



Article

# Potassium Disorder in the Defect Pyrochlore $\text{KSbTeO}_6$ : A Neutron Diffraction Study

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**Abstract:**  $\text{KSbTeO}_6$  defect pyrochlore has been prepared from  $\text{K}_2\text{C}_2\text{O}_4$ ,  $\text{Sb}_2\text{O}_3$ , and 15% excess  $\text{TeO}_2$  by solid-state reaction at 850 °C. Direct methods implemented in the software EXPO2013 allowed establishing the basic structural framework. This was followed by a combined Rietveld refinement from X-ray powder diffraction (XRD) and neutron powder diffraction (NPD) data, which unveiled additional structural features.  $\text{KSbTeO}_6$  is cubic,  $a = 10.1226(7)$  Å, space group  $Fd\bar{3}m$ ,  $Z = 8$  and it is made of a mainly covalent framework of corner-sharing  $(\text{Sb,Te})\text{O}_6$  octahedra, with weakly bonded  $\text{K}^+$  ions located within large cages. The large K-O distances, 3.05(3)–3.07(3) Å, and quite large anisotropic atomic displacement parameters account for the easiness of  $\text{K}^+$  exchange for other cations of technological importance.

**Keywords:** pyrochlores;  $\text{AB}_2\text{O}_6$ ;  $\text{ASbTeO}_6$ ; neutron powder diffraction; ionic diffusion

## 1. Introduction

Recently, the defect pyrochlore oxide  $(\text{H}_3\text{O})\text{SbTeO}_6$  has been described as an excellent proton conductor [1,2], showing a conductivity ( $\sigma$ ) of  $10^{-1}$  S·cm<sup>-1</sup> at 30 °C under saturated water vapor partial pressure, matching the performance of Nafion<sup>®</sup> as proton conductor for low-temperature fuel cells. Among the most promising candidates to replace Nafion, the so-called antimonic acids (of general stoichiometry  $\text{HSbO}_3 \cdot n\text{H}_2\text{O}$  or  $\text{Sb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ ) show a relatively high proton conductivity of  $\sim 10^{-4}$  S·cm<sup>-1</sup> at room temperature (RT) [3], and some yttrium-doped derivatives reach conductivities as high as  $10^{-3}$  S·cm<sup>-1</sup> [4]. An even larger  $\sigma$  value of  $10^{-1}$  S·cm<sup>-1</sup> at 30 °C under saturated water vapor partial pressure was described by Turrillas et al. [5], for an original derivative of the antimonic acid obtained by partial replacement of Sb by Te, giving rise to a well-defined oxide with pyrochlore structure and composition  $(\text{H}_3\text{O})\text{SbTeO}_6$  [5]. The pyrochlore structure is very appealing while searching for materials of high ionic conductivity, since its open framework containing three-dimensional interconnected channels enables  $\text{H}_3\text{O}^+$  ion diffusion. The general crystallographic formula of pyrochlore oxides is  $\text{A}_2\text{B}_2\text{O}_6\text{O}'$ , consisting of a covalent  $\text{B}_2\text{O}_6$  network of  $\text{BO}_6$  corner-sharing octahedra with an approximate B-O-B angle of 130°, and the  $\text{A}_2\text{O}'$  sub-lattice forming an interpenetrating network which does not interact with the former. It is well known that

both A cations and O' oxygens may be partially absent in defect pyrochlores with  $A_2B_2O_6$  or even  $AB_2O_6$  stoichiometry [6].

