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Crystal structure of $Te(OH)_6 \cdot 2KNO_3 \cdot 2H_2O$: n addition compound of telluric acid

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Abstract. $Te(OH)_6 \cdot 2KNO_3 \cdot 2H_2O$ is monoclinic, $P2_1/a$, with $a = 13.34(1)$, $b = 7.160(5)$, $c = 6.939(5)$ Å, $\beta = 100.14(1)°$, $V = 652.4$ Å^3, $Z = 2$ and $d_x = 2.381$ $g \times cm^{-3}$. The structure has been solved with the heavy atom method, using 2296 independent data with a final R value of 0.023 for 1858 reflections. The main characteristic of this compound is the coexistence of two different and independent anionic groups $(TeO_6)^{6-}$ and $(NO_3)^{1-}$.

Introduction

$Te(OH)_6 \cdot 2KNO_2 \cdot 2H_2O$ has been prepared for the first time by Gutbier et al. (1904). Later, von Rosenhein et al. also mention the existence of this compound (1912).

The study of this salt takes place in a general investigation on addition compounds of telluric acid with various salts. Several compounds of this type have already been studied:

- Telluric acid and phosphates
  - monophosphates (Averbuch-Pouchot et al., 1981)
  - diphosphate (Averbuch-Pouchot et al., 1983)
  - trimetaphosphates (Boudjada et al., 1983)
  - tetrametaphosphate (Durif et al., 1982)
- Telluric acid and sulfates (Zilber et al., 1982)
- Telluric acid and iodates (Averbuch-Pouchot, 1983)

Chemical preparation

Crystals of the new compound have been prepared, at room temperature, by slow evaporation of a water solution containing telluric acid and potassium

Table 1. Parameters used for the X-ray data collection

Apparatus:	Philips PW 1100
Wavelength:	MoKα (0.7107 Å)
Monochromator:	graphite plate
Scan mode:	Ω
Theta range:	3 – 35°
Crystal size:	$0.22 \times 0.24 \times 0.24\,\mathrm{mm}^3$
μ:	$30.65\ \mathrm{cm}^{-1}$
Scan width:	1.1°
Scan speed:	$0.02°\ \mathrm{s}^{-1}$
Number of collected reflexions:	2296 ($\pm h, k, l$)
Total background measurement time:	20 s
Reference reflexions:	$4\bar{4}0$ and $\bar{4}40$ (every two hours)

Table 2. Final atomic coordinates. Temperature factors B_{eq} are given for nonhydrogen atoms and B_{iso} for hydrogen atoms.

$B_{eq} = 4/3 \sum_i \sum_j \vec{a}_i \cdot \vec{a}_j \cdot \beta_{ij}$

Atoms	$x(\sigma)$	$y(\sigma)$	$z(\sigma)$	$B_{eq}(\sigma)$
Te	0	0	0	1.054(3)
K	0.15917(5)	−0.0194(1)	0.49110(9)	1.81(1)
O(1)	0.4418(2)	0.2844(3)	0.8585(3)	1.68(4)
O(2)	0.4740(2)	0.4003(3)	0.2417(3)	1.65(4)
O(3)	0.1315(2)	0.1151(3)	0.0551(4)	1.81(5)
O(4)	0.3380(2)	0.1207(4)	0.4031(5)	3.31(6)
O(5)	0.1508(3)	0.3260(4)	0.6421(5)	3.83(7)
O(6)	0.0442(2)	0.5143(4)	0.7445(4)	2.72(5)
O(W)	0.2827(2)	−0.0080(5)	0.8644(4)	2.49(5)
N	0.3806(2)	−0.0139(4)	0.3419(4)	2.01(5)
				$B_{iso}(\sigma)$
H(1)	0.991(4)	0.302(7)	0.806(7)	3.0(11)
H(2)	0.473(3)	0.294(7)	0.240(7)	2.4(9)
H(3)	0.189(5)	0.075(9)	0.983(9)	5.5(16)
H(1W)	0.188(4)	0.395(8)	0.133(8)	4.3(13)
H(2W)	0.331(4)	0.094(9)	0.869(8)	5.1(15)

nitrate in the ratio of one mole of the acid for two of the nitrate. Crystals are stout multi-faceted monoclinic prisms.

Crystal chemistry

A preliminary study by the Weissenberg method shows the compound to be monoclinic, $P2_1/a$ and gives approximative values for the cell parameters. A refinement of these values using powder angular data obtained with a Philips Norelco diffractometer leads to the following cell constants:

$a = 13.33(1)$ Å
$b = 7.155(8)$ $\beta = 100.14(1)°$
$c = 6.925(8)$

These values slightly differ from those calculated from single crystal angular data. These last values, given in the abstract, are used in the crystal structure determination and for the calculation of interatomic distances.

