

The Crystal Structure of Te_4O_9

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Single crystals of Te_4O_9 have been prepared by hydrothermal synthesis. The structure has a hexagonal cell with $a=b=9.320$, $c=14.486$ Å. Unusual extinctions indicate that the crystals are disordered. The structure is built up of covalent $(\text{TeO}_3 \cdot 3\text{TeO}_2)_n$ layers and the disorder arises since these layers can be stacked either normally or inverted. It was possible to divide the reflexions into three classes, corresponding to contributions from the normally stacked structure, from the inverted structure, and from both. The normally stacked structure crystallizes in space group $R\bar{3}$. It was refined to an R of 0.042 and revealed the expected Te^{IV} and Te^{VI} coordination in the layers. Te^{VI} is octahedrally surrounded by oxygen atoms with $\text{Te}^{\text{VI}}\text{-O}=1.903$ and 1.948 Å. The four strong $\text{Te}^{\text{IV}}\text{-O}$ bonds of 1.883, 1.902, 2.020 and 2.144 Å are directed towards four corners of a trigonal bipyramid.

Introduction

Some crystallographic investigations of the structures of phases in the $\text{TeO}_3\text{-TeO}_2\text{-H}_2\text{O}$ system have been carried out recently (Lindqvist, 1973). A new compound in this system, Te_4O_9 , has been isolated and characterized (Moret & Lindqvist, 1972) and the present study has been undertaken to establish its crystal structure.

The oxygen coordination of Te^{VI} has always been found to be octahedral, while that of Te^{IV} usually varies considerably in different tellurates(IV), according to the structural environment (Zemann, 1971; Lindqvist, 1973). However, Te_2O_5 (Lindqvist & Moret, 1973*a*) and $\text{H}_2\text{Te}_2\text{O}_6$ (Lindqvist & Moret, 1973*b*) have been shown to have closely related structures, the coordination polyhedra of Te^{IV} and Te^{VI} in the one compound showing remarkable similarity to the corresponding polyhedra in the other. Since a comparison of these polyhedra has proved valuable in the clarification of the effect of the environment on the Te^{IV} and Te^{VI} oxygen bonds, it was hoped that the structure of Te_4O_9 , which is another simple $\text{Te}^{\text{IV,VI}}$ oxide, would supply further information.

Experimental

Single crystals of Te_4O_9 were prepared by hydrothermal synthesis at 350°C starting from $\text{Te}(\text{OH})_6$, TeO_2 and H_2O (Moret, 1972). Te_4O_9 has a hexagonal lattice, and the cell dimensions $a=b=9.320$ (5), $c=14.486$ (5) Å, were calculated from measurements on a powder diffractometer (Moret & Lindqvist, 1972). The density was 5.9 g cm^{-3} , which indicated $Z=6$ ($\rho_{\text{calc}}=5.97$ g cm^{-3}). Weissenberg and precession photographs of

different crystals showed no ordinary extinctions other than for $00l$, $l \neq 3n$. On the other hand, only those reflexions with either $-h+k+l=3n$ or $h-k+l=3n$ were present, indicating that Te_4O_9 might be twinned or disordered. It was not possible to detect any continuous intensity distribution in reciprocal space for any of the crystals mounted.

The crystal selected for data collection had well-developed faces (Table 1) and was mounted along c on a Pailred two-circle diffractometer. Graphite-monochromated $\text{Mo K}\alpha$ radiation was used. The intensities were collected by the ω -scan procedure with a scanning speed of $2.5^\circ \text{min}^{-1}$. The background was measured for 24 s at each end of the scan interval, which was varied between 3.0 and 6.0° for different scattering vectors. One quarter of the reciprocal sphere was explored out to $2\theta \sim 60^\circ$. Within this limit reflexions corresponding to the hexagonal lattice were measured and thus the unusual reflexion conditions $-h+k+l=3n$

Table 1. Dimensions of the crystal used for data collection

Plane			d (mm)
h	k	l	
0	0	1	0.088
0	0	-1	0.088
1	1	1	0.102
-2	1	1	0.102
1	-2	1	0.102
2	-1	-1	0.102
-1	2	-1	0.102
-1	-1	-1	0.102

Crystal volume: 0.65×10^{-2} mm^3

and $h-k+l=3n$ could be checked. The 1618 reflexions with $\sigma(I)/I < 0.3$ were defined as significant.

