te(3)–O(1') (ac)	81 (1)
O(2)–Te(3)–O(3) (ad)	95 (1)
O(1)–Te(3)–O(1') (bc)	87 (2)
O(1)–Te(3)–O(3) (bd)	140 (3)
O(3)–Te(3)–O(1') (cd)	133 (2)
O(3')–S—O(3'')	108 (1)
O(3')–S—O(4)	111 (3)
O(3')–S—O(5)	108 (2)
O(4)–S—O(5)	112 (3)

located on the vertices of a distorted trigonal bipyramid, with equatorial Te–O distances equal to (a), (b) and axial distances equal to (c), (d) [see Table 2, values labelled according to Zemmann (1971) and Lindqvist

(1973)]. The average Te–O distances are 1.957 and 2.04 Å for the CN's 3+1 and 3 respectively. The average angles in the bipyramid are 96, 101 and 133°, the ideal values being 120, 90 and 180°. The average S–O distance in the sulphate anion is 1.487 Å, and the average O–S–O angle 109.7°. The shortest Te–O distance to the closer sulphate anion is 2.29 and to the more distant one 2.89 Å.

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Cyclo-*N*-methyl-L-alanyl-L-alanyl (c-*N*-Me-L-Ala-L-Ala)

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Abstract. C₁₇H₁₂O₂N₂, *P*₂₁₂₁, *a* = 5.9775 (11), *b* = 9.5370 (21), *c* = 14.6981 (22) Å, *V*_c = 837.9 Å³, *Z* = 4, *D*_c = 1.24 g cm⁻³. The diketopiperazine ring assumes a boat conformation with both methyl substituents quasi-axial.

Introduction. Data were collected from a crystal of dimensions approximately 0.4 × 0.3 × 0.3 mm. Systematic absences of *h*00 for *h* = 2*n* + 1, 0*k*0 for *k* = 2*n* + 1 and 00*l* for *l* = 2*n* + 1 indicated space group *P*₂₁₂₁. Cell dimensions were measured with Cu *K*α radiation on a Siemens four-circle diffractometer by the five-values measuring technique as described, for example, by Allen, Roger & Troughton (1971). 995 reflexions, of which 954 had *I* ≥ 3σ, were measured in the range 0 ≤ θ ≤ 70° with a scan width of 0.6–1.0° determined from the resolution function of the instrument. Data reduction was performed by means of the program *SIEM1* written for the Siemens diffractometer. Normalized structure factors were calculated and phases determined by the program *MULTAN* (Ger-

main, Main & Woolfson, 1971). All 11 non-H atoms were found by calculation of an *E* map from the phase set with highest ABSFOM (1.13). Three cycles of full-matrix least-squares refinement with isotropic temperature factors reduced *R* (= ∑||*F*_o| – |*F*_c||/∑|*F*_o|) to 0.11 following which a difference Fourier synthesis revealed unambiguously all 12 H atoms. Further anisotropic refinement with elimination of seven very strong reflexions for which the agreement between |*F*_o| and |*F*_c| was very poor reduced *R* to its final value of 0.0374 for all observed reflexions.*

Atomic coordinates and temperature factors from the final cycle of refinement are given in Tables 1 and 2. All Fourier and refinement calculations were carried out with the X-RAY 70 system on the PDP10 computer of the Institut Laue-Langevin.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31880 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.