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Neutron Structural Refinement of Cubic Orthotelluric Acid*

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Abstract

The cubic form of orthotelluric acid ('H_6TeO_6') has been analyzed by neutron diffraction, which required the assignment of the space group to be $F4_132$, in contrast to the previous X-ray diffraction conclusion that the space group is $Fd3c$. The cell constant is $a_0 =$ 15·699 (2) Å which, given the measured density of 3·120 (2) Mg m^{-3}, requires the unit cell to contain 32 molecules. The molecular species present in the lattice are $Te(OH)_6$ octahedra linked into chains by hydrogen bonds in which the hydrogens are statistically disordered into two equivalent positions. Two independent Te—O distances were found, whose mean value is 1·90 Å. The O—H···O bond distances range from 2·56 (1) to 3·02 (1) Å, which are representative of strong and weak hydrogen bonds. The mean O—H and O···H bond distances are 0·94 and 1·83 Å. The final cycle of the refinement gave $R(F)$ and $R_w(F)$ indices of 0·108 and 0·093 for 255 unique reflections above background. Results of X-ray diffraction, dehydration and infrared measurements are also described.

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Introduction

The preparation and crystallographic identification of orthotelluric acid ('H_6TeO_6') have been available for some time. Optimal preparatory conditions can be found in the treatise by Mellor (1931) and in the work of Bayer (1968). Also, it is well established that this substance crystallizes in the cubic and monoclinic systems. Gossner & Kraus (1934) were the first to assign to the latter modification space group No. 14 (*International Tables for X-ray Crystallography*, 1952), and recently accurate investigations by single-crystal X-ray (Lindqvist, 1970) and neutron diffraction methods (Lindqvist & Lehmann, 1973) have shown that monoclinic orthotelluric acid indeed crystallizes in the space group $P2_1/n$. Further structural studies of the monoclinic form are, therefore, unnecessary.

Much work and controversy have been associated with the cubic form of orthotelluric acid. The first single-crystal study was that reported by Kirkpatrick & Pauling (1926), who concluded that the face-centered cubic lattice was compatible with the space groups T_d^2, T_d^5, O^3, O^4, O_h^5, O_h^6, O_h^7 and O_h^8. This led to a disagreement between them and Gossner & Kraus (1934) which appeared to be settled by Pauling (1935). More recently, X-ray work was contributed by Bayer (1968) and by Falck & Lindqvist (1978), who sided with Pauling. Cohen-Addad (1971) presented neutron powder diffraction data which were analyzed using a

model proposed by Mahalaxmi (1968) in which the correct space group was assumed to be *Fd*3*c*. Below, we present compelling evidence from single-crystal neutron diffraction data that the correct space group is not *Fd*3*c*, but rather the non-centrosymmetric subgroup, $F4_132$.

Experimental

Clear octahedral crystals of cubic telluric acid were prepared by dissolving commercial H_6TeO_6 (ACS grade) in hot 10·65 *M* HNO_3 and allowing the solution to evaporate slowly at room temperature. One of these octahedra was chosen for single-crystal X-ray diffraction analysis. For neutron diffraction analysis, the clear octahedral crystals were dissolved in deionized water which was redistilled from potassium permanganate and sulfuric acid in an all-glass distillation apparatus. Extremely slow evaporation yielded large crystals which upon standing turned cloudy white, but still showed single-crystal diffraction when checked by X-ray methods.

A Siemens cylindrical powder camera (114·8 mm) using Ni-filtered Cu $K\alpha$ radiation was employed to determine the unit-cell parameter. 2θ values up to 160° were obtained, with the $K\alpha$ doublet resolved at high angles ($K\alpha_1$ = 1·54051; $K\alpha_2$ = 1·54433 Å). The measured values were placed into a least-squares program (Roof, 1968) utilizing the Nelson–Riley extrapolation function to compute the lattice constant and its standard deviation, 15·699 (2) Å. This result is consistent with the values obtained by Bayer (1968) and Lindqvist (1978), 15·695 (5) and 15·705 (3) Å, respectively. Observed compared to calculated *d*-spacing values were found to be <0·05% in error in the high-angle back-reflection region. Diiodomethane and 1,1,2,2-tetrabromoethane were the constituents used in the suspension method of density determination [ρ_{exp} = 3·120 (2) Mg m^{-3}]. A Perkin–Elmer TGS-1 thermobalance (operated at 2·5 K min^{-1}) was used to ascertain the thermal degradation of telluric acid. The complex decomposition proceeds in five basic steps according to

$$H_6TeO_6 \xrightarrow[-2H_2O]{(1)} (H_2TeO_4)_n \xrightarrow[-0{\cdot}5H_2O]{(2)}$$

$$H_2Te_2O_7 \xrightarrow[-nH_2O,\, nO_2]{(3)}$$

$$[\text{Intermediates} + TeO_3] \xrightarrow[-nO_2]{(4)} Te_2O_5 \xrightarrow[-nO_2]{(5)} TeO_2.$$

A Perkin–Elmer 521 grating IR spectrophotometer was employed to explore the stretching and deformation properties of the hydrogen bonding present in the cubic form of $Te(OH)_6$.

