SHORT STRUCTURAL PAPERS

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Acta Cryst. (1978). B**34**, 2830–2832

Diammonium Ditellurium(IV) Pentaoxide Dihydrate

BY GÖRAN B. JOHANSSON

Department of Inorganic Chemistry, Chalmers' University of Technology and the University of Göteborg, PO Box, S-402 20, Göteborg 5, Sweden

(*Received* 6 *April* 1978; *accepted* 5 *May* 1978)

Abstract. $(NH_4)_2Te_2O_5.2H_2O$, M_r = 407·30, monoclinic, $P2_1/c$, a = 10·357 (3), b = 11·000 (3), c = 8·178 (2) Å, β = 93·23 (1)°, Z = 4, V = 930·2 (2) Å^3, μ(Mo $K\alpha$) = 65·08 cm^{-1}, D_x = 2·91 g cm^{-3}. Refinement based on 2439 independent reflections gave R = 0·031. The Te_2O_5 groups are connected to form chains, the Te–O bond distances within the Te_2O_5 group ranging from 1·834 (4) to 2·034 (3) Å, while the Te–O bridging distances are 2·218 (3) and 2·506 (4) Å. The coordination of the two independent Te^{IV} atoms is fourfold and can be described as distorted trigonal bipyramidal with the free $5s^2$ electron pair occupying one of the equatorial positions.

Introduction. The preparation of crystalline ammonium tellurate(IV) was described by Lehner & Wolesensky (1913) who suggested the formula $(NH_4)_2TeO_3$. In the present investigation, single crystals of composition $(NH_4)_2Te_2O_5.2H_2O$ were prepared by hydrothermal synthesis. $TeO_2(s)$ was mixed with an excess of concentrated ammonia and held in an autoclave at 100 °C for 24 h, colourless, prismatic crystals, suitable for X-ray work, being obtained. The crystals are unstable in air and were therefore mounted in glass capillaries in a glove-box containing $NH_3(g)$. If the autoclave is opened in air, the $(NH_4)_2Te_2O_5.2H_2O$ crystals deliquesce, a new phase, hexagonal $(NH_4)_2Te_4O_9.4H_2O$, subsequently being formed. These crystals are stable in air and their crystal structure is under investigation.

Data collected with a Syntex $P2_1$ diffractometer are summarized in Table 1. The integrated intensities were evaluated according to the Lehmann & Larsen (1974) profile analysis method using a local program, *LELA* (Lindqvist & Ljungström, 1978). The intensities were corrected for Lp effects with a local program (*SYN*), but it was not possible to apply a correction for absorption as the crystal was submerged in epoxy glue within the glass capillary.

The structure was solved from Patterson and electron density calculations (program *DRF*; A. Zalkin, Berkeley). The structural parameters were refined with a block-diagonal-approximation program (*BLOCK*; Lindgren, 1977). Refinement with isotropic temperature factors gave R = 0·060 and when anisotropic temperature factors were included R dropped to 0·036.

Table 1. *Experimental summary*

Crystal dimensions:	0·10 × 0·09 × 0·19 mm
Radiation:	λ(Mo $K\alpha$) = 0·71069 Å
Monochromator:	Graphite crystal
Scan mode:	$\omega/2\theta$
Maximum 2θ angle:	60·0°
Scan interval ($\Delta\omega$):	2·4–3·6°
Scan speed (in ω):	2·0–12·0° min^{-1}
Standard reflection:	113 at intervals of 25 reflections 67 000 ± 1500 counts, stable
Recorded reflections:	3040
Independent reflections:	2828
Significant reflections:	2439 [$I/\sigma(I) > 3·0$]
Refinement of cell parameters:	15 reflections with $12·4 < 2\theta < 27·4°$

Table 2. *Final atomic parameters in* $(NH_4)_2Te_2O_5.2H_2O$

Estimated standard deviations are given in parentheses [W(1) and W(2) denote the oxygen atoms in the two water molecules].

	x	y	z
Te(1)	0·12346 (2)	0·07289 (2)	0·08168 (3)
Te(2)	0·64698 (2)	−0·01038 (2)	0·61033 (3)
W(1)	0·0822 (5)	0·3678 (4)	0·1236 (6)
O(2)	0·0739 (3)	0·4370 (3)	0·7930 (4)
O(3)	0·0843 (3)	0·5850 (3)	0·4991 (5)
N(1)	0·1610 (4)	0·7161 (4)	0·1850 (6)
W(2)	0·3344 (5)	0·3103 (5)	0·2807 (7)
O(6)	0·3119 (3)	0·6501 (3)	0·9232 (4)
O(7)	0·3021 (3)	0·4907 (3)	0·6538 (4)
N(2)	0·4552 (4)	0·6438 (4)	0·4553 (6)
O(9)	0·4741 (3)	0·4996 (3)	0·1634 (5)

This relatively large difference and low *B* values for Te indicate that the anisotropic temperature factors may be influenced by absorption effects. It was evident that a few strong reflections were subject to secondary extinction. Eight such reflections were therefore discarded giving a final *R* of 0·031. The corresponding structural parameters are listed in Table 2.* Atomic scattering factors due to Cromer & Waber (1965) were used for Te, while the values given by Doyle & Turner (1968) were used for O and N. The real part of the anomalous scattering for Te (Cromer, 1965) was taken into account. Weights were calculated according to the formula $w = (a + F_o + cF_o^2 + dF_o^3)^{-1}$ (Cruickshank, 1970) with $a = 40{\cdot}0$, $c = 0{\cdot}04$ and $d = 0{\cdot}0001$.