The program *DATAP1*, written by O. Lindgren, Göteborg, was used for the calculation of Lp factors, and the data were corrected for absorption with the program *DATAP2* (Coppens, Leiserowitz & Rabinovich, 1965). $\mu_{\text{Mo K}\alpha} = 164.8 \text{ cm}^{-1}$ was calculated from *International Tables for X-ray Crystallography* (1962), the transmission factors varying from 0.070 to 0.142.

Structure determination

The reflexions with $-h+k+l=3n$, according to the above definition of axes, were generally much stronger than the others. These strong reflexions, corresponding to one of the rhombohedral space groups $R\bar{3}$ or $R\bar{3}$, were selected for a preliminary investigation of the structure. The Patterson function revealed two Te positions in $R\bar{3}$: 6(c) for Te^{VI} and 18(f) for Te^{IV} . A subsequent electron-density calculation gave maxima corresponding to all the O atoms [three positions 18(f)]. A preliminary refinement of the atomic coordinates and isotropic temperature factors gave an R as low as 0.084, although it was evident that this structure model could not explain the weaker intensities, *i.e.* those with $h-k+l=3n$, $l \neq 3n$.

To account for the weak reflexions, different models with hexagonal symmetry lower than $R\bar{3}$ were tried

(*e.g.* $P3, P\bar{3}, P3_1$ and $P3_2$), but none gave satisfactory agreement. In every refinement the structure converged towards the $R\bar{3}$ arrangement.

Since the $R\bar{3}$ structure ought to be essentially correct, according to the results of the refinement, it was studied geometrically to see if some type of disorder might be possible. The structure is built up from layers of covalently connected Te and O atoms perpendicular to c (Fig. 1). These layers are held together by van der Waals forces only, being in accordance with the observed crystal cleavage planes (*cf.* Fig. 2). In the $R\bar{3}$ structure, adjacent layers, defined as $L0$ and $L1$, are related by $[x, y, z] \rightarrow [\frac{2}{3} + x, \frac{1}{3} + y, \frac{1}{3} + z]$ (Fig. 3). If instead the operation $[x, y, z] \rightarrow [\frac{1}{3} + x, \frac{2}{3} + y, \frac{1}{3} + z]$ is considered the resulting transformed layer, $L1'$, (Fig. 4) would correspond to a structure in which c is inverted. As indicated in Figs. 3 and 4 there are no great differences between the inter-layer $\text{O} \cdots \text{O}$ contacts if the packing sequence $L0, L1'$ occurs instead of $L0, L1$. It was therefore assumed that the Te_4O_9 crystals prepared were disordered, *i.e.* that they contained sheets of normal layers as well as sheets of inverted layers. Since there is no continuous intensity distribution along c^* , each ordered sheet must contain a large number of layers (Fig. 2).

According to this model, the two inverted structures should have the reflexion conditions $-h+k+l=3n$ (normal structure) and $h-k+l=3n$ (inverted structure).

Table 2. Final atomic parameters in the Te_4O_9 layers

The fractional coordinates refer to the normal $R\bar{3}$ symmetry. The anisotropic temperature factor is

$$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2hka^*b^*U_{12} + 2hla^*c^*U_{13} + 2klb^*c^*U_{23}) \times 10^{-4}].$$