Crystal structure determination

The experimental conditions used during the intensity data collection are reported in Table 1. Given the value of $\mu R(\sim 0.34)$, no correction for absorption was applied. Classical methods were used for the structure determination: three-dimensional Patterson and successive Fourier syntheses. A final difference Fourier synthesis reveals the positions of all the hydrogen atoms.

After the last refinement cycles using anisotropic thermal parameters for the non-hydrogen atoms, the final R value converges to 0.023 for a set of 1858 reflections satisfying the criteria:

$|F_o| > 3\sigma(F_o)$ (438 reflections rejected)
$|F_o - F_c| > 66$ in a scale varying from 0 to 1284 (19 reflections rejected).

For the complete set of 2296 reflections the R value is 0.034.

All along the structure determination, a unitary weighting scheme has been applied.

Table 2 reports the atomic coordinates and Table 3 the thermal parameters isotropic for the hydrogen atoms and anisotropic for all other one[1].

Description of the structure

The Figure 1 shows a projection of the atomic arrangement along the c axis.

The main feature of this structure is the coexistence of two different anionic groups TeO_6^{6-} and NO_3^-.

As usual, the Te(VI) atoms are surrounded by a quite regular oxygen octahedron (Table 5). These TeO_6 octahedra form a C-centered network.

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[1] Additional material to this paper can be ordered referring to the no. CSD 50705, name(s) of the author(s) and citation of the paper at the Fachinformationszentrum Energie Physik Mathematik, D-7514 Eggenstein-Leopoldshafen 2, FRG

Table 3. Main interatomic distances and bond angles in the $Te(OH)_6$, NO_3 and KO_9 polyhedra

TeO_6 octahedron

Te – O(1)	1.919(2) (× 2) Å	O(1) – Te – O(2)	91.66(10)°
Te – O(2)	1.910(2) (× 2)	O(1) – Te – O(3)	88.27(11)
Te – O(3)	1.915(2) (× 2)	O(2) – Te – O(3)	91.98(11)

NO_3 group

N – O(4)	1.231(4) Å	O(4) – N – O(5)	120.8(3)°	O(4) – O(5)	2.142(4) Å
N – O(5)	1.232(4)	O(4) – N – O(6)	119.2(3)	O(4) – O(6)	2.159(4)
N – O(6)	1.272(4)	O(5) – N – O(6)	119.9(3)	O(5) – O(6)	2.168(4)

Least squares planes $Ax + By + Cz - D = 0$		Atomic deviations from this plane:	
$A = -0.5223$	$C = -0.8500$	N 0.051 Å	O(5) –0.068
$B = -0.0691$	$D = -4.4630$	O(4) –0.036	O(6) –0.053

KO_9 polyhedron

K – O(1)	2.919(2)	K – O(3)	3.134(3)	K – O(5)	2.696(3)
K – O(2)	2.843(3)	K – O(4)	2.755(3)	K – O(5)	3.055(4)
K – O(2)	2.885(2)	K – O(4)	2.678(3)	K – O(W)	2.816(2)

Table 4. Main interatomic distances and bond angles in the hydrogen bond scheme

Water molecule

	O(W) – H	H...O	O(W) – O	O(W) – H..O	H – O(W) – H
O(W) – H(1W)...O(3)	0.80(6)	2.17(7)	2.745(4)	163(6)	110(5)
O(W) – H(2W)...O(1)	0.97(7)	2.03(7)	2.987(4)	172(6)	

$Te(OH)_6$ group

	O – H	H...O	Te – O – H	O – H...O	O – O
Te – O(1) – H(1)...O(6)	1.02(5)	1.76(5)	117(4)	158(4)	2.729(4)
Te – O(2) – H(2)...O(6)	0.77(5)	2.02(5)	111(5)	172(5)	2.778(4)
Te – O(3) – H(3)...O(W)	1.02(6)	1.73(6)	121(6)	176(6)	2.745(4)

Each octahedron is located in a channel, parallel to the *c* axis and delimited by six NO_3 groups alternatively in $z = 1/3$ and $2/3$. The NO_3 groups have an almost perfect ternary symmetry. The sum of the three ONO angles is 359.9, very near to 360°. The atomic deviations from the NO_3 least-squares plane confirms that this NO_3 group is quasi planar. It is also to be noticed that the longest NO distance in this group corresponds to the oxygen atom

