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The Crystal Structure of $Te_3Nb_2O_{11}$

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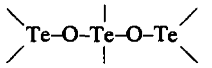
$Te_3Nb_2O_{11}$ crystallizes in space group $P2_12_12$ with $a = 7.700(2)$, $b = 15.700(3)$, $c = 3.980(1)$ Å, and $Z = 2$. Full matrix least squares refinement gave an R value of 0.033 using 521 independent reflections. Infinite double Nb–O octahedra, sharing corners, are connected *via* finite

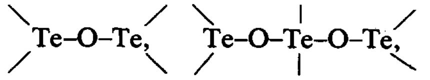


$$\rangle\mathrm{Te{-}O{-}\overset{|}{\underset{|}{Te}}{-}O{-}Te}\langle$$

strings, to form a three-dimensional net. The threefold coordinated Te(IV) atom has the normal pyramidal configuration (Te–O = 1.84, 1.86 and 1.86 Å), while the fourfold coordinated Te(IV) atom has an unusual configuration in which the centre of the Te(IV) atom is placed within a tetrahedron of oxygen atoms. The average Nb–O bond distance of 2.00 Å is normal. However, the Nb atom is displaced from the centre of the oxygen octahedron along the polar axis (chain direction), to give alternating short and long Nb–O distances of 1.80 Å and 2.19 Å, respectively.

Introduction

A variety of structural building units have been found in different tellurate(IV) compounds and discussed by Zemann (*1*) and by the present authors (*2, 3*). Usually Te(IV) has a one-sided three- or fourfold coordination of oxygen atoms. The most simple unit is the pyramidal TeO_3^{2-} ion which is present in $K_2TeO_3{\cdot}3H_2O$ (*4*). Other isolated units containing two or more tellurium(IV) atoms have also been found, e.g. groups of composition Te_2O_5, Te_3O_8 and Te_4O_{11}, containing central chains



$$\rangle\mathrm{Te{-}O{-}Te}\langle,\quad \rangle\mathrm{Te{-}O{-}\overset{|}{\underset{|}{Te}}{-}O{-}Te}\langle,$$

etc. The Te_2O_5 unit was first found in 1965 by Walitzi (*5*) in the mineral denningite, and later also in $CuTe_2O_5$ (*6*), $Te_2V_2O_9$ (*7*) and $Cr_2Te_4O_{11}$ (*8*). The only known structure containing the Te_3O_8 unit is $Zn_2Te_3O_8$, which was investigated by Hanke (*9*) in 1966. Infinite sheets are present in $Te_2O_3SO_4$ (*10*). In α-TeO_2 (*11*) and β-TeO_2 (*12*) the tellurium and oxygen atoms are connected to form three-dimensional network structures.

It is well known that the geometry of the TeO_3 unit (and the TeO_4 unit, when four-coordinated) is strongly dependent on the character of the metal atom in different Me-tellurates. This is probably due to the polarizability of the Te(IV) free electron pair. It is now of interest to investigate whether the central ···Te–O–Te···

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configuration in more condensed tellurates(IV) is stable, or if it is as easily affected by the structural environment as is the TeO_3 coordination polyhedron.

We chose to work with the Nb_2O_5–TeO_2 system, since it would appear to be possible to prepare single crystals of a niobium tellurate(IV) with a structure similar to that of $Te_2V_2O_9$. The preparation of three different phases in the Nb_2O_5–TeO_2 system, namely $TeNb_6O_{17}$, $Te_2Nb_2O_9$ and $Te_4Nb_2O_{13}$ has been described by Guillaume (*13*).

Experimental

Starting from the composition $1Nb_2O_5:2TeO_2$, single crystals of a niobium tellurate(VI) were obtained in a sealed gold tube at a temperature of 750°C. The crystal quality, cell dimensions and space group extinctions were first investigated on a precession camera. The collection of integrated intensities was performed with an automatic 4-circle diffractometer, CAD-4. Important crystal data and constants, and the settings of the diffractometer are given in Table I.