	x	y	z	$B(\text{\AA}^2)$		
Te^{IV} in 6(c)	$\frac{1}{3}$	$\frac{2}{3}$	0.48473 (8)	0.42 (4)		
Te^{IV} 18(f)	0.73555 (7)	0.02099 (8)	0.42020 (4)	0.57 (3)		
O(1) 18(f)	0.3650 (10)	0.8458 (10)	0.5667 (6)	0.80 (13)		
O(2) 18(f)	0.8218 (10)	0.3153 (11)	0.5928 (5)	0.66 (12)		
O(3) 18(f)	0.2581 (11)	0.0282 (10)	0.4543 (5)	0.98 (13)		
	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te^{VI}	51 (5)	51 (5)	38 (3)	26 (5)	0	0
Te^{IV}	66 (3)	73 (3)	48 (2)	34 (2)	3 (2)	-15 (2)
O(1)	78 (33)	72 (33)	71 (21)	10 (27)	5 (25)	-83 (25)
O(2)	88 (35)	141 (36)	53 (21)	97 (30)	-34 (25)	-13 (25)
O(3)	124 (36)	129 (36)	64 (22)	35 (30)	-28 (25)	88 (25)

Isotropic extinction parameter (Coppens & Hamilton, 1970): $g = 1.36 (14) \times 10^4$.

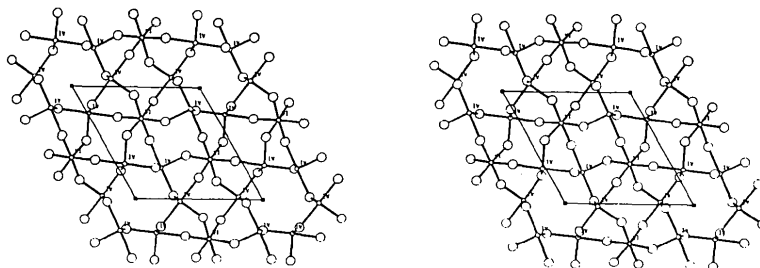


Fig. 1. Stereoscopic picture of part of an infinite layer, $L0$, in Te_4O_9 .