For the X-ray measurements, a single crystal of $Te(OH)_6$ (cubic) was selected and mounted on an Enraf–Nonius CAD-4 diffractometer, and 15 reflections were automatically centered to obtain an orientation matrix for data collection. With Mo $K\bar{\alpha}$ (λ = 0·71073 Å), measured intensities were collected by the θ–2θ scan technique at a rate of 0·4–5·0° min^{-1} in the range of $3 < 2\theta < 60°$. Monitoring standards were checked every 2 h of exposure time to verify crystal stability and hardware reliability (*ca* <1·2% deviation). Reflections used in the structural refinement obeyed the condition $I_{net} > 3\sigma(I)$ with $I_{net} = (P - B)$ and $\sigma(I) = (P + B + p^2 I_{net}^2)^{1/2}$, where P is the peak intensity, B is the sum of the time-scaled background counts on both sides of the peak and p is the ignorance factor (0·02). After correcting the resultant intensities for Lorentz and polarization effects, an analytical correction to minimize error due to absorption was applied and 82 unique reflections were obtained after averaging redundant data. The standard deviations of the averaged data were calculated from $\sigma(F) = N^{-1} \sum 1{\cdot}02\sigma_{F_i}$ (Peterson & Levy, 1957), where N = number of redundant reflections and σ_{F_i} = standard deviation for each individual reflection.

For neutron diffraction, a large octahedral crystal (edges 1 mm) mounted on a glass fiber was placed on a computer-controlled four-circle diffractometer at the Brookhaven National Laboratory High-Flux Beam Reactor. The working wavelength, obtained by means of a Be(002) crystal monochromator, was 1·0503 Å. Three-dimensional intensities were collected at ambient room temperature using a θ–2θ step-scan method. Fixed scan widths of 2·4, 3·4 and 4·2° were used in respective data-collection ranges of 4–60°, 60–85° and 85–100° 2θ. 29 reflections were used to obtain the orientation matrix. After carefully establishing the Laue group to be *m*3*m* and verifying an *F*-centered lattice, three redundant zones of data were collected. Two reflections were monitored every 40 data points, and these intensity standards revealed random variations only, *i.e.* <1·6% deviation from mean values. The data were corrected for Lorentz effects and an analytical absorption factor was applied [μ(neutron) = 0·2007 mm^{-1}]. A resultant total of 255 unique reflections having $I_{net} > 3\sigma(I)$ were saved for the least-squares refinement.

Crystal data

$Te(OH)_6$(cubic), M_r = 229·644, a_0 = 15·699 (2) Å, V = 3869·15 Å^3, ρ_{exp} = 3·120 (2), ρ_{calc} = 3·153 Mg m^{-3}, Z = 32, μ(X-ray, Mo $K\bar{\alpha}$) = 6·485 mm^{-1}, μ(neutron) = 0·2007 mm^{-1}.

Structure determination

The starting model used in the X-ray diffraction study was refined in space group *Fd*3*c*, as first proposed by

Kirkpatrick & Pauling (1926). The Te and O atoms were placed at (0,0,0) and (0·14,0·54,0·28); the respective positional number sets with Wyckoff notation are 32(*c*) and 192(*h*). The model was refined using a full-matrix least-squares program written by Larson (1967) and, after several cycles varying the anisotropic thermal parameter of the O atom and applying a secondary-extinction correction, it yielded final residual index values $R_1 = \sum \Delta F / \sum |F_o| = 0{\cdot}037$ and $R_2 = [\sum w(\Delta F)^2 / \sum w(F_o)^2]^{1/2} = 0{\cdot}044$ where $\Delta F = ||F_o| - |F_c||$ and $w = \sigma^{-2}(|F_o|)$. The final positional parameters and pertinent bond distances and angles obtained from the X-ray diffraction data are presented in Table 1,* which also lists the X-ray refinement results of Falck & Lindqvist (1978).