TABLE I

EXPERIMENTAL DATA

1. *Physical and crystallographic data*
   Formula: $Te_3Nb_2O_{11}$
   Crystal system: orthorhombic
   $a = 7.700(2)$ Å
   $b = 15.700(3)$
   $c = 3.979(1)$
   Absorption factor: $(MoK\alpha) = 116$ cm^{-1}
   Morphology: parallelepiped;
   0.0030 × 0.0048 × 0.0012 cm
   Molecular weight: 744.6
   Space group: $P2_12_12$
   $V = 481$ Å^3
   $Z = 2$
   $F(000) = 326$
   $\rho_X = 5.14$ g · cm^{-3}
2. *Data collection*
   Temperature: 20°C
   Radiation: Mo$K\alpha = 0.71069$ Å
   Monochromator: oriented graphite crystal
   Crystal-detector distance: 208 mm
   Detector window[a]: height = 3 mm, width = 2.10 + 1.0 tan θ mm
   Take off angle[a]: 3.0°
   Scan mode: omega
   Maximum bragg angle (θ): 30°
   Scan angle: $\theta = \theta_0 + B \cdot \tan\theta$ $B = 0.35$
   Values determining scan speed:[a] SIGPRE = 0,400 SIGMA = 0.018
   VPRE = 10°/min TMAX = 80 sec

Controls:	Intensity	Orientation
Reflections:	1 4 0, 0 $\bar{3}$ 1, 0 10 0	3 $\bar{7}$ 1, 0 10 0, 3 4 1
Periodicity:	3600 sec	50 reflections

3. *Conditions for refinement*
   Reflections for the refinement of cell dimensions: 25

Recorded reflections:	924
Independent reflections:	787
Significant reflections:	521
Refined parameters:	75
Reliability factors:	$R = \sum\lvert k\lvert F_0 - \lvert F_c\rvert\rvert / \sum k\lvert F_0\rvert = 0.033$
	$R_w = [\sum w^2(k\lvert F_0\rvert - \lvert F_c\rvert)^2 / \sum w^2k^2F_0^2]^{1/2} = 0.036$

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[a] Cf. A. Mossett, J. J. Bonnet, and J. Galy, *Acta Cryst.*, *B* **33**, 2639–2644 (1977).

Structure Determination and Absolute Configuration

It was possible to index the powder diagram reported for $Te_2Nb_2O_9$ by Guillaume (*13*) with the parameters refined on the CAD-4 diffractometer, using 25 strong *hkl* reflections (cf. Table II). The structure determination was then commenced assuming that there were two formula units of $Te_2Nb_2O_9$ in the cell. In the Patterson function most of the predominant peaks could be explained assuming that the Te and Nb atoms were situated in two general positions $P2_12_12:4(c)$. However, it was also evident that there were two further Te atoms in the twofold position $2(a)$. Thus the formula is $Te_3Nb_2O_{11}$, rather than $Te_2Nb_2O_9$. Assuming that the Te(IV) free electron pair occupies the same volume as an oxygen atom (*14, 15*), the new formula corresponds to an average volume of 17.2 $Å^3$/oxygen atom, which agrees well with previously observed values. It proved possible to identify oxygen peaks in the successive electron density maps in accordance with the formula $Te_3Nb_2O_{11}$.

TABLE II

POWDER PATTERN OF $Te_3Nb_2O_{11}$

h k l	d_{obs}(Å)	d_{calc}(Å)	I/I_0
1 1 0	6.90	6.91	7
1 3 0	4.33	4.33	25
0 0 1	3.98	3.98	65
0 1 1 2 0 0	3.853	3.858 3.850	30
2 1 0	3.735	3.739	90
0 2 1 1 0 1	3.534	3.550 3.536	50
1 4 0	3.497	3.497	100
1 1 1 2 2 0	3.453	3.457 3.449	90
1 2 1	3.222	3.224	65
0 3 1	3.166	3.168	70
2 3 0	3.097	3.101	7
1 3 1	2.928	2.930	60
1 5 0	2.908	2.908	60
0 4 1	2.794	2.795	10
2 1 1	2.722	2.725	20
2 2 1	2.609	2.610	45
1 6 0	2.479	2.478	20
3 2 0	2.439	2.440	10
1 5 1	2.348	2.348	7
3 3 0	2.302	2.304	7
2 5 1	2.076	2.076	20
0 0 2	1.990	1.990	20
0 7 1	1.955	1.954	15
4 1 0	1.910	1.911	15
2 6 1	1.889	1.901	30
1 2 2 4 2 0	1.870	1.871 1.870	7
0 3 2	1.860	1.860	7
2 1 2	1.754	1.757	7
2 8 0	1.749	1.748	7
4 4 0	1.727	1.728	7
4 1 1	1.722	1.722	10

