# The Formation and Structure of $Bi_{1.34}CrNbO_6$

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A new ternary oxide $Bi_{1.34}CrNbO_6$ was prepared and characterized. The unit cell is face-centered cubic with $a=10.455$ Å. The structure is assumed to be of the pyrochlore type based on the space group Fd3m and was refined by trial and error to a reliability factor $R$ of 0.0564. This pyrochlore phase was confirmed to have vacancies at the A sites and also at the special oxygen sites, and may be considered to be a rare case in this respect. The $Cr^{3+}$ and $Nb_5^+$ ions are bonded octahedrally to six oxygen atoms at 2.03 Å, while the $Bi^{3+}$ ions are bonded to six oxygen atoms at 2.56 Å. The structure consists of bismuth tetrahedra, the centers of which are vacant. The highly polarizable $Bi^{3+}$ ion plays an important role in the formation of the defect pyrochlore structure. Related pyrochlores were also prepared.

A number of mixed oxides with pyrochlore structure are known by the formula $A_2B_2O_7$,[1] and have provided a rich source of new materials exhibiting ferroelectricity[2] and promising electrooptic effects.[3] In recent years, many defect pyrochlores have been prepared based on the correlation between the perovskite and pyrochlore phases, and their structures and electrical properties have been investigated.[4-7] Most are oxygen deficient pyrochlores having the formula $A_2B_2O_{7-x}$ ($0<x\leqq 1$). Like $Pb_{1.5}Nb_2O_{6.5}$[2] and $TlSbWO_6$,[8] there are few pyrochlores which have vacancies at the A sites. During a study on the synthesis of Bi-containing mixed oxides, a new phase, $Bi_{1.34}CrNbO_6$, was found which exhibits an X-ray pattern of pyrochlore structure. As can be estimated from the chemical formula, this phase may be regarded as a rare defect pyrochlore which has vacancies at the A sites. In $TlSbWO_6$[8] and $Tl_{1-\alpha}(Ta_{1+\alpha}W_{1-\alpha})O_6$,[9] however, the A ions occupy special sites and, therefore, the latter is a nonstoichiometric pyrochlore. It is of interest to know how the structure of the defect pyrochlores can tolerate a balanced deficiency of both bismuth and oxygen ions. A suggestion can be given on the basis of systematic synthesis of defect pyrochlores. In the present study, the defect pyrochlore, $Bi_{1.34}CrNbO_6$, was prepared and are characterized. The position of the $Bi^{3+}$ ions in the cubic unit cell was determined and is discussed, and the formula of this compound is established. Also, related pyrochlores, $Bi_{1.34}B'B''O_6$, where B′=Cr and Fe and B″=Nb, Ta, and Sb were prepared and their dielectric properties were measured.

## Experimental

For sample preparations, starting materials were employed in the form of metal oxides. The reactants were all of reagent or better grade. Appropriate mixtures of metal oxides were fired at 950 °C for 2 h, ground and refined at 1050 °C for 3 h in air. An examination of the samples was carried out using the powder diffraction method employing a Philips diffractometer with Cu$K\alpha$ and occasionally Fe$K\alpha$ radiation. The lattice constants were determined from the reflection lines of (662) and (840). The X-ray intensity data were measured integrally for each peak. The theoretical intensity of a group of equivalent reflection lines was taken to be $I=KM(\mathrm{Lp})F^2$, where $K$ is the scale factor, $M$ the multiplicity, Lp the Lorentz-polarization factor, and $F$ the structure factor.[10] For structural refinement, the observed intensity was compared with the sum of the calculated intensities which appeared at the same value of $2\theta$. The scattering factors for $Bi^{3+}$, $Cr^{3+}$, $Nb^{5+}$, and $O^{2-}$ ions were taken from Moore.[11] The calculated structure factors were also corrected for anomalous dispersion using values given by Cromer.[12] The intensity computations were performed using a FACOM 270-30 computer with a Fortran program developed by this laboratory for the minimization of the reliability factor $R=\Sigma|I_{\mathrm{obsd}}-I_{\mathrm{calcd}}|/\Sigma I_{\mathrm{obsd}}$. Stereographic pictures of the pyrochlore structure were drawn using a computer program.[13] The dielectric constants were measured on a YHP universal bridge at 1 kHz. For the measurements, disks about 1 cm in diameter and 0.15 cm thick were coated with silver paint.

