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Crystal structure of a new telluric acid adduct: $Te(OH)_6 \cdot 2\ CsCl$

M. T. Averbuch-Pouchot

Laboratoire de Cristallographie, Centre National de la Recherche Scientifique,
Laboratoire associé à l'U.S.T.M.G., 166 X, 38042 Grenoble Cédex, France

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Abstract. Crystals of the title compound were prepared by slow evaporation of a water solution of the two components. The space group is $P2_1/c$ with the lattice dimensions: $a = 6.243(1)$, $b = 11.154(3)$, $c = 7.862$ Å, $\beta = 107.48(2)°$ and $Z = 2$.

The crystal structure was solved by direct methods, using single crystal diffractometer data and refined by a least squares method to a final R value 0.027 for 2083 independent reflexions ($\lambda AgK\alpha$). H atom positions located by difference-Fourier syntheses have been refined. The Te – O and Cl – O mean distances are respectively 1.912 and 3.146 Å. The hydrogen bond scheme connects the $Te(OH)_6$ groups and Cl atoms in a three-dimensional way.

Introduction

$Te(OH)_6 \cdot 2\ CsCl$ was prepared and examined as a part of a systematic investigation of adducts between telluric acid and various inorganic salts like sulfates (Zilber et al., 1982), nitrates (Averbuch-Pouchot, 1984), alkali-iodates (Averbuch-Pouchot, 1983), and phosphates (Averbuch-Pouchot et al., 1984). The present work is to be compared with previous studies of adducts between telluric acid and alkali-halides: $Te(OH)_6 \cdot NaF$ (Allman 1976), $Te(OH)_6 \cdot 2KF$ (Allman and Haase, 1976) and $Te(OH)_6 \cdot 2CsF \cdot 2H_2O$ (Allman and Rius, 1978). Like in these last salts all the OH radicals of the $Te(OH)_6$ groups are involved in O – H ... X (here Cl) hydrogen bonds.

Preparation and crystal data

Single crystals are prepared by slow evaporation, at room temperature, of a water solution of telluric acid and caesium chloride in the stoichiometric

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

Crystal size	$0.20 \times 0.20 \times 0.18$ mm^3	Total background measurement time	from 16 to 27 s
Apparatus	Enraf-Nonius CAD4	Number of independent collected reflexions	3071 ($\pm h,k,l$)
Wavelength	Ag$K\alpha$ (0.5608 Å)	Intensity reference reflexions	$1\bar{8}\bar{4}$ and $\bar{1}\bar{1}\bar{5}$
Monochromator	graphite plate	Orientation reference reflexions	$\bar{1}\bar{7}\bar{3}$ and $1\bar{5}\bar{5}$
Scan mode	Ω	μ	5.355 mm^{-1}
Theta range	$3-30°$	$F(000)$	500
Scan width	1.3°		
Scan speed	from 0.024 to 0.042° s^{-1}		

ratio. Crystals appear like stout monoclinic prisms up to 1 cm of length. A preliminary study of a single crystal by a film technique shows the space group to be $P2_1/c$ ($h0l$, $l = 2n$ and $0k0$, $k = 2n$) and gives approximative values for the unit-cell dimensions. These constants have been refined by a least-squares method using 22 angular data ($10.5 < \theta < 14.25°$) measured with a four-circle diffractometer during the intensity data collection and also by using angular data from a powder diagram (Table has been deposited[1]. The two sets of values so obtained for the unit-cell dimensions are the following ones:

Automatic diffractometer	**Powder diagram**
$a = 6.243(1)$ Å	$a = 6.247(5)$ Å
$b = 11.154(3)$	$b = 11.16(1)$
$c = 7.862(2)$	$c = 7.867(5)$
$\beta = 107.48(2)°$	$\beta = 107.495(20)°$
$V = 522.2(4)$ Å^3	$V = 523.1(1.3)$ Å^3
$D_x = 3.601$ g $\times$ cm^{-3}	$D_x = 3.595$ g $\times$ cm^{-3}

The cell values used for the structure determination and for interatomic distances and bond angles calculations are those obtained with the automatic four-circle diffractometer.

Crystal structure determination

Table 1 reports the experimental conditions used during the intensity data collection. The intensities have been corrected for Lorentz and polarization effects, but due to the wavelength used and the crystal size, no absorption correction has been applied. The crystal structure has been solved by direct methods (Main et al., 1977) and successive Fourier syntheses. Hydrogen atoms have been located by difference-Fourier synthesis. After final refinement cycles, using anisotropic thermal parameters for nonhydrogen atoms and isotropic for H atoms the R value is 0.027 for a set of 2083 reflexions such that $I > 9\sigma_I$. For the complete set of 3071 independent data this value is 0.043. The extinction coefficient was refined according to Stout and Jensen (1968) to a value $g = 1.17 \cdot 10^{-6}$. All the calculations have been done using the SDP System (1977), the computer used was a MicroVax II. Atomic scattering factors for neutral atoms were taken from International Tables for X-ray Crystallography, Table 2−2B (Cromer and Waber, 1974) and anomalous dispersion has been taken into account.

Full-matrix refinements on F have been run using a unitary weighting scheme. Table 2 reports the final atomic coordinates for $Te(OH)_6 \cdot 2\,CsCl$. The lists of hkl, F_{obs}, F_{cal} and of the anisotropic thermal parameters have been deposited[1].