Discussion. Interatomic distances and angles were calculated with the program *DISTAN* (A. Zalkin, Berkeley) and are presented in Fig. 1 and Table 3. The structure of $(NH_4)_2Te_2O_5.2H_2O$ consists of Te_2O_5 groups connected *via* double oxygen bridges to form chains (Fig. 2). The chains are held together by hydrogen bonds involving the ammonium ions and water molecules (Fig. 3).

The coordination of the two independent Te atoms can be described as fourfold. The four Te—O bonds are directed towards the axial and two of the equatorial corners of a trigonal bipyramid. The third corner in the equatorial plane is occupied by the $5s^2$ free electron pair. In α-TeO_2 (Lindqvist, 1968) the axial bonds are significantly longer than the equatorial bonds, which are 2·10 and 1·90 Å respectively. The coordination around Te(1) in the present study is a good example of Te^{IV} fourfold coordination (Fig. 1) while that of Te(2) has elements of threefold coordination, one of the axial bonds being short [Te(2)—O(7) = 1·971 (4) Å] and the other being considerably longer [Te(2)—O(9′) = 2·506 (4) Å].

The ideal O_{ax}—Te—O_{ax} and O_{eq}—Te—O_{eq} angles are 180 and 120°, respectively, but the repulsion between the bonding electrons and the free electron pair results in decreased angles. In $(NH_4)_2Te_2O_5.2H_2O$ the axial

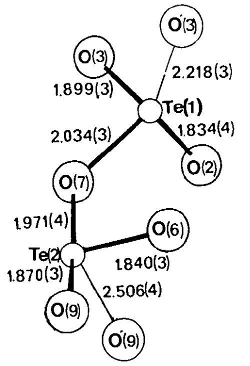

Fig. 1. The tellurium–oxygen coordination.

Table 3. *Distances* (Å) *and angles* (°) *within the* Te—O *coordination polyhedra in* $(NH_4)_2Te_2O_5.2H_2O$ (*cf.* *Fig.* 1)

Te(1)—O(2)	1·834 (4)	O(2)—Te(1)—O(3′)	88·0 (1)
—O(3)	1·899 (3)	O(2)—Te(1)—O(3)	102·4 (2)
—O(7)	2·034 (3)	O(2)—Te(1)—O(7)	90·3 (2)
—O(3′)	2·218 (3)	O(3)—Te(1)—O(3′)	76·4 (1)
—*W*(1)	3·292 (5)	O(3′)—Te(1)—O(7)	163·3 (1)
		O(3)—Te(1)—O(7)	87·8 (1)
Te(2)—O(6)	1·840 (3)		
—O(9)	1·870 (3)	O(6)—Te(2)—O(7)	95·1 (2)
—O(7)	1·971 (4)	O(6)—Te(2)—O(9′)	86·2 (1)
—O(9′)	2·506 (4)	O(6)—Te(2)—O(9)	101·9 (2)
—O(2)	3·010 (4)	O(7)—Te(2)—O(9′)	165·2 (1)
—*W*(2)	3·420 (5)	O(7)—Te(2)—O(9)	88·8 (2)
		O(9)—Te(2)—O(9′)	76·5 (2)

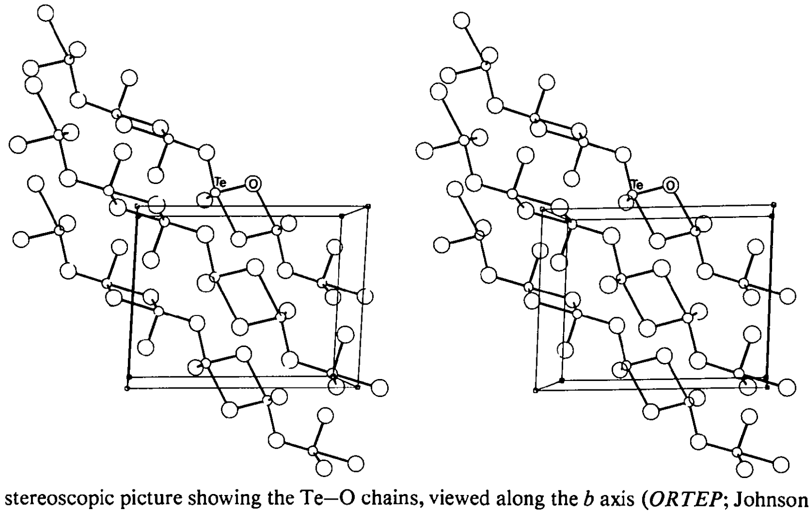

Fig. 2. A stereoscopic picture showing the Te—O chains, viewed along the *b* axis (*ORTEP*; Johnson, 1965).