The full characterization of the crystal structure of  $(H_3O)SbTeO_6$  was performed by neutron diffraction, leading to the location of the protons in the framework [1].  $(H_3O)SbTeO_6$  has been prepared by ion exchange from  $KSbTeO_6$  pyrochlore in sulfuric acid at 453 K for 12 h [1,2]. The crystal structure of  $KSbTeO_6$  has not been described in detail, although a pioneering study reports the synthesis of the  $A(SbTe)O_6$  pyrochlore family ( $A = K, Rb, Cs, Tl$ ) [7]. The crystal structures of these oxides were defined in the  $Fd\bar{3}m$  space group (No. 227), with  $Z = 8$ . For  $A = K$ , the unit-cell parameter reported is  $a = 10.1133(2)$  Å. Sb and Te atoms were defined to be statistically distributed at  $16d$  Wyckoff sites; oxygen atoms were placed at  $48f$  sites, and A cations at  $32e$  ( $x,x,x$ ) Wyckoff positions with  $x = 0.109$ , from XRD data [7]. In the present work, we report the ab-initio crystal structure determination of  $KSbTeO_6$  from NPD data, followed by a Rietveld refinement from combined XRD and NPD data, yielding complementary information on the  $K^+$  positions.

## 2. Experimental

$KSbTeO_6$  was prepared by the solid-state reaction between potassium oxalate ( $K_2C_2O_4$ ),  $TeO_2$ , and  $Sb_2O_3$  in a 1:2.3:1 molar ratio, providing an excess of  $TeO_2$  to compensate for volatilization losses. The starting mixture was thoroughly ground and heated at 823, 973, 1073, and 1123 K for 24 h at each temperature, with intermediate grindings in order to ensure total reaction.

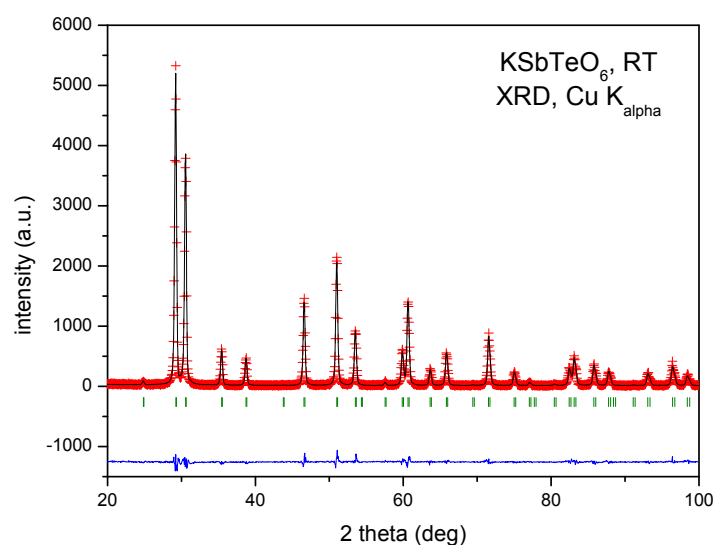
The initial product characterization was carried out by XRD with a Bruker-AXS D8 Advance diffractometer (40 kV, 30 mA) (Germany) controlled by the DIFFRACT<sup>PLUS</sup> software, in Bragg–Brentano reflection geometry, with Cu  $K_\alpha$  radiation ( $\lambda = 1.5418$  Å). A nickel filter was used to remove Cu  $K_\beta$  radiation. NPD experiments were carried out in the D2B high-resolution powder diffractometer ( $\lambda = 1.595$  Å) at the Institut Laue-Langevin, in Grenoble, France. About 2 g of sample was contained in a vanadium can. The full diffraction pattern was collected in 3 h.

The crystal structure was solved ab-initio from NPD data using direct methods and the software EXPO2013 [8]. The model obtained was refined by the Rietveld method [9] with the program FULLPROF (Grenoble, France, version Nov. 2016) [10], from combined XRD and NPD data. A pseudo-Voigt function was chosen to generate the line shape of the diffraction peaks. The following parameters were refined in the final Rietveld fit: scale factor, background coefficients, zero-point error, pseudo-Voigt profile function parameters corrected for asymmetry, atomic coordinates, anisotropic atomic displacement parameters for all atoms, and the occupancy factor of the  $K^+$  positions. The coherent scattering lengths of K, Sb, Te and O were 3.67, 5.57, 5.80 and 5.803 fm, respectively.