A preliminary refinement of the atomic positions and isotropic temperature factors gave an R value of 0.076. At this stage, an absorption correction was applied to the observations, using the program AGNOST. In the final refinement, which included anisotropic temperature factors and an isotropic extinction parameter, the R value dropped to 0.033 ($R_w = 0.036$). The refinement was performed with the full matrix least squares program NUCLS, adapted to the IRIS80 computer by Bonnet. Both the ordinary and the anomalous scattering factors were obtained from the "International Tables," Vol. IV (*16*). The observed structure factors were weighted using unmodified $\sigma(F)$ quantities calculated as $abs \cdot (2F \cdot Lp)^{-1} \cdot \sigma(I)$. Those reflections with $I \geq 2\sigma(I)$ were regarded as being significant. The final atomic parameters are given in Table III and a list of observed and calculated structure factors has been deposited.[1] The orientation given in Table III may be regarded as correct, since a refinement based on a set of identical parameters with reversed signs for the z coordinates gave an R value of 0.036 ($R_w =$

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[1] Structure factor tables have been deposited with the National Auxilary Publications Service (NAPS), P.O. Box 3513, Grand Central Station, New York 10017.

TABLE III

FINAL ATOMIC PARAMETERS FOR $Te_3Nb_2O_{11}$[a]

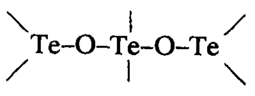
Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Te(1)	0.3104(2)	0.2978(1)	0.8619(4)	52(2)	12(1)	114(8)	3(1)	0(5)	−1(2)
Te(2)	1/2	1/2	0.1443(6)	164(5)	18(1)	70(12)	−27(2)	0	0
Nb	0.3717(2)	0.1014(1)	0.2493(5)	44(3)	7(1)	50(9)	1(1)	10(6)	1(3)
0(1)	0.355(2)	0.107(1)	0.699(4)	92(30)	34(7)	143(110)	25(14)	−11(52)	7(25)
0(2)	0.247(2)	0.217(1)	0.181(4)	32(22)	18(7)	292(129)	18(10)	−30(49)	18(24)
0(3)	1/2	0	0.200(5)	95(41)	17(8)	41(155)	−13(17)	0	0
0(4)	0.140(2)	0.044(1)	0.187(4)	109(33)	26(7)	342(143)	−32(13)	10(65)	−7(26)
0(5)	0.579(2)	0.172(1)	0.198(5)	70(28)	40(9)	454(159)	−45(14)	−57(58)	5(33)
0(6)	0.360(2)	0.385(1)	0.157(4)	104(32)	24(7)	313(116)	−8(13)	−1(68)	−30(26)

[a] The ansotropic temperature factor is given as $\exp[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})\cdot 10^4]$.

0.039). The $R_w(xy\bar{z})/R_w(xyz)$ ratio is thus 1.083, which is significant according to Hamilton's (*17*) R-value test ($\mathcal{R}_{1,454,0.005} = 1.005$).

Discussion of the Structure

A projection of the structure on the (001) plane is shown in Fig. 1. Interatomic bond distances and angles are listed in Table IV. The niobium atom is octahedrally coordinated by oxygen atoms, while the tellurium(IV) coordination is of two kinds: one Te(IV) atom is threefold pyramidally coordinated while the other has a fourfold coordination. The structure is best described in terms of infinite, double chains of niobium–oxygen octahedra sharing corners (cf. Fig. 2). These chains are cross-linked by finite



$$\begin{array}{c} \diagdown \qquad\quad | \qquad\quad \diagup \\ \text{Te–O–Te–O–Te} \\ \diagup \qquad\quad | \qquad\quad \diagdown \end{array}$$

chains. The tellurium atoms are also bonded to oxygen atoms in the Nb–O octahedra, thus forming a three-dimensional net structure with Nb–O octahedral chains and Te_3O_8 groups, as indicated in Fig. 1.

The niobium–oxygen coordination distances (Table IV) all lie within the range 1.73–2.31 Å found in Nb_2O_5 by Gatehouse and Wadsley (*18*). The average Nb-O distance of 2.00 Å also compares well with the corresponding value of 1.99 Å in Nb_2O_5.