The neutron structural refinement of $Te(OH)_6$ (cubic) was initiated using the atomic positions obtained from our X-ray analysis and, again, space group *Fd*3*c* was assumed. The first least-squares attempt rejected 46 out of the 255 observed reflections as being space-group extinct. More than 30 reflections were *hhl* (*l* odd) and the remaining reflections were 0*kl* ($k + l = 2n$). Space groups *Fd*3*c*, $F\bar{4}3c$, *Fm*3*c* and *Fd*3*m* were immediately dismissed from consideration while $F\bar{4}3m$, *F*432, $F4_132$ and *Fm*3*m* were retained. The presence of a 4_1 screw axis unambiguously indicated $F4_132$, and after consulting the literature (Buerger, 1956), a direct relationship was found to exist between $F4_132$ and *Fd*3*c*. Point group $\frac{4}{m}\frac{\bar{3}}{m}\frac{2}{m}$ is one of the isometric point groups containing an operation of the second sort, $F4_132.i(\frac{1}{8},\frac{1}{8},\frac{3}{8}) \rightarrow Fd3c$, a combination of an inversion and a translation. An analysis of the X-ray Patterson map, dominated by the heavy Te atom, produced a trial structure in space group $F4_132$ and thus the phase problem was partially solved. A few isotropic cycles produced an *R* value of 0·25 and a resultant three-dimensional electron density map phased from the partial model revealed the O atom positions. Continued least-squares refinement yielded results leading to a difference Fourier map which revealed disordered H atom positions. The model for the disorder will be discussed in another section. The application of a secondary-extinction correction to the neutron diffraction data, $\zeta = 9\ (1) \times 10^{-5}\ e^{-2}$ (Zachariasen, 1968), in conjunction with anisotropic refinement, except for the

* Lists of structure factors from the X-ray and the neutron diffraction data and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35510 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *X-ray (using space group Fd3c as the model) positional parameters, bond distances* (Å) *and angles* (°)

	This work	Falck & Lindqvist (1978)
Te(*x*,*y*,*z*)	0	0
O *x*	0·1389 (5)	0·1402 (4)
y	0·5413 (5)	0·5425 (4)
z	0·2802 (5)	0·2809 (4)
Te–O	1·917 (4)	1·913 (6)
O···O(inter)	2·663 (15)	2·639 (12)
O···O(inter)	2·737 (15)	2·773 (13)
O–Te–O	90·1 (2)	90·2 (3)
	89·9 (2)	89·2 (3)

Table 2. *Final positional parameters* (×10⁴) *for cubic* $Te(OH)_6$ *from neutron and X-ray methods (using model* $F4_132$*)*

	x	*y*	*z*	U_{eq}* (Å²)	Occupancy
Neutron					
Te	3771 (4)	3771 (4)	3771 (4)	10·4	1
O(1)	4072 (4)	3384 (4)	2607 (4)	12·0	1
O(2)	3503 (5)	4207 (5)	4806 (4)	16·6	1
H(1-1)	4509 (12)	3724 (18)	2343 (13)	16·0	½
H(1-2)	4051 (13)	2775 (12)	2596 (12)	17·9	½
H(2-1)	3575 (15)	4713 (14)	5068 (13)	16·3	½
H(2-2)	3215 (12)	3883 (11)	5224 (12)	15·9	½
X-ray					
Te	3757 (2)	3757 (2)	3757 (2)	10·4	1
O(1)	4043 (15)	3335 (17)	2631 (11)	12·0	1
O(2)	3430 (20)	4177 (18)	4841 (9)	16·6	1

* Equivalent isotropic *U* values (×10²).

Table 3. *Bond distances* (Å) *and angles* (°) *for cubic* $Te(OH)_6$ *from neutron and X-ray methods (using model* $F4_132$*)*