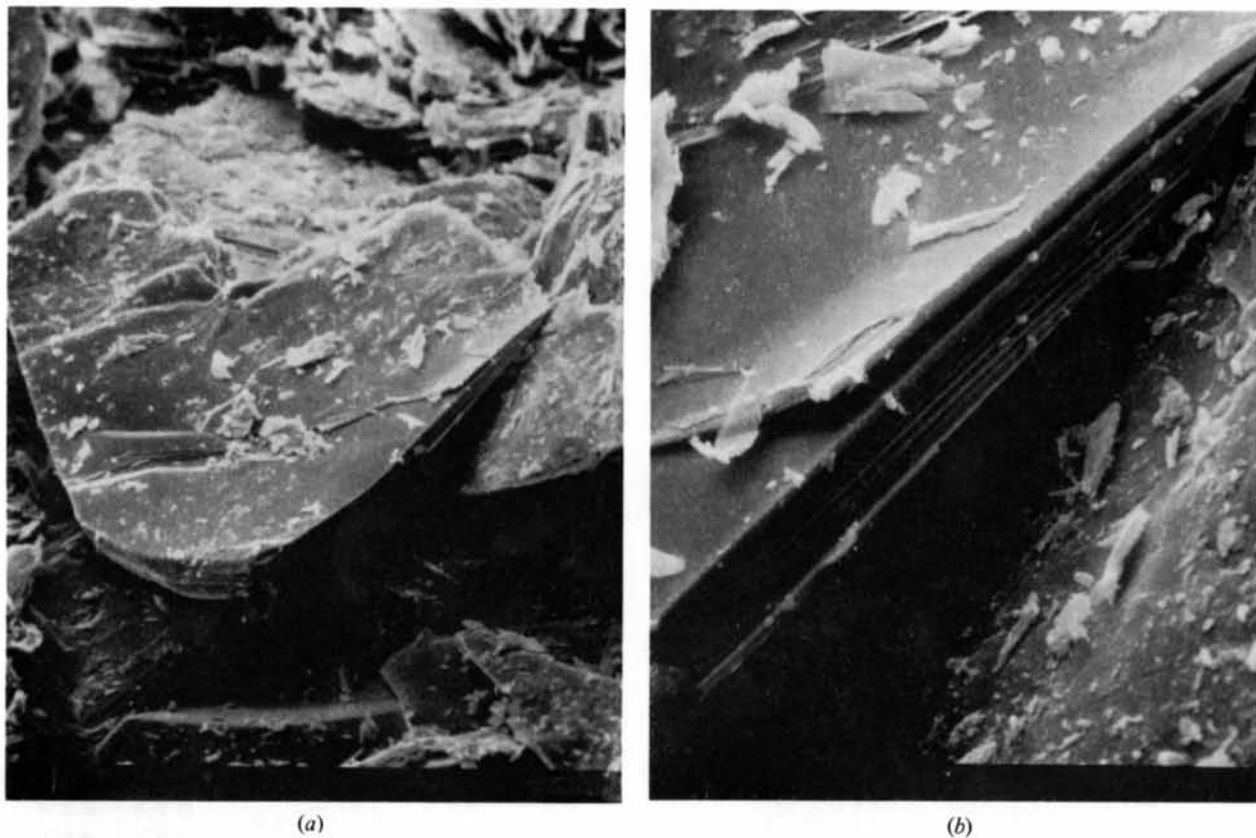


Fig. 2. Electron micrographs of a fragment of a Te_4O_9 crystal. The cleavage planes perpendicular to the c axis are visible. The magnifications are (a) $360\times$ and (b) $1800\times$.

To confirm the proposed arrangement the two classes of reflexions were treated separately, and those reflexions fulfilling both conditions, *i.e.* with $l=3n$, were discarded. A preliminary refinement based on the weaker set of reflexions ($h-k+l=3n, l\neq 3n$), with atomic parameters corresponding to the inverted structure, gave an *R* of 0.11, which verifies the hypothesis given above.

Table 3. *Observed and calculated structure factors for the normal structure of Te₄O₉*

The columns are *l*, *F_o* and *F_c*, respectively.