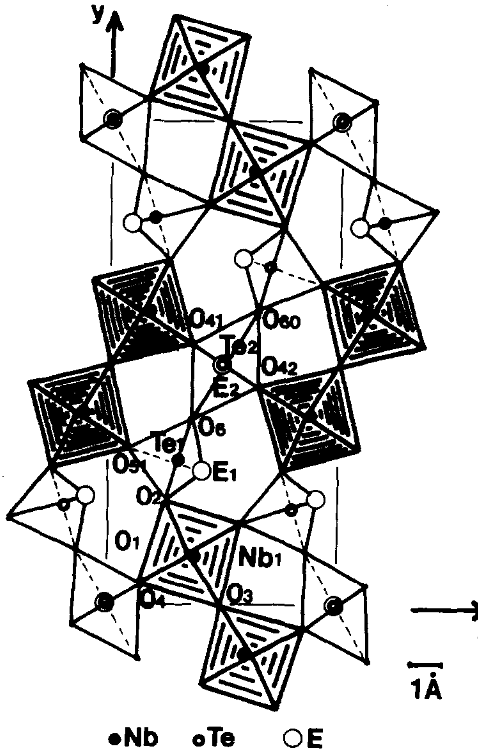


FIG. 1. Projection of the structure of $Te_3Nb_2O_{11}$ onto the (001) plane. The different polyhedra NbO_6 (octahedron), $Te(1)O_3E(1)$ (tetrahedron) and $Te(2)O_4E(2)$ (trigonal bipyramid) share corners only (E = Te(IV) free electron pair).

TABLE IV

INTERATOMIC BOND DISTANCES AND ANGLES IN $Te_3Nb_2O_{11}$[a]

Te(1)–O(2)	1.86(2) Å	O(2)–O(4)	2.83(2) Å
Te(1)–O(51)	1.86(2)	O(2)–O(5)	2.65(2)
Te(1)–O(6)	1.84(2)	O(2)–O(51)	2.64(2)
Te(2)–O(41)	1.84(2)	O(2)–O(6)	2.78(2)
Te(2)–O(42)	1.84(2)	O(3)–O(4)	2.86(2)
Te(2)–O(6)	2.10(2)	O(3)–O(5)	2.76(2)
Te(2)–O(60)	2.10(2)	O(41)–O(42)	2.47(3)
Nb–O(1)	1.80(1)	O(41)–O(6)	2.78(2)
Nb–O(1)	2.19(1)	O(42)–O(6)	2.85(2)
Nb–O(2)	2.07(1)	O(51)–O(6)	2.73(2)
Nb–O(3)	1.885(3)		
Nb–O(4)	2.01(1)		
Nb–O(5)	1.95(1)		
O(1)–O(2)	2.82(2)	O(2)–Te(1)–O(51)	90.6(7)°
O(1)–O(3)	2.83(2)	O(2)–Te(1)–O(6)	97.2(7)
O(1)–O(4)	2.80(2)	O(51)–Te(1)–O(6)	95.1(8)
O(1)–O(5)	2.83(2)		
O(1')–O(2)	2.71(2)	O(41)–Te(2)–O(42)	88.4(9)
O(1')–O(3)	2.83(2)	O(6) –Te(2)–O(60)	182.8(9)
O(1')–O(4)	2.73(2)		
O(1')–O(5)	2.82(2)	Te(1)–O(6)–Te(2)	136.6(9)

[a] The notation is in accordance with Fig. 1.

The Nb–Nb distance across the chains in $Te_3Nb_2O_{11}$ is 3.784 Å, which is in good agreement with the value 3.822 Å in Nb_2O_5. However, along the chains in $Te_3Nb_2O_{11}$, the Nb–Nb distance is longer and is identical with the *c*-axis separation, i.e. 3.979 Å. It is interesting to note that the shortest and longest Nb–O distances of 1.80 Å and 2.19 Å, respectively, also occur alternatingly in the ···O–Nb–O–Nb··· chain along the *c* axis (cf. Fig. 2). The corresponding displacement of the niobium atom from the central plane in the oxygen octahedron is 0.22 Å. A similar arrangement exists in ferroelectric barium sodium niobate whose structure has been determined by Jamieson, Abrahams, and Bernstein (*19*). In this structure there are four independent niobium atoms in the asymmetric unit of space group *Cmm*2. The displacements of the niobium atoms from the central oxygen planes lie in the range 0.17–0.21 Å, all in the same direction in the crystal. The corresponding Nb–Nb distance is also increased, being 3.99 Å. However, $Te_3Nb_2O_{11}$ cannot exhibit spontaneous ferroelectricity, since it belongs to space group $P2_12_12$, which is without a polar direction.