	Neutron	X-ray
Te–O(1)	1·98 (1)	1·94 (2)
Te–O(2)	1·81 (1)	1·89 (2)
O(1)–O	2·57 (1)	2·56 (4)
O(1)–O	2·80 (1)	2·74 (4)
O(2)–O	2·56 (1)	2·63 (4)
O(2)–O	3·02 (1)	2·76 (4)
O(1)–H(1-1)	0·96 (2)	
O(1)–H(1-2)	0·96 (2)	
O(2)–H(2-1)	0·90 (2)	
O(2)–H(2-2)	0·94 (2)	
H(1-1)···O	1·65 (2)	
H(1-2)···O	1·85 (2)	
H(2-1)···O	1·71 (2)	
H(2-2)···O	2·10 (2)	
O(1)–H(1-1)···O	158 (2)	
O(1)–H(1-2)···O	170 (2)	
O(2)–H(2-1)···O	156 (2)	
O(2)–H(2-2)···O	166 (2)	
O(1)–Te–O(2)	88·3 (3)	89 (1)
O(1)–Te–O(2)	93·6 (3)	91 (1)
O(1)–Te–O(2)	175·7 (4)	178 (2)
Te–O(1)–H(1-1)	113 (1)	
Te–O(1)–H(1-2)	108 (1)	
Te–O(2)–H(2-1)	118 (2)	
Te–O(2)–H(2-2)	122 (2)	
H(1-1)–O(1)···H	111 (2)	
H(1-2)–O(1)···H	114 (2)	
H(2-1)–O(2)···H	119 (2)	
H(2-2)–O(2)···H	118 (2)	

H atoms associated with O(2), yielded final reliability factors (R_1 and R_2) of 0·108 and 0·093. The neutron scattering lengths were: b(Te) = 5·60, b(O) = 5·75 and b(H) = −3·72 fm. The quantity minimized was $R' = \sum w[|F_o| - (k\zeta)^{-1}|F_c|]^2$ where k is the scale factor and $w = \sigma^{-2}(|F_o|)$. In the final cycle of refinement no parameter shifted by more than $0{\cdot}01\sigma$. The goodness of fit was 1·91 and is defined as $\sum_2 = [\sum w(|F_o| - |F_c|)^2/(N_o - N_{vp})]^{1/2}$, where N_o is the number of independent observations and N_{vp} is the number of varied parameters in the full-matrix least-squares refinement. The applied correlation matrix showed no unusual relationships between variables. A final difference Fourier map was virtually featureless. The maximum and minimum peaks were 1·5 (3) and −1·3 (3) fm Å^{-3} which can be attributed to randomly fluctuating background. Tables 2 and 3 list the positional parameters and bond distances and angles for Te(OH)$_6$ obtained by neutron diffraction in space group $F4_132$ (O^4, No. 210).*

Discussion

The completion of single-crystal analyses of cubic Te(OH)$_6$ by X-ray and neutron diffraction presented a discrepancy in space-group assignment. The early X-ray data seem to lead to the $Fd3c$ model whereas neutron diffraction analysis rejects this most explicitly and indicates that the data best fit the cubic model $F4_132$. When our X-ray data were placed into model $F4_132$ the refinement converged to an R value of 0·019 as compared to 0·037 for $Fd3c$. According to Hamilton (1964), the non-centrosymmetric $F4_132$ refinement is statistically superior to the centrosymmetric $Fd3c$, as follows: H_0: $Fd3c$ is the correct space group, $\mathscr{R} = R(Fd3c)/R(F4_132) = 1{\cdot}95$ where the numbers of respective variables are 12 and 23 for 82 independent reflections. R'' (b, $n - m$, α) = R'' (9, 59, 0·005) = 1·205. Therefore $\mathscr{R} > R''$ and thus the null hypothesis is rejected, and the alternative hypothesis, H_a: $F4_132$ is the correct space group, is accepted at a 0·5% confidence level (α = 0·005). Hamilton's (1964) example 6 is an exact analogy of the problem at hand. X-ray data positional coordinates and bond distances and angles derived from model $F4_132$ are also listed in Tables 2 and 3.*

Cubic telluric acid has the stacking form of distorted Te(OH)$_6$ octahedra joined by a network of hydrogen bonding. This can be seen quite clearly in Fig. 1, which is a packing diagram of the middle half of the unit cell, showing the layered nature of the molecular arrangement. Each proton involved in the chain-like linkage was determined to have statistical occupancy with a probability of 0·5 in the neutron powder diffraction work by Cohen-Addad (1971). Our investigation is in agreement with these findings, with the difference between the two studies being the starting model used in the structural refinements. The $Fd3c$ model employed by Cohen-Addad (1971) excluded the possible occurrence of reflections hhl where h and l are odd, and $0kl$ where $k + l = 2n$, even though the 064 reflections and conceivably the 331 and 115 reflections may be observed in the experimental neutron diffraction spectrum. Nevertheless, the assumed disorder of the H atom over two equivalent positions was useful information for our work.