## 3. Results and Discussion

$KSbTeO_6$  oxide was obtained as a well-crystallized powder. The XRD pattern, shown in Figure 1, is characteristic of a pyrochlore-type structure, with  $a = 10.1226(7)$  Å. As input data for EXPO2013 [8], the unit-cell parameters,  $Fd\bar{3}m$  space group symmetry and unit-cell contents were given: 8 K, 48 O and 16 Sb, due to the similar Sb and Te scattering lengths. NPD data were used for the crystal structure determination, given their monochromaticity, well-defined peak shape, and the large  $2\theta$  range covered (from 0 to  $159^\circ$ ). EXPO2013 readily gave a structural model with O positions  $(\frac{1}{8}, \frac{1}{8}, 0.429)$  corresponding to  $48f$  Wyckoff sites, Sb positions  $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$  corresponding to  $16d$  sites, and two possible Wyckoff sites for K:  $(\frac{1}{8}, \frac{1}{8}, \frac{1}{8})$ , i.e.,  $8a$  sites; and  $(x,x,x)$ , i.e.,  $32e$  sites with  $x = 0.248$ , defined in the origin choice 2 of the space group  $Fd\bar{3}m$  (No 227). A combined XRD and NPD Rietveld refinement was carried out in that setting. The Sb and Te atoms were considered to be statistically distributed at  $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$   $16d$  Wyckoff sites, and K at  $(x,x,x)$   $32e$  sites. The  $K^+$  ions were allowed to shift along the  $(x,x,x)$   $32e$  position adopting intermediate  $x$  values between those suggested by the ab-initio crystal structure determination. At the stage of refining isotropic atomic displacement parameters,  $x = 0.1429(6)$  was reached for the  $(x,x,x)$   $32e$  Wyckoff position after convergence, accompanied by large temperature factors (B) of  $1.2(2)$  Å<sup>2</sup>.

A further fit improvement was achieved by refining anisotropic atomic displacement parameters, leading to the crystallographic data and Rietveld agreement factors gathered in Table 1.



**Figure 1.** Rietveld-refined XRD pattern of  $\text{KSbTeO}_6$  at 298 K, characteristic of a cubic pyrochlore phase. The experimental XRD data is represented with red crosses, the calculated profile is shown with a black solid line, and their difference is shown at the bottom (blue line). Vertical green symbols indicate allowed peak positions.

**Table 1.** Unit-cell, fractional atomic coordinates, atomic displacement parameters, refined occupancy factors and Rietveld agreement factors of  $\text{KSbTeO}_6$  in the cubic space group  $Fd\bar{3}m$  (No. 227), with  $Z = 8$ .