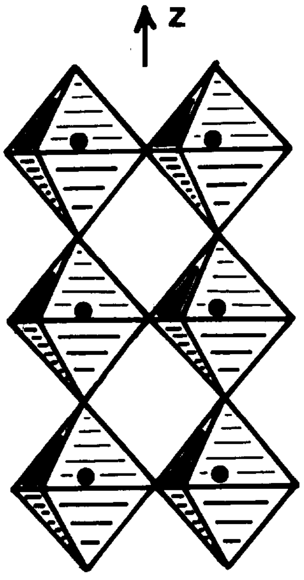


FIG. 2. Infinite double chains of NbO_6 octahedra sharing corners.

The threefold coordinated Te(1) atom has a relatively symmetrical pyramidal arrangement of oxygen atoms (Table IV). This indicates that the nonbonded electron pair of Te(1) is essentially unaffected by the surrounding oxygen atoms. Accordingly the shortest Te–O distance, behind Te(1), is 3.03 Å. The coordination of Te(1) agrees well with that of the TeO_3^{2-} ion in $K_2TeO_3 \cdot 3H_2O$ (*4*), in which the average value for the Te–O bond length is 1.85 Å. It also fits well in the general scheme described for the (18+2) shell ions Ge(II)···Bi(III) (*15, 20*).

The fourfold coordinated Te(2) atom in $Te_3Nb_2O_{11}$ lies on a twofold rotation axis, which is also the case in α-TeO_2 (*11*) and in $Zn_2Te_3O_8$ (*9*). This type of coordination is usually described as a trigonal bipyramid with one of the equatorial positions occupied by the Te(IV) free electron pair.

It is useful to describe the free electron pair as a sphere with a volume similar to that of an oxygen atom (*15*). Although the volume does not appear to alter appreciably (*15*) it is reasonable to assume that the packing of oxygen atoms around the free electron pair leads to distortion from spherical symmetry, i.e. the free electron pair becomes polarized. Zemann (*1*) has demonstrated that there are preferred orientations of the fifth and sometimes even the sixth nearest oxygen atoms on the "backside" of the four-coordinated tellurium(IV) atom.

The structural arrangement of the oxygen atoms in contact with the free electron pair, i.e. on the "backside" of the tellurium atom, influences the repulsion between the free electron pair and the $Te{-}O_{eq}$ and $Te{-}O_{ax}$ bonding electron pairs. Usually this leads to $O_{ax}{-}Te{-}O_{ax}$ and $O_{eq}{-}Te{-}O_{eq}$ angles of about 165° and 100° instead of the regular trigonal bipyramidal angles of 180° and 120° (Fig. 3). The different values of these angles exhibited in the structures listed in Table V indicate that the free electron pair is strongly polarizable.

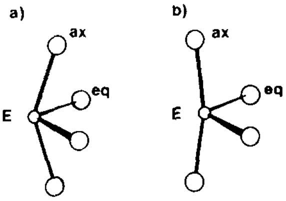
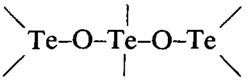


FIG. 3. Schematic representations of the tellurate(IV) trigonal bipyramide: (a) As usually found (cf. Table V); (b) As found in $Te_3Nb_2O_{11}$.

A new feature of the trigonal bipyramidal configuration in $Te_3Nb_2O_{11}$ is that it is not one-sided but $Te{-}O_{ax}$ is slightly bent towards the Te(IV) free electron pair (cf. Fig. 3 and Table V). The oxygen atoms around Te(2) and its free electron pair in $Te_3Nb_2O_{11}$ form a distorted hexagonal bipyramid, and from Fig. 4 it would appear that the free electron pair is elongated in the plane of the equatorial bonds. This ought to lead to increased repulsion in the equatorial plane, but have a lesser effect on the axial bonds, which fits well with the structural results obtained.