In our refinement of the single-crystal neutron diffraction data, two significantly different Te—O lengths were found, 1·98 (1) and 1·81 (1) Å associated, respectively, with O(1) and O(2). These are consistent with the distances obtained from the X-ray data when placed into space group $F4_132$, although the differences were not as pronounced there (see Table 3). The experimental difference is probably due to the electron scattering in the X-ray analysis *vs* the scattering by the nuclei in the neutron analysis. The deformation is further evidenced by the significant deviations of the O—Te—O angles from 90° in the neutron results, with such distortions from ideality perhaps attributable to the role of the O atoms as acceptors in hydrogen bonding. Fig. 2 shows a schematic picture of the skewed octahedron with the experimental results. Despite the elongation of one and shortening of the other Te—O distance from a mean bond length of 1·91 Å (Lindqvist & Lehmann, 1973), the experimental determinations of Te—O(1) and Te—O(2) bond distances (av. 1·90 Å) are quite reasonable when compared to the wide range (1·73–2·04 Å) found in the literature (*Bond Index of the Determination of Inorganic Crystal Structures*, 1969–1977). More specifically, mixed bond lengths within the same octahedron were found by Lindqvist & Lundgren (1966) and Lindqvist (1969) in alkali tellurates, ranges 1·86–2·03 Å and 1·90–2·04 Å respectively. In another study, more directly related to Te(OH)$_6$, Lammers

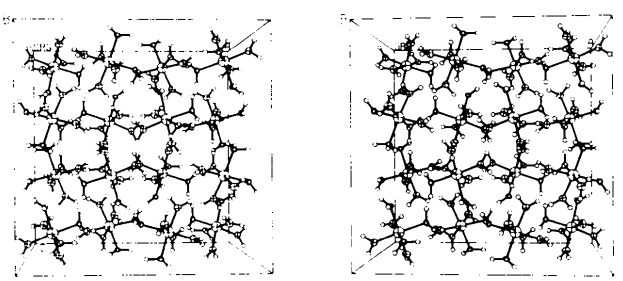

Fig. 1. Stereoscopic packing diagram of the contents of one-half of the unit cell. Both disordered hydrogen positions on each oxygen are shown simultaneously at full weight, although it is understood that only *one* hydrogen occupies the site between two given oxygens at a time. The view represents the *average* of all unit cells in the crystal.

* See deposition footnote.

(1964) found one Te atom and two O atoms in the unit cell such that the stacking of the O atoms about the metal atom was a distorted octahedron. The two Te—O distances were 1·84 (2) and 2·01 (2) Å, and the oxygen to oxygen contact distances varied from 2·52 to 2·88 Å.

The formation of hydrogen bonds within any system causes changes in the involved molecules, and the title compound is no exception. The telluric acid molecules are solely joined by hydrogen bonding, and therefore the bridging should influence the normal free octahedral coordination. Twelve hydrogen bonds are associated with each telluric acid molecule, where each O atom is linked to an O from each of two adjacent molecules. The environment about each $Te(OH)_6$ unit is shown in Fig. 3, which gives the contents of the sphere about Te itself out to 6 Å. The arrangement of the twelve adjacent molecules is essentially that of a 'cube octahedron' (Muetterties & Wright, 1967), which resembles a tetracapped square prism having a Te atom at each corner of the prism and one above the middle of each face. The hydrogen-bonding description would be (12,12) according to the convention of Hamilton & Ibers (1968, p. 21).

The intermolecular O···H—O lengths range from 2·56 to 3·02 Å, indicating a mixture of strong and weak hydrogen bonding throughout the crystal lattice. The strength of H-bonding has been shown by Hamilton (1962) and Pedersen (1974) to be correlated with the O—H···O angle, and there is also an interrelationship between this value and the O···O distances, the hydrogen bond becoming weaker as the angle deviates more and more from linearity. The average O—H and O···H bond distances are 0·94 and 1·83 Å, respectively. According to the functional presentation of an empirical curve given by Hamilton & Ibers (1968, pp. 52–54), these average values and the determined experimental lengths of O—H and O···H are in good agreement with expected values (see Table 3). Two hydrogen-bonded O—H bands were observed in the IR spectrum of cubic H_6TeO_6 (pure), a strong band at 3200 cm^{-1} (3·1 μ, *vs*) and a medium shoulder at 3400 cm^{-1} (2·90–5 μ, *m*). Similar IR observations were made in the course of an X-ray diffraction study by Christensen & Broch (1967) and in a neutron diffraction investigation of $In(OH)_3$ (Mullica, Beall, Milligan, Korp & Bernal, 1979).