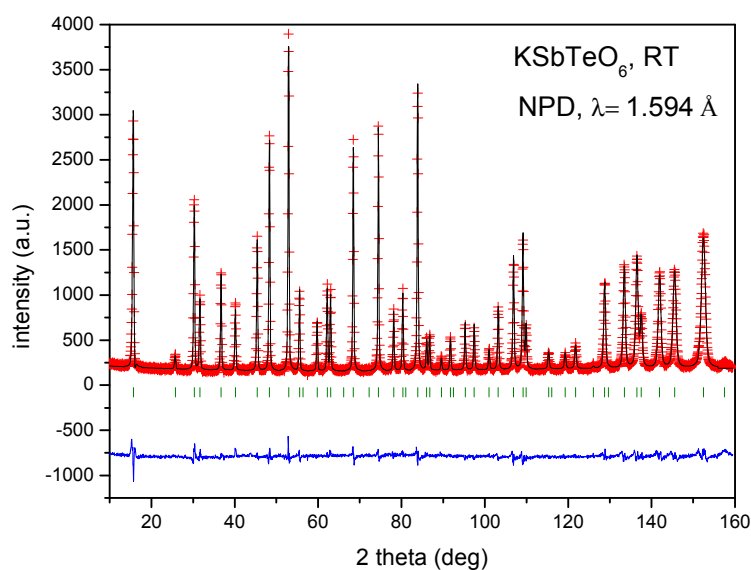
Crystal Data						
Cubic, $Fd\bar{3}m$			X-ray radiation, $\lambda = 1.5418 \text{ \AA}$			
			Neutron radiation, $\lambda = 1.595 \text{ \AA}$			
$a = 10.1226(7) \text{ \AA}$ $V = 1037.22(12) \text{ \AA}^3$			Particle morphology: powder $Z = 8$			
Rietveld Agreement Factors						
XRD data $R_p = 7.55\%$ $R_{wp} = 11.77\%$ $R_{exp} = 9.11\%$ $R_{Bragg} = 3.40\%$ $\chi^2 = 1.67$			NPD data $R_p = 4.75\%$ $R_{wp} = 6.27\%$ $R_{exp} = 3.85\%$ $R_{Bragg} = 3.59\%$ $\chi^2 = 2.65$			
1801 data points			3240 data points			
Atomic Coordinates, Isotropic Atomic Displacement Parameters ( $\text{\AA}^2$ ) and Refined Occupancy Factors						
	$x$	$y$	$z$	$U_{eq}$	Occupancy	
K	0.126(3)	0.126(3)	0.126(3)	0.060(4)	0.256(4)	
Sb1	0.50000	0.50000	0.50000	0.0037(3)		
Te1	0.50000	0.50000	0.50000	0.0037(3)		
O1	0.42760(9)	0.12500	0.12500	0.0099(3)		
Anisotropic Atomic Displacement Parameters ( $\text{\AA}^2$ )						
	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
K	0.055(3)	0.055(3)	0.055(3)	0.025(8)	0.025(8)	0.025(8)
Sb	0.0037(3)	0.0037(3)	0.0037(3)	−0.0004(3)	−0.0004(3)	−0.0004(3)
Te	0.0037(3)	0.0037(3)	0.0037(3)	−0.0004(3)	−0.0004(3)	−0.0004(3)
O	0.0075(4)	0.0111(3)	0.0111(3)	0.0	0.0	−0.0065(4)

In the final Rietveld refinement, the  $x$  parameter in the  $32e$  position shifted to 0.126(3). Thus, K practically occupies the  $(\frac{1}{8}, \frac{1}{8}, \frac{1}{8})$   $8a$  Wyckoff sites. The main interatomic distances and angles are shown in Table 2. Figures 1 and 2 illustrate the good agreement between the observed and calculated XRD and NPD patterns, respectively.

The Sb:Te ratio could not be refined, given the similar scattering factors (or scattering lengths for neutrons) of both elements using XRD or NPD. This ratio has to be 1:1 if K fully resides at  $8a$  Wyckoff sites, or at  $32e$  sites with an occupation of 1/4. The excess of  $\text{TeO}_2$  added to compensate for volatilization losses could also result in a slight over-occupation of the position with Te; therefore, an even lower occupation of the K position would occur. To address this problem, the occupancy of K was also refined: it converged to 1 atom per formula unit, within standard deviations (see Table 1), thus confirming the 1:1 Sb:Te ratio.

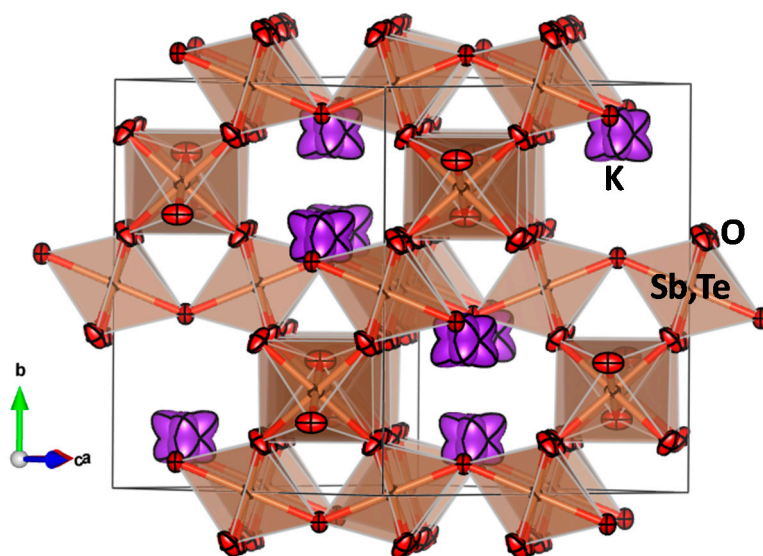
**Table 2.** Selected interatomic distances and angles for  $\text{KSbTeO}_6$  at 298 K.