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[1] Additional material to this paper can be ordered referring to the no. CSD 52842, name of the author and citation of the paper at the Fachinformationszentrum Energie, Physik, Mathematik GmbH, D-7514 Eggenstein-Leopoldshafen 2, FRG.

Table 2. Final atomic coordinates. Temperature factors $B_{eq.}$ (Å^2) are given for non hydrogen atoms and B_{iso} (Å^2) for hydrogen atoms. $B_{eq.} = 4/3\ \Sigma_i \Sigma_j a_i \cdot a_j \cdot \beta_{ij}$.

Atoms	$x(\sigma)$	$y(\sigma)$	$z(\sigma)$	$B_{eq.}(\sigma)$
Cs	0.75774(5)	0.12969(3)	0.44006(4)	2.227(4)
Te	0	0	0	1.308(4)
Cl	0.3780(2)	0.3300(1)	0.1833(1)	2.25(2)
O1	0.7342(5)	0.4344(3)	0.5367(4)	2.29(5)
O2	0.0155(5)	0.1394(3)	0.8638(4)	2.05(5)
O3	0.1671(6)	0.4263(3)	0.7198(4)	2.49(6)
				$B_{iso}(\sigma)$
H1	0.68(1)	0.077(7)	0.949(9)	2(2)
H2	0.13(1)	0.149(7)	0.84(1)	3(2)
H3	0.20(1)	0.355(6)	0.693(8)	1(1)

Structure description

Figure 1 shows a projection of the atomic arrangement along the c axis, while Table 3 reports the main interatomic distances and bond angles and Table 4 the hydrogen bond scheme.

Te(VI) atoms have their usual surrounding made of an almost regular oxygen octahedron, the Te−O distances ranging from 1.907 to 1.915 Å and the O−Te−O angles between 87.1 and 8.0°. Tellurium atoms are located on inversion centers, so the TeO_6 groups are centrosymmetric and located inside the atomic arrangement as to form an A centered network.

All the hydrogen atoms of the $Te(OH)_6$ groups are involved in hydrogen bonds of O−H...Cl type (Table 4), connecting such a group to six different chlorine atoms with a mean Cl−O distance of 3.146 Å, while each Cl atom

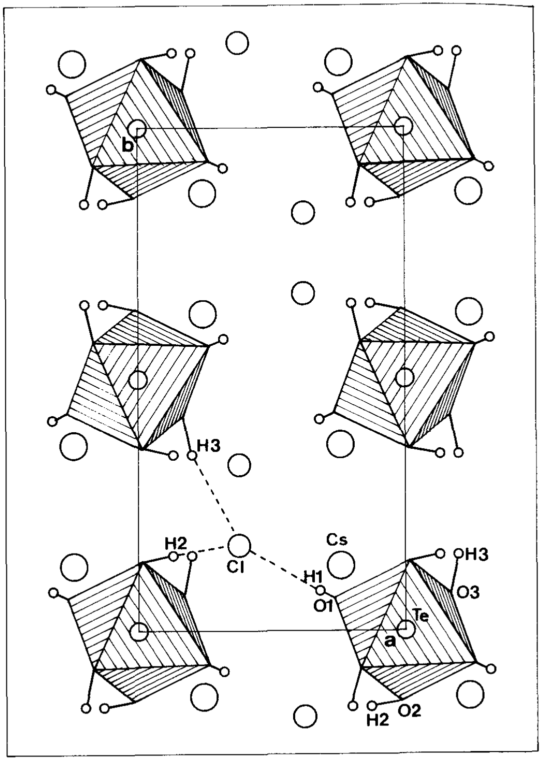

Fig. 1. Projection of the atomic arrangement of $Te(OH)_6 \cdot 2\,CsCl$ along the *c* axis.

Table 3. Main interatomic distances (Å) and bond angles (°) for the TeO_6 octahedron and for the $Cs(O_5Cl_2)$ polyhedron.

The $Te(OH)_6$ group		**$Cs(O_5Cl_2)$polyhedron**			
Te–O1	1.913(3) (×2)	Cs–Cl	3.437(1)	Cs–O2	3.237(3)
Te–O2	1.907(3) (×2)	Cs–Cl	3.494(1)	Cs–O2	3.187(3)
Te–O3	1.915(3) (×2)	Cs–O1	3.495(3)	Cs–O3	3.430(3)
O1–Te–O2	88.0(1)	Cs–O1	3.210(3)		
O1–Te–O3	87.8(1)				
O2–Te–O3	87.1(1)				

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

O–H···Cl	O–H	H···Cl	O–Cl	O–H···Cl	Te–O–H
O1–H1···Cl	0.69(7)	2.56(7)	3.211(3)	158(9)	99(7)
O2–H2···Cl	0.79(9)	2.26(9)	3.030(4)	165(7)	118(6)
O3–H3···Cl	0.86(7)	2.35(7)	3.196(4)	165(4)	106(4)

is connected by these H bonds to three oxygen atoms belonging to three different $Te(OH)_6$ groups. This network of O–H...Cl hydrogen bonds is a three-dimensional one. It is to be noticed that one does not observe direct hydrogen bonds between $Te(OH)_6$ groups.

Within a range of 3.5 Å the caesium atoms have a sevenfold coordination made by two chlorine atoms [$\bar{d}(Cl-Cs = 3.466$ Å)] and five oxygen atoms [$\bar{d}(Cs-O = 3.312$ Å)] belonging to two different $Te(OH)_6$ groups.

The intensity data collection has been performed within the "groupement grenoblois de diffractométrie".

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