## Results

*Formation of $Bi_{1.34}CrNbO_6$ and Related Pyrochlores.* The new phase was prepared by firing a powdered mixture of $Bi_2O_3$, $Cr_2O_3$, and $Nb_2O_5$ having a molar ratio of 4: 3: 3. The X-ray diffraction powder pattern was completely indexed on the basis of a face-centered cubic unit cell with the dimension $a=10.455$ Å, as shown in Table 1. This pattern exhibits the reflection lines characteristic of pyrochlore structure. The chemical formula of the new phase $Bi_{1.34}CrNbO_6$, however, does not differ from that of normal or oxygen deficient pyrochlore. When mixtures of metal oxides corresponding to the formulae $Bi_2CrNbO_7$ and $Bi_{1.5}CrNbO_{6.25}$ were fired separately, the formation of phases other than the cubic pyrochlore phase was observed. In the case of Bi-rich components, the reacted products were partly melted even when fired at 900 °C. In all cases, there was almost not change in the relative intensity ratio and in the positions of the reflection lines observed in the powder patterns. The best preparation of this new pyrochlore phase resulted from the mixture with a molar ratio of 4: 3: 3, and it was found that no nonstoichiometric pyrochlore phase appears to exist.

Thermal analysis was carried out in order to determine whether oxidation or reduction occurs in the sample during the formation process. In the TG and DTA curves, an increase in weight began at *ca.* 520 °C and then the broad peak of the exothermic reaction was observed at 700 °C at a heating rate of 10 °C/min. An X-ray diffraction analysis of the intermediate stage showed the formation of $(BiO)_2CrO_4$ and supported the hypothesis that the oxidation of Cr ion in the sample took place during the heating run in air. Upon heating above

Table 1. X-Ray powder diffraction data for $Bi_{1.34}CrNbO_6$

| $h\ k\ l$ | $d_{obsd}$ | $I_{obsd}$ | $I_{calcd}$ |
|---|---|---|---|
| 1 1 1 | 6.03 | 10.0 | 10.8 |
| 3 1 1 | 3.15 | 9.4 | 9.6 |
| 2 2 2 | 3.02 | 100.0 | 100.0 |
| 3 3 1 | 2.40 | 28.2 | 29.8 |
| 5 1 1<br>3 3 3 | 2.01 | 2.4 | 1.8 |
| 4 4 0 | 1.848 | 30.1 | 31.7 |
| 5 3 1 | 1.766 | 1.7 | 0.5 |
| 6 2 0 | 1.652 | 0.9 | 0.1 |
| 5 3 3 | 1.594 | 0.2 | 0.1 |
| 6 2 2 | 1.575 | 23.3 | 23.3 |
| 4 4 4 | 1.513 | 6.0 | 5.2 |
| 7 1 1<br>5 5 1 | 1.463 | 0.9 | 0.6 |
| 8 0 0 | 1.307 | 2.4 | 2.5 |
| 7 5 1<br>5 5 5 | 1.207 | 0.9 | 0.5 |
| 6 6 2 | 1.199 | 6.2 | 5.2 |
| 8 4 0 | 1.169 | 5.8 | 4.1 |
| 9 1 1<br>7 5 3 | 1.147 | 0.4 | 0.5 |
| 9 3 1 | 1.096 | 0.2 | 0.2 |
| 8 4 4 | 1.067 | 3.0 | 2.7 |
| 9 5 1<br>7 7 3 | 1.011 | 0.4 | 0.1 |
| 6 6 6<br>10 2 2 | 1.006 | 3.0 | 2.5 |
| 8 8 0 | 0.924 | 0.6 | 0.6 |

760 °C, however, loss of oxygen occurred and the TG curve returned to its original level at *ca.* 950 °C. This level remained unchanged up to 1050 °C. After heating at this temperature for 2 h and then cooling, only formation of the new pyrochlore phase was found in the product. During a reheating run, the product showed no change in thermal analysis. Consequently, the Cr ions in this compound were assumed to be trivalent.

Table 2. Lattice and dielectric constants for some $Bi_{1.34}B'B''O_6$ pyrochlores

| Compound | $a$ (Å) | $\varepsilon$ |
|---|---|---|
| $Bi_{1.34}CrNbO_6$ | 10.455 | 72 |
| $Bi_{1.34}CrTaO_6$ | 10.449 | 35 |
| $Bi_{1.34}CrSbO_6$ | 10.374 | 56 |
| $Bi_{1.34}FeNbO_6$ | 10.506 | 116 |
| $Bi_{1.34}FeTaO_6$ | 10.501 | 43 |
| $Bi_{1.34}FeSbO_6$ | 10.421 | 39 |

The preparation of related compounds, all of which were of the pyrochlore type, was attempted. These lattice constants are shown in Table 2. The $Cr^{3+}$-containing pyrochlores are olive green in color, while the $Fe^{3+}$-containing compounds are dark organge, with a slight difference in hue. Attempts to prepare the $V^{5+}$-containing pyrochlores were unsuccessful even at high pressure (900 °C, 60 kb, 45 min). The lattice constant of $Bi_{1.34}CrNbO_6$ was slightly larger than that of $Bi_{1.34}CrTaO_6$, although both $Nb^{5+}$ and $Ta^{5+}$ ions are of the same size (0.64 Å) according to Shannon and Prewitt.[14] In this respect the $Fe^{3+}$-containing defect pyrochlores gave the same result as in the $Cr^{3+}$-containing ones. This is valid evidence for the existence of a defect pyrochlore type, since six pyrochlores could be prepared with starting compositions of $Bi_{1.34}B'B''O_6$.