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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 33597 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

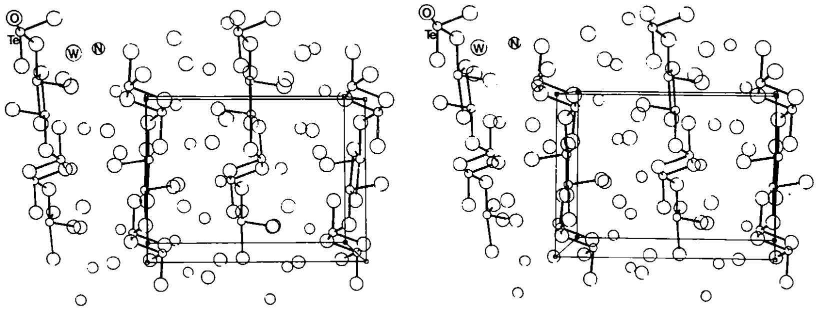

Fig. 3. The $(NH_4)_2Te_2O_5.2H_2O$ structure viewed along the *a* axis (*ORTEP*; Johnson, 1965).

Table 4. *Possible hydrogen bonds*

W(1) and *W*(2) denote the oxygen atoms in the two water molecules.

W(1)···O(2)	2·796 (6) Å	O(2)···*W*(1)···O(2)	92·3 (2)°
···O(2)	2·805 (6)	O(2)···*W*(1)···*W*(2)	117·5 (2)
···*W*(2)	2·914 (7)	O(2)···*W*(1)···*W*(2)	125·1 (2)
W(2)···O(9)	2·740 (6)	*W*(1)···*W*(2)···N(2)	150·6 (2)
···*W*(1)	2·914 (7)	*W*(1)···*W*(2)···N(2)	99·1 (2)
···N(2)	3·022 (7)	N(2)···*W*(2)···O(9)	75·5 (2)
N(1)···O(3)	2·756 (6)	O(2)···N(1)···O(3)	55·6 (1)
···O(6)	2·817 (6)	O(2)···N(1)···O(3)	105·8 (2)
···O(6)	2·838 (6)	O(2)···N(1)···O(6)	113·0 (2)
···O(2)	2·973 (6)	O(2)···N(1)···O(6)	132·2 (2)
···O(3)	3·089 (6)	O(3)···N(1)···O(3)	138·6 (2)
		O(3)···N(1)···O(6)	133·8 (2)
N(2)···O(6)	2·714 (5)	O(3)···N(1)···O(6)	80·2 (2)
···O(7)	2·877 (6)	O(3)···N(1)···O(6)	86·6 (2)
···O(9)	2·883 (6)	O(3)···N(1)···O(6)	95·6 (2)
···*W*(2)	3·022 (7)	O(6)···N(1)···O(6)	110·3 (2)
···O(7)	3·092 (6)		
		W(2)···N(2)···O(6)	107·3 (2)
		W(2)···N(2)···O(7)	95·3 (2)
		W(2)···N(2)···O(7)	73·8 (2)
		W(2)···N(2)···O(9)	127·1 (2)
		O(6)···N(2)···O(7)	103·2 (2)
		O(6)···N(2)···O(7)	145·2 (2)
		O(6)···N(2)···O(9)	116·2 (2)
		O(7)···N(2)···O(7)	111·5 (2)
		O(7)···N(2)···O(9)	102·2 (2)
		O(7)···N(2)···O(9)	53·3 (1)

and equatorial angles (Table 3) compare well with the values found in α-TeO_2 (Lindqvist, 1968) and in β-TeO_2 (Beyer, 1967) in which they are 101–102° (eq) and 168–169° (ax).

Another comparable structure with fourfold Te^{IV} coordination is $Te_2O_4.HNO_3$ (Swink & Carpenter, 1966) in which Te_2O_4 groups are connected *via* double oxygen bridges to form layers. The angle around the O atom with strong bonds to both Te atoms within the group is 126·2 (8)° (2×) in $Te_2O_4.HNO_3$ which is to be compared with 118·1 (2)° for Te(1)–O(7)–Te(2) in $(NH_4)_2Te_2O_5.2H_2O$. The corresponding angle in $Te_2O_3SO_4$ (Johansson & Lindqvist, 1976) is 134·9 (7)°. In $Te_2O_3SO_4$, Te_2O_5 units are also connected to form chains, but the oxygen coordination around tellurium is different.

The hydrogen bonds within the structure are probably as indicated in Table 4 in which relevant O–O and N–O distances are given. A neutron diffraction study of $(NH_4)_2Te_2O_5.2H_2O$ is in progress to elucidate the detailed structure of the water molecules and the ammonium ions.

The author wishes to thank Professor Oliver Lindqvist and Dr Jacques Moret for valuable discussions and Dr Susan Jagner for revising the English text. Financial support has been obtained from the Swedish Natural Science Research Council (NFR, Contract No. 3396).

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