Distances (Å)	
K-O (x3)	3.05(3)
K-O' (x3)	3.07(3)
(Sb,Te)-O (x6)	1.9338(6)
Angles (°)	
O-(Sb,Te)-O	86.10(3)
	93.90(3)
(Sb,Te)-O-(Sb,Te)	135.45(2)



**Figure 2.** Rietveld-refined NPD pattern of  $\text{KSbTeO}_6$  at 298 K in the cubic  $Fd\bar{3}m$  space group. The experimental NPD data is represented with red crosses, the calculated profile is shown with a black solid line, and their difference is shown at the bottom (blue line). Vertical green symbols indicate allowed peak positions.

Figure 3 displays the pyrochlore structure of  $\text{KSbTeO}_6$ , which can be described as composed of a mainly covalent network of  $(\text{Sb,Te})\text{O}_6$  units sharing corners, with a  $(\text{Sb,Te})\text{-O-(Sb,Te)}$  angle of  $135.45(2)^\circ$  (Table 2). The cage-like holes within this network contain the  $\text{K}^+$  ions statistically distributed at  $32e$  Wyckoff positions, with four times the required multiplicity to host  $\text{K}^+$  ions (eight per unit cell); thus, only one in four lobes within each  $\text{K}^+$  cluster shown in Figure 3 must be considered as occupied.



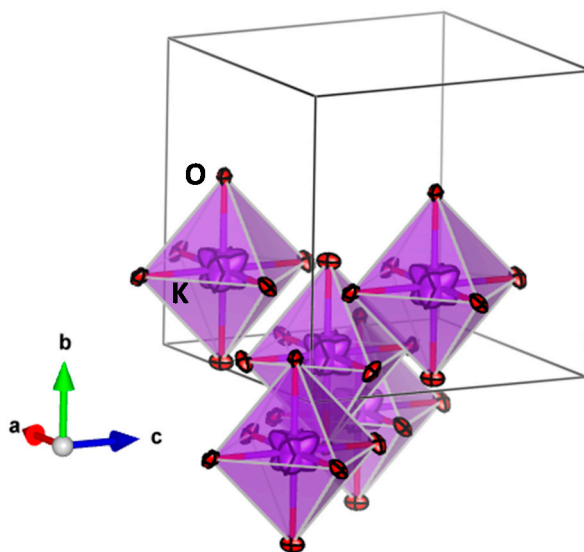
**Figure 3.** View of the  $\text{KSbTeO}_6$  pyrochlore structure approximately along the  $[110]$  direction. It consists of a mainly covalent framework of  $(\text{Sb,Te})\text{O}_6$  octahedra sharing vertices, forming large cages wherein  $\text{K}^+$  ions are distributed at  $32e$  Wyckoff sites with  $1/4$  occupancy and large anisotropic atomic displacement parameters.

The so-called  $(\text{Sb,Te})\text{O}_6$  octahedra are in fact slightly axially distorted, but they contain six equal  $(\text{Sb,Te})\text{-O}$  interatomic distances of  $1.9338(6)$  Å (Table 2), which compare well with  $1.96$  Å, Shannon's ionic radius sum [11].