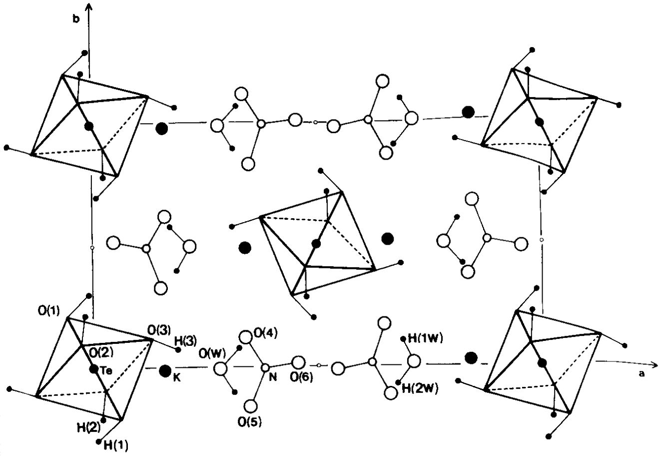

Fig. 1. Projection of the atomic arrangement of $Te(OH)_6 \cdot 2KNO_3 \cdot 2H_2O$ along the $\vec{c}$ axis

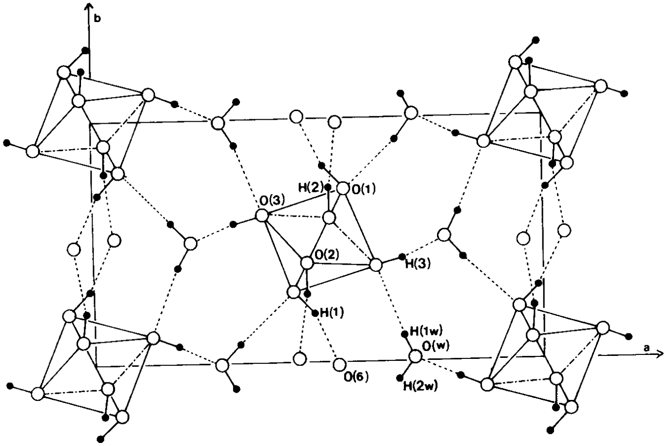

Fig. 2. Hydrogen bond scheme in projection along the $\vec{c}$ axis. Oxygen atoms not involved in hydrogen bridges are omitted

O(6) implicated twice as a donor in the hydrogen bonds. This oxygen atom is also the only one to be not involved in the potassium coordination polyhedron.

Edge sharing KO_9 polyhedra give rise to a two-dimensional network, parallel to the (a, b) plane in $z \simeq 0.5$.

Figure 2 represents the hydrogen bridge scheme. Two hydrogen atoms of the $Te(OH)_6$ group are related to water molecules and the four other ones to NO_3 groups.

The H atoms of the water molecules are only bridged to oxygen atoms of the TeO_6 octahedra.

The hydrogen bonds form a two-dimensional network in (a, b) planes.

References

Gutbier, A., Wagenknecht, W.: Über die Einwirkung von Hydroperoxyd und Tellurdioxyd: Ein neues Verfahren zur Darstellung der Tellursäure. Z. Anorg. Chem. **40**, 260–263 (1904)

Von Rosenhein, A., Jander, G.: Über die Tellursäure und ihre Alkalisalze in ihrem Verhalten als Halbkolloide, Kolloid Z. **22**, 23–42 (1912)

Zilber, R., Durif, A., Averbuch-Pouchot, M. T.: Structure of thallium sulfate tellurate: $Te(OH)_6 \cdot Tl_2SO_4$. Acta Crystallogr. **B38**, 1554–1556 (1982)

Averbuch-Pouchot, M. T., Durif, A.: Crystal data for two new phosphate tellurates: $Te(OH)_6 \cdot 2TlH_2PO_4 \cdot Tl_2HPO_4$ and $Te(OH)_6 \cdot 2TlH_2PO_4$. Crystal structure of $Te(OH)_6 \cdot 2TlH_2PO_4$. Mater. Res. Bull. **16**, 71–76 (1981)

Boudjada, N., Boudjada, A., Guitel, J. C.: Structure du trimétaphosphate tellurate d'ammonium, $2(NH_4)_3P_3O_9 \cdot Te(OH)_6$. Acta Crystallogr. C**39**, 656–658 (1983)

Averbuch-Pouchot, M. T., Durif, A.: Structure of a potassium diphosphate tellurate hydrate, $K_3HP_2O_7 \cdot Te(OH)_6 \cdot H_2O$. Acta Crystallogr. C**39**, 27–28 (1983)

Durif, A., Averbuch-Pouchot, M. T., Guitel, J. C.: $(NH_4)_4P_4O_{12} \cdot {}_2Te(OH)_6 \cdot 2H_2O$, the first example of a tetrametaphosphate-tellurate. J. Solid State Chem. **41**, 153–159 (1982)

Averbuch-Pouchot, M. T.: Crystal chemistry of some addition compounds of alkali iodates with telluric acid. J. Solid State Chem. **49**, 368–378 (1983)