-10 0 L	11 31 27	37 73 00	-11 3 87	-3 4 L	11 551 466
5 4 61 62	10 10 138	20 20 0	1 32 87	2 83 36	2 83 36
8 151 130		5 125 110	7 41 62	5 585 582	17 129 100
11 114 113		8 79 73	10 63 61	10 43 47	20 71 75
-8 0 L	10 80 82	1 211 212	-10 1 L	11 130 114	
1 127 127	10 129 134	7 183 181	2 115 117	11 130 114	10 67 72
7 184 181	10 153 154	5 125 110	5 146 146	17 261 250	15 82 78
10 42 47		13 85 86	8 100 100	17 261 250	13 82 78
13 174 170			10 135 132	10 135 132	13 82 78
16 137 145	1 142 138	9 1 L	14 96 87	14 96 87	19 108 115
-17 0 L	17 86 100	2 87 86	-8 1 L	7 32 34	-8 1 L
2 87 86	2 87 86	4 203 201	1 36 36	1 36 36	5 112 111
5 354 368	8 126 126	5 125 110	4 291 296	13 81 79	8 102 103
8 126 126	5 125 110	7 183 181	7 183 181	10 135 132	11 924 535
10 42 47	5 125 110	11 86 66	11 86 66	19 111 109	14 63 68
13 174 170	-11 2 L	2 129 132	-11 2 L	2 129 132	17 100 111
16 137 145	11 86 66	5 99 94	5 99 94	5 99 94	17 100 111
-17 0 L	17 86 100	11 86 66	-17 0 L	17 86 100	10 71 75
2 87 86	2 87 86	11 86 66	2 87 86	2 87 86	5 226 232
5 354 368	-11 2 L	5 125 110	-11 2 L	5 125 110	8 204 210
8 126 126	5 125 110	7 183 181	7 183 181	7 183 181	11 80 87
10 42 47	5 125 110	11 86 66	11 86 66	11 86 66	14 63 68
13 174 170	11 86 66	5 99 94	5 99 94	5 99 94	20 132 131
16 137 145	17 86 100	11 86 66	17 86 100	17 86 100	
-17 0 L	17 86 100	11 86 66	-17 0 L	17 86 100	
2 87 86	2 87 86	11 86 66	2 87 86	2 87 86	
5 354 368	5 125 110	7 183 181	5 354 368	5 125 110	
8 126 126	7 183 181	11 86 66	8 126 126	7 183 181	
10 42 47	11 86 66	5 99 94	10 42 47	11 86 66	
13 174 170	17 86 100	11 86 66	13 174 170	17 86 100	
16 137 145	17 86 100	11 86 66	16 137 145	17 86 100	
-17 0 L	17 86 100	11 86 66	-17 0 L	17 86 100	
2 87 86	2 87 86	11 86 66	2 87 86	2 87 86	
5 354 368	5 125 110	7 183 181	5 354 368	5 125 110	
8 126 126	7 183 181	11 86 66	8 126 126	7 183 181	
10 42 47	11 86 66	5 99 94	10 42 47	11 86 66	
13 174 170	17 86 100	11 86 66	13 174 170	17 86 100	
16 137 145	17 86 100	11 86 66	16 137 145	17 86 100	
-17 0 L	17 86 100	11 86 66	-17 0 L	17 86 100	
2 87 86	2 87 86	11 86 66	2 87 86	2 87 86	
5 354 368	5 125 110	7 183 181	5 354 368	5 125 110	
8 126 126	7 183 181	11 86 66	8 126 126	7 183 181	
10 42 47	11 86 66	5 99 94	10 42 47	11 86 66	
13 174 170	17 86 100	11 86 66	13 174 170	17 86 100	
16 137 145	17 86 100	11 86 66	16 137 145	17 86 100	
-17 0 L	17 86 100	11 86 66	-17 0 L	17 86 100	
2 87 86	2 87 86	11 86 66	2 87 86	2 87 86	
5 354 368	5 125 110	7 183 181	5 354 368	5 125 110	
8 126 126	7 183 181	11 86 66	8 126 126	7 183 181	
10 42 47	11 86 66	5 99 94	10 42 47	11 86 66	
13 174 170	17 86 100	11 86 66	13 174 170	17 86 100	
16 137 145	17 86 100	11 86 66	16 137 145	17 86 100	

For the continued refinement of the structural parameters of Te₄O₉, only the reflexions with $-h+k+l=3n, l\neq 3n$ were used. These reflexions were generally strong and had been measured with good statistics. The *F_o/F_c* scale factors for the two different sets of data indicated that the inverted structure only occurred to an extent of ~12% in the crystal used for data collection.

Since the reflexions selected can be considered to correspond to a fully ordered structure, the final refinement could be performed in the usual manner. After inclusion of anisotropic temperature factors and an isotropic extinction parameter (*g*) a final *R* of 0.042 was obtained. The atomic parameters with their standard deviations are given in Table 2. A difference synthesis showed a maximum electron density of 1.5 e Å⁻³. Observed and calculated structure factors for the 495 independent reflexions used in the final refinement are given in Table 3.

Scattering factors given by Cromer & Waber (1965) were used for Te and those by Doyle & Turner (1968) for O. The Te scattering factors were corrected for the real part of the anomalous dispersion (Cromer, 1965). The weights used in the refinement were calculated from $w=(a+F_o+cF_o^2+dF_o^3)^{-1}$ (Cruikshank, 1970) with $a=40.0, c=0.003$ and $d=0.00002$. A weight analysis is given in Table 4.

The Fourier summations were performed with the program *DRF*, written by A. Zalkin, Berkeley, Cali-

Table 4. *Weight analysis after the last cycle of refinement*

The quantities $w\Delta^2$ are normalized sums, $K_{norm}(\sum w|F_o - |F_c||^2)$, and *N* is the number of reflexions within each *F_o* interval.

