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PRELIMINARY NOTE

Synthesis and Structure of Dirubidium Oxotetrafluorotellurate (IV) Rb_2TeOF_4

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SUMMARY

Dirubidium oxotetrafluorotellurate (IV) Rb_2TeOF_4 is described for the first time. This compound is isostructural with the two known oxotetrafluorotellurates M_2TeOF_4 (M = K, Cs). It crystallizes in the orthorhombic system with lattice constants a = 6,45 Å ; b = 14,18 Å ; c = 6,715 Å.

In a recent study [1] it has been clearly proved that vitrification of alkaline pentafluorotellurates (IV) $MTeF_5$ (M = K, Rb, Cs) is strictly governed by their partial reaction with oxygen.

The glassy material thus obtained contains an oxyfluoride M_2TeOF_4. The relative amount $MTeF_5/M_2TeOF_4$ depends on the conditions of the heat treatment and on the oxygen content of the gas surrounding the sample. To our knowledge the corresponding compound for M = Rb has not been described so far.

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Synthesis of Rb_2TeOF_4 has been performed to allow a conclusive identification of the phases which appear upon crystallization of the glass.

The reaction described by Milne and Moffett [2] for K and Cs compounds was used :

$$RbTeF_5 + 3\ RbF + TeO_2 \longrightarrow 2\ Rb_2TeOF_4$$

RbF and TeO_2 are commercial reagents (Merck pur $\geqslant$ 99,8 %). $RbTeF_5$ is obtained as described elsewhere [3],[4].

Manipulations, heat treatments and sampling were carried out in a glove-box under an atmosphere of dry and oxygen-free argon.

A mixture of powdered $RbTeF_5$, RbF and TeO_2 with the above stoichiometric proportions was heated at 500°C for 1 hour. After cooling and grinding another treatment at 500°C was performed for 3 hours. Under these conditions Rb_2TeOF_4 was obtained quantitatively as a white solid.

The crystallographic study was performed with a Philips assembly (horizontal goniometer and 360 mm camera, copper source 1,5418 Å). X-ray powder data are given in Table I.

TABLE I

X-ray powder diffraction data for Rb_2TeOF_4

I/Io	dobs.	dcalc.	h k l	I/Io	dobs.	dcalc.	h k l
10	4.86	4.875	0 2 1	10	2.227	2.229	0 6 1
5	4.41	4.420	1 1 1	20	1.947	1.945	2 4 2
10	3.78	3.810	1 3 0	10	1.906	1.906	2 6 0
5	3.53	3.545	0 4 0	20	1.880	1.879	3 3 1
20	3.37	3.360	0 0 2	5	1.836	1.834	2 6 1
100	3.31	3.315	1 3 1	5	1.792	1.796	3 1 2
25	3.21	3.225	2 0 0	5	1.714	1.714	0 8 1
5	2.528	2.520	1 3 2	5	1.695	1.695	1 5 3
15	2.362	2.363	0 6 0	10	1.664	1.660	3 5 1
30	2.335	2.326	2 0 2	5	1.6130	1.6125	4 0 0

The intensities were estimated visually. The observed reflexions were indexed using an a priori procedure. The cell dimensions were determined by a least-square analysis. Rb_2TeOF_4 is orthorhombic with a = 6.45 Å, b = 14.18 Å, c = 6.715 Å, Dx = 4.22 g/cm^3, Z = 4.

This compound is isostructural with the two known M_2TeOF_4 (M = K, Cs) [2] having the M_2SbF_5 structure (M = K, Rb, Cs, Tl [5] or NH_4 [5],[6]). Cell dimensions are given for comparison in Table II.

TABLE II

Orthorhombic cell dimensions (Å) for M_2TeOF_4 and M_2SbF_5 compounds

M_2TeOF_4				M_2SbF_5			
M	a	b	c	M	a	b	c
K (a)	6.24	13.83	6.46	K (c)	6.244	13.67	6.491
				Rb (c)	6.399	13.88	6.745
Rb (b)	6.45	14.18	6.715	Tl(c)	6.47	14.05	6.710
				NH_4 (c)	6.489	14.09	6.710
				NH_4 (d)	6.497	14.162	6.772
Cs (a)	6.65	14.53	7.04	Cs (c)	6.652	14.45	7.099

(a) : ref. 2 - (b) : this work ; (c) : ref. 5 ; (d) : ref. 6

Moreover the indexing of the observed X-ray reflexions for Rb_2TeOF_4 (Table I) is compatible with the same space group Cmcm (63) as for M_2SbF_5 compounds [5],[6].

The structure of these compounds contains isolated SbF_5E or $TeOF_4E$ anions whose geometry can be described by a slightly distorted octahedra in which the lone pair of electrons E located on an apical position plays a stereochemically active role.

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