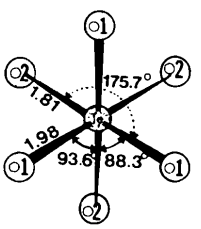

Fig. 2. Description of the distorted octahedral coordination of oxygens about the tellurium. (Distances are in Å.)

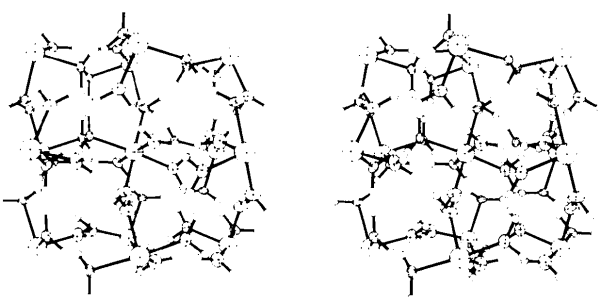

Fig. 3. Stereoscopic view of the contents of a sphere of radius 6 Å centered at Te. The disordered hydrogens are all shown at full weight, each directed towards the oxygen of a different adjacent molecule. Note the cube octahedral arrangement of peripheral molecules about the central one.

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$Rb_xP_8W_{32}O_{112}$: A Tunnel Structure Built up from ReO_3-Type Blocks and P_2O_7 Groups

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Abstract

The structure of a $Rb_{1\cdot6}P_8W_{32}O_{112}$ single crystal has been established by X-ray diffraction. The composition of this oxide with general formula $Rb_xP_8W_{32}O_{112}$ ($1\cdot6 \le x \le 2$) has been confirmed by a powder work. In a first step, the mean structure was solved in the space group $P2/c$ with $a = 16\cdot194$ (3), $b = 3\cdot7719$ (4), $c = 17\cdot095$ (4) Å, $\beta = 93\cdot89$ (2)°, leading to $R = 0\cdot038$ and $R_w = 0\cdot043$. Examination of the mean structure and the splitting of O atoms suggested the doubling of the b parameter. The actual structure, corresponding to the space group $P2_1/c$ with the cell a, $2b$, c, was confirmed by an accurate X-ray investigation and by electron microscopy. The host lattice of this structure, which is built up from WO_6 octahedra and PO_4 tetrahedra, can be described as ReO_3-type slabs connected through P_2O_7 groups. Several sorts of cages, with a geometry derived from that of perovskite, are formed; these cages are empty. The Rb^+ ions are inserted in tunnels with a strongly distorted hexagonal section, running along [010] and situated at the boundary between two ReO_3-type slabs. The possibility of insertion in this structure and the existence of microphases $Rb_xP_8W_{8n}O_{24n+16}$ are discussed.

Introduction

Few tunnel structures with a tetrahedra and octahedra corner-sharing host lattice are known at present, probably due to the great rigidity of the tetrahedral configuration. The X_2O_7 groups, which are formed of two corner-sharing tetrahedra, can however, sometimes be accommodated in an octahedral framework. This is the case for the siliconiobates and silicotantalates $A_3M_6Si_4O_{26}$ (Shannon & Katz, 1970; Choisnet, Nguyen, Groult & Raveau, 1976) with a pentagonal tunnel structure and for the intergrowths $(A_3M_6Si_4O_{26})_n.A_3M_8O_{21}$ (Nguyen, Studer, Groult, Choisnet & Raveau, 1976; Choisnet, Hervieu, Groult & Raveau, 1977). This ability to form such a framework can be explained by the height of the Si_2O_7 group, which is very close to that of a TaO_6 (or NbO_6) octahedron, allowing the connection of these polyhedra through their corners. In this respect, the P_2O_7 group should be able to accommodate the WO_6 octahedra due to the sizes of P and W which are respectively smaller than Si and Ta (or Nb). The present study describes the behaviour of the P_2O_7 groups and WO_6 octahedra in an oxide $Rb_xP_8W_{32}O_{112}$, characterized by a new tunnel structure.

Experimental

Sample preparation

During the synthesis of the hexagonal rubidium tungsten bronze from a mixture of composition $Rb_{0\cdot20}WO_3$ (Labbé, Goreaud, Raveau & Monier, 1978), in an evacuated silica ampoule at 1173 K, a single crystal of unknown composition was isolated.

The crystallographic data collected from this crystal showed that its structure and probably its composition had nothing to do with the hexagonal tungsten bronze structure. All attempts to obtain such crystals, from the composition $Rb_{0\cdot20}WO_3$, but in a platinum crucible *in vacuo*, were unsuccessful. The reaction of $Rb_{0\cdot20}WO_3$

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