<i>F_o</i> interval	$w\Delta^2$	<i>N</i>
0 - 46.5	3.49	56
46.5- 71.2	1.02	60
71.2- 91.2	0.41	47
91.2-116.1	0.91	56
116.1-142.2	0.49	46
142.2-171.9	0.50	46
171.9-212.1	0.93	49
212.1-261.9	0.57	45
261.9-345.9	0.63	47
345.9-630.0	0.52	43

$$R = \sum |F_o - |F_c|| / \sum F_o = 0.042$$

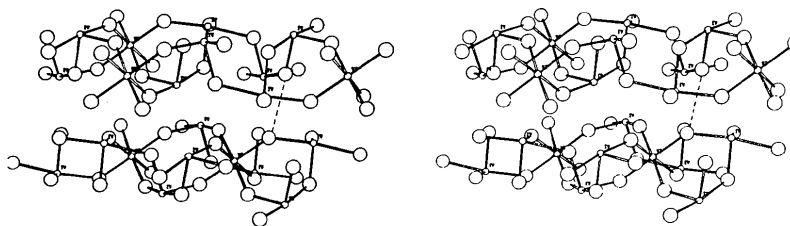
$$R_w = (\sum w|F_o - |F_c||^2 / \sum wF_o^2)^{1/2} = 0.065$$


Fig. 3. Stereoscopic view of two normally packed layers, L0 and L1. The shortest inter-layer oxygen-oxygen [O(2) in L0, O(3) in L1] distance of 2.94 Å is indicated by a dotted line.

fornia, and the refinement with *BLOCK*, written by O. Lindgren, Göteborg, and *LINUS* (Coppens & Hamilton, 1970) for the final cycles. Interatomic distances and angles (*cf.* Tables 5 and 6) were calculated with the program *DISTAN* written by A. Zalkin.

Table 5. Distances (Å) and angles (°) within the tellurium oxygen polyhedra

The notation is in accordance with Figs. 5 and 6.

$\text{Te}^{\text{VI}}\text{-O}(2)$	1.903 (8) (3 ×)	$\text{Te}^{\text{IV}}\text{-O}(1)$	1.902 (9)
$\text{Te}^{\text{VI}}\text{-O}(1)$	1.948 (9) (3 ×)	$\text{Te}^{\text{IV}}\text{-O}(3)$	1.883 (8)
$\text{O}(1)\text{-Te}^{\text{VI}}\text{-O}(1)$	86.7 (4)	$\text{Te}^{\text{IV}}\text{-O}(2)$	2.020 (9)
$\text{O}(1)\text{-Te}^{\text{VI}}\text{-O}(2)$	94.6 (4)	$\text{Te}^{\text{IV}}\text{-O}(3')$	2.144 (8)
$\text{O}(1)\text{-Te}^{\text{VI}}\text{-O}(2)$	90.1 (4)	$\text{Te}^{\text{IV}}\text{-O}(3'')$	2.820 (9)
$\text{O}(2)\text{-Te}^{\text{VI}}\text{-O}(2)$	88.7 (3)	$\text{O}(1)\text{-Te}^{\text{IV}}\text{-O}(2)$	88.3 (4)
$\text{Te}^{\text{VI}}\text{-O}(1)\text{-Te}^{\text{IV}}$	135.1 (5)	$\text{O}(1)\text{-Te}^{\text{IV}}\text{-O}(3)$	98.5 (4)
$\text{Te}^{\text{VI}}\text{-O}(2)\text{-Te}^{\text{IV}}$	138.5 (4)	$\text{O}(1)\text{-Te}^{\text{IV}}\text{-O}(3')$	90.1 (3)
$\text{Te}^{\text{IV}}\text{-O}(3)\text{-Te}^{\text{IV}}$	118.1 (4)	$\text{O}(2)\text{-Te}^{\text{IV}}\text{-O}(3)$	90.8 (3)
		$\text{O}(2)\text{-Te}^{\text{IV}}\text{-O}(3')$	172.0 (3)
		$\text{O}(3)\text{-Te}^{\text{IV}}\text{-O}(3')$	81.7 (3)