On both sides of Te(2), the axial atom O(6) is also bonded to Te(1), resulting in a Te_3O_8 group. This building unit has been previously studied only in $Zn_2Te_3O_8$ (*9*). The arrangement of the central



$$\diagdown\!\!\!\!\!\diagup\, Te{-}O{-}\overset{|}{\underset{|}{Te}}{-}O{-}Te \,\diagup\!\!\!\!\!\diagdown$$

chain is similar in the two structures, both having a twofold symmetry axis through the central Te atom. The Te–O–Te angle in some different tellurates(IV) containing Te_2O_5 and Te_3O_8 groups are compared in Table VI. The variations indicate that the Te–O–Te bridge, in a similar manner to the oxygen coordination of a single Te(IV) atom, is relatively easily affected by the surrounding structural arrangement.

Acknowledgments

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TABLE V

FOURFOLD OXYGEN COORDINATION OF TELLURATES (IV)[a]

Compound	Te–O_{eq}	Te–O_{ax}	O_{eq}–Te–O_{eq}	O_{ax}–Te–O_{ax}	Ref.
α-TeO_2	2×1.90(2) Å	2×2.10(2) Å	102°	169°	(*11*)
Te_2O_5	1.892(6), 1.913(4)	2.072(5), 2.080(6)	91	166	(*21*)
$Zn_2Te_3O_8$	2×1.83(3)	2×2.10(3)	106	175	(*9*)
$Te_3Nb_2O_{11}$	2×1.84(2)	2×2.10(2)	86	183	
$H_2Te_2O_6$	1.861(6), 1.937(4)	2.065(5), 2.107(5)	91	172	(*22*)
$Te(C_6H_4O_2)_2$	1.98(2), 1.98(2)	2.01(3), 2.11(3)	98	154	(*23*)
$Te_2O_3(HPO_4)$	1.79(4), 1.99(4)	2.02(4), 2.12(4)	95	168	(*24*)
$TiTe_3O_8$	2×1.85(2)	2×2.12(2)	102	159	(*25*)
$Te_2O_4NHO_3$	1.88(2), 1.95(1)	2.02(2), 2.16(2)	100	148	(*26*)
$UO_2Te_3O_7$	1.78(4), 2.02(4)	2.15(4), 2.16(4)	96	157	(*27*)
β-TeO_2	1.88(2), 1.93(2)	2.07(2), 2.19(2)	101	168	(*12*)

[a] The compounds are listed in order of the longest Te–O_{ax} bond up to 2.20 Å.

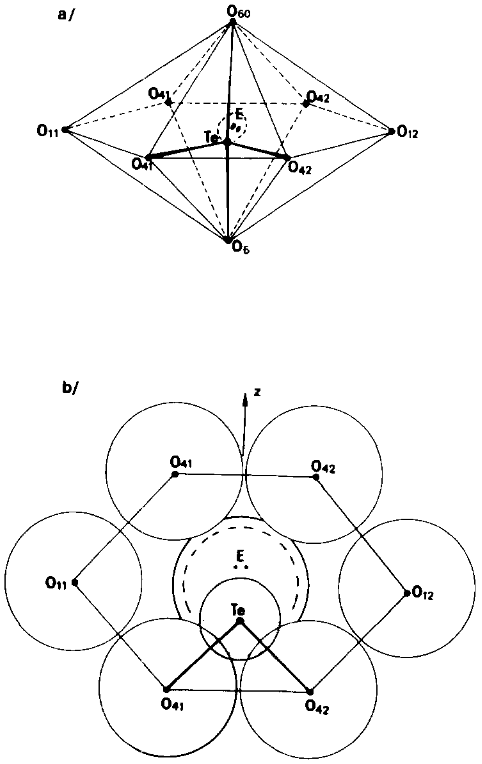

FIG. 4. (a) The distorted hexagonal bipyramid around Te(2); (b) The packing of oxygen atom in the Te(2) equatorial plane.

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TABLE VI

Te–O–Te ANGLES IN SOME TELLURATES (IV)

Compound	Te–O–Te	O–Te distances	Ref.
Cu Te_2O_5	120.6(2)°	1.931(3), 2.019(3) Å	(*6*)
$Zn_2Te_3O_8$	126.0 (2x)	1.98, 2.10	(*9*)
$Te_2V_2O_9$	143.3(2)	1.921(3), 1.921(3)	(*7*)
$(NH_4)_2Te_2O_52H_2O$	118.0(2)	2.030(3), 1.901(3)	(*28*)
$Nb_2Te_3O_{11}$	132.1(9) (2x)	1.84(2), 2.10(2)	

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