The location of  $\text{K}^+$  ions at  $32e$  Wyckoff sites has been previously reported for the  $\text{ASbTeO}_6$  series [6]. It is noteworthy that, in pioneering work on defect  $\text{AB}_2\text{O}_6$  pyrochlores [12–14], the position of the A atoms was thought to be  $8a$ ; later on, the occupancy of  $(x,x,x)$   $32e$  positions, with  $x$  close to  $1/8$  was suggested [15–17]. For  $\text{KSbTeO}_6$ , the present work underlines the different results obtained refining isotropic atomic displacement parameters [ $x(\text{K}) = 0.1429(6)$ ], thus with  $\text{K}^+$  at  $32e$  Wyckoff sites; or anisotropic atomic displacement parameters, resulting in  $x(\text{K}) = 0.126(3)$ , very close to  $1/8$  and thus equivalent (within experimental error) to  $8a$  Wyckoff sites. If the  $\text{K}^+$  positions are fixed at the  $8a$  site, the Rietveld fit does not improve and the atomic displacement parameters of all atoms remain similar.

The  $\text{K}^+$  coordination is shown in Figure 4, with  $\text{K-O}$  distances of  $3.05$  and  $3.07$  Å (Table 2) in a pseudo-octahedral coordination to oxygen atoms. In defect  $\text{AB}_2\text{O}_6$  pyrochlores, it is worth recalling that for  $x$  equal or close to zero, the A atom can be considered as coordinated to six oxygen atoms only, forming a corrugated hexagon normal to the three-fold axis along the  $[111]$  direction. For increasing  $x$ , some new  $\text{A-O}$  distances decrease in such a way that for  $x$  equal to  $1/8$  ( $8a$  Wyckoff position in the  $Fd\bar{3}m$  space group), A atoms occupy the center of a wide cage formed by 18 oxygens, six of them at relatively short distances ( $3\text{O} + 3\text{O}'$ ), and 12 at larger distances ( $3\text{O}'' + \text{nine-additional oxygens}$ , which are not shown in Figure 4).

In the present structural description, with  $x$  virtually  $1/8$ , quite large anisotropic thermal ellipsoids (Figure 4) were determined, with r.m.s. displacements of  $0.324$  Å and  $0.172$  Å along the long and short ellipsoid axes, respectively. Furthermore, the crystal structure described accounts for the large mobility of  $\text{K}^+$  ions within the pyrochlore cages and the easiness of ion exchange that leads to  $(\text{H}_3\text{O})\text{SbTeO}_6$  by treatment in  $\text{H}_2\text{SO}_4$  [1,2], thus enabling the conversion of the present material in a technologically important compound with exceedingly high ionic conductivity.



**Figure 4.** Close up of the coordination polyhedra around  $K^+$  ions enhancing the lobes of the anisotropic thermal ellipsoids, with  $K^+$  statistically occupying one in four lobes within each polyhedron.  $(Sb,Te)O_6$  octahedra are not represented for clarity.

#### 4. Conclusions

$KSbTeO_6$  exhibits a defect pyrochlore structure defined in the cubic  $Fd\bar{3}m$  symmetry. The mainly covalent network formed by vertex-sharing  $(Sb,Te)O_6$  octahedra enables weak interatomic interactions with  $K^+$  ions. A combined XRD and NPD study showed that  $K^+$  occupies 32e Wyckoff sites indistinguishable (within experimental error) from 8a sites, placed in the center of a large cage determined by 6 K-O distances in the range 3.05(3)–3.07(3) Å. The quite big anisotropic atomic displacement parameters account for the easiness of ion exchange of this material to yield a product of technological importance,  $(H_3O)SbTeO_6$  [2].

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**Author Contributions:** José Antonio Alonso and Xabier Turrillas conceived and designed the experiments; Sergio Mayer, Horacio Falcón and María Teresa Fernández-Díaz performed the experiments; José Antonio Alonso and Xabier Turrillas analyzed the data; they all wrote the paper.

**Conflicts of Interest:** The authors declare no conflict of interest.

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