Table 6. Inter-layer oxygen-oxygen distances below 4.0 Å

(a) Normally packed layers

$\text{O}(1)\text{-O}(1)$	3.282 (15) (2 ×)
$\text{O}(1)\text{-O}(3)$	3.720 (12)
$\text{O}(2)\text{-O}(2)$	3.593 (17)
$\text{O}(2)\text{-O}(3)$	2.943 (11)

(b) One layer inverted

$\text{O}(1)\text{-O}(2)$	2.782 (11)
$\text{O}(1)\text{-O}(2)$	3.139 (11)
$\text{O}(1)\text{-O}(2)$	3.959 (12)
$\text{O}(1)\text{-O}(3)$	3.288 (11)
$\text{O}(2)\text{-O}(2)$	3.793 (11)
$\text{O}(2)\text{-O}(3)$	3.487 (11)
$\text{O}(2)\text{-O}(3)$	2.920 (11)

Discussion

The layers found in Te_4O_9 are built up from $\text{Te}^{\text{VI}}\text{O}_6$ octahedra and $\text{Te}^{\text{IV}}\text{O}_4$ units which share corners (Fig. 1). The three-dimensional structure is formed by the stacking of such layers upon each other in the *c* direction. The disorder, as described above, may occur since the shortest inter-layer O-O van der Waals distances between normal layers are only slightly longer than those obtained if one layer is inverted (Table 6).

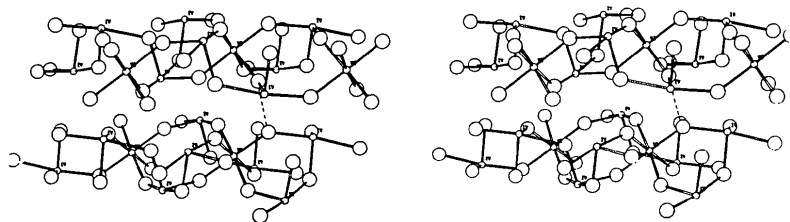


Fig. 4. Stereoscopic view of two layers (L_0, L_1') where the above layer is inverted in relation to layer L_1 in Fig. 3. The dotted line indicates the shortest inter-layer oxygen-oxygen distance of 2.78 Å [$\text{O}(2)$ in L_0 , $\text{O}(1)$ in L_1'].

The identical building units in the two possible stacking sequences suggest that fragments of infinite layers shown in Fig. 1 might be present in aqueous solution during the hydrothermal preparation. Formally, it is difficult to classify the disorder found in Te_4O_9 . It does not conform to the definition of OD structures (Dornberger-Schiff, 1966), since the vicinity condition required for such structures is not fulfilled. Nor is it a twin, since no twin plane is present.

The coordination polyhedra of Te^{VI} and Te^{IV} in Te_4O_9 (Figs. 5 and 6) are very similar to those in Te_2O_5 (Lindqvist & Moret, 1973*a*) and in $\text{H}_2\text{Te}_2\text{O}_6$ (Lindqvist & Moret, 1973*b*). The octahedral coordination of Te^{VI} deviates somewhat from the ideal symmetry (Table 5) and the difference between the $\text{Te}^{\text{VI}}\text{-O}(2)$ and $\text{Te}^{\text{VI}}\text{-O}(1)$ bond lengths of 1.903 (8) and 1.948 (9) Å has a clear significance.

The four-coordination of Te^{IV} conforms well to the usual description as a trigonal bipyramid with one of the equatorial positions occupied by the free electron pair. The difference between the two axial bonds, 2.020 (9) and 2.144 (8) Å, is of the same magnitude as in $\text{Te}(\text{C}_6\text{H}_4\text{O}_2)_2$ (Lindqvist, 1967) and in $\text{Te}_2\text{O}_4 \cdot \text{HNO}_3$

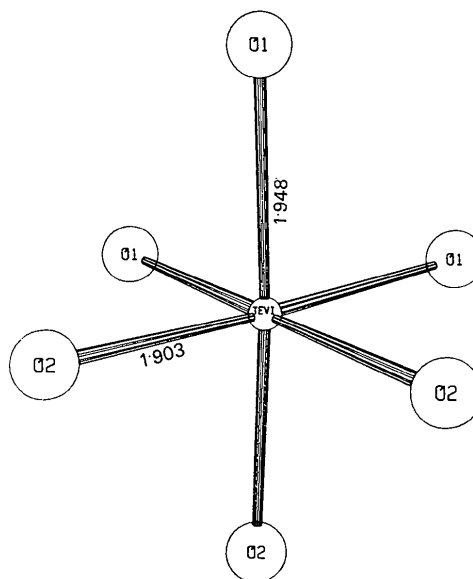


Fig. 5. The octahedral coordination of Te^{VI} with distances in Å.

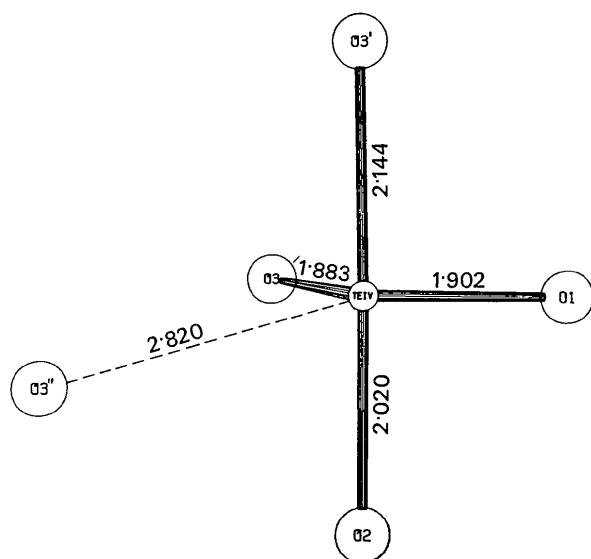


Fig. 6. The four-coordination of Te^{IV} with distances in Å.

(Swink & Carpenter, 1966) and may be explained as a weak tendency towards threefold Te^{IV} coordination. The continuous variation of the fourth $\text{Te}^{\text{IV}}\text{-O}$ length from ~ 2.05 Å in pure fourfold coordination to ~ 3.00 Å in pure threefold coordination has been reviewed previously (Zemann, 1971; Lindqvist, 1973) and has also been discussed in connexion with the investigation of CuTe_2O_5 (Hanke, Kupčik & Lindqvist, 1973).

The Te_2O_5 and the $\text{H}_2\text{Te}_2\text{O}_6$ structure investigations indicated a correlation between the variation in the $\text{Te}^{\text{VI}}\text{-O}$ lengths and the Te^{IV} axial and equatorial bonds. In the $\text{Te}^{\text{VI}}\text{-O}-\text{Te}^{\text{IV}}_{\text{eq}}$ sequence the $\text{Te}^{\text{VI}}\text{-O}$ bond was found to be significantly longer than in the $\text{Te}^{\text{VI}}\text{-O}-\text{Te}^{\text{IV}}_{\text{ax}}$ bridge. The explanation suggested for this effect (Lindqvist & Moret, 1973a) is that the O electrons available for π bonding are engaged in the $\text{Te}^{\text{IV}}\text{-O}_{\text{eq}}$ bond to a much larger extent than in the $\text{Te}^{\text{IV}}\text{-O}_{\text{ax}}$ bond. In the $\text{Te}^{\text{VI}}\text{-O}-\text{Te}^{\text{IV}}_{\text{ax}}$ bridge the oxygen p orbital

available for π bonding can therefore be oriented favourably for π contribution to the $\text{Te}^{\text{VI}}\text{-O}$ bond. This is clearly also the case in Te_4O_9 (Table 5), in which the $\text{Te}^{\text{VI}}\text{-O}$ bonds are 1.903 (8) and 1.948 (9) Å, as the oxygen atom is connected to Te^{IV} in axial or equatorial position, respectively.

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