*Refinement of $Bi_{1.34}CrNbO_6$.* The cell dimension and qualitative intensities of $Bi_{1.34}CrNbO_6$ strongly suggest that this compound has the pyrochlore structure belonging to the space group Fd3m.[15] Assuming $Bi_{1.34}CrNbO_6$ is of the pyrochlore type, one-third of the $Bi^{3+}$ ions and one-seventh of the oxygen ions are missing in comparison with normal pyrochlore. Such a defect pyrochlore is rare. In $Pb_{1.5}Nb_2O_{6.5}$, the $Pb^{2+}$ ions have been assumed to occupy the 16(d) sites.[2] In $TlSbWO_6$, the $Tl^{1+}$ ions have been reported to occupy the 32(e) sites between the 8(b) and 16(d) sites.[8] Although the pyrochlore structure itself has been studied extensively, it is important in the establishment of the defect pyrochlore structure to determine the arrangement of atoms in the new phase, $Bi_{1.34}CrNbO_6$. Since there are only one or two positional parameters for the pyrochlore structure, the 23 reflection lines observed in the powder pattern are considered to be sufficient for structural analysis. Their intensities were calculated using the positions listed in Table 3. The site with $\bar{3}$m symmetry

Table 3. Atomic positions for $Bi_{1.34}CrNbO_6$

| | | Space group: Fd3m (No. 227) |
|---|---|---|
| | | Lattice constant: 10.455 Å |
| | | Cell content: $8Bi_{1.34}CrNbO_6$ |
| | (0,0,0; 0,1/2,1/2; 1/2,0,1/2; 1/2,1/2,0)+ | |
| Bi | 16(d) | 1/2,1/2,1/2; 1/2,1/4,1/4; 1/4,1/2,1/4; 1/4,1/4,1/2 occupancy probability: 67% |
| Cr(Nb) | 16(c) | 0,0,0; 0,1/4,1/4; 1/4,0,1/4; 1/4,1/4,0 |
| O | 48(f) | $u$,1/8,1/8; $\bar{u}$,7/8,7/8; 1/4−$u$,1/8,1/8; 3/4+$u$,7/8,7/8; 1/8,$u$,1/8; 7/8,$\bar{u}$,7/8; 1/8,1/4−$u$,1/8; 7/8,3/4+$u$,7/8; 1/8,1/8,$u$; 7/8,7/8,$\bar{u}$; 1/8,1/8,1/4−$u$; 7/8,7/8,3/4+$u$ $u$=0.330 |
| O | 8(b) | 3/8,3/8,3/8; 5/8,5/8,5/8 occupancy probability: 0% |

was chosen as the origin. Firstly, the bismuth and oxygen vacancies were assumed to lie on one-third of the 16(d) sites and on the 8(b) sites, respectively, as shown in Fig. 1. Then, minimization of the reliability factor $R$ to the final value of 0.0564 yielded the following results, $u$=0.330 and $B$=4.18 Å$^2$. The observed and calculated intensities for these parameters are listed on the right side of Table 1, and good agreement between both intensities is obtained. The density of the sample measured with a pycnometer was 6.01 g/cm$^3$, which corresponds to 8 formula weights of $Bi_{1.34}CrNbO_6$ per unit cell (theoretical density, 6.04 g/cm$^3$). When the random distribution of $Bi^{3+}$ ions in the 16(d) and 8(b) sites or of oxygen ions in the 48(f) and 8(b) sites was assumed, the minimum values of $R$ became considerably larger. This fact indicates erroneous assumptions. Next, when the $Bi^{3+}$ ions were assumed to lie only on the

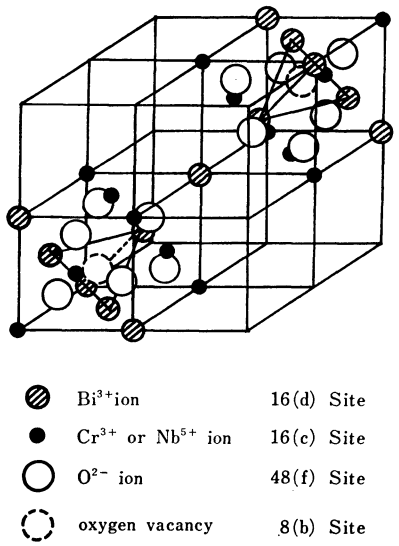


Fig. 1. Crystal structure of defect pyrochlore $Bi_{1.34}CrNbO_6$.

32(e) sites, instead of the 16(d) sites, the final $R$ value was refined to be 0.0562 with $u_a=0.495$ (32(e) positional parameter) and $B=4.04$ Å$^2$. The slight decrease in the $R$ value might be related with experimental errors in the intensity measurement. The $u_a$ value obtained is close to 0.500, a value indicating the 16(d)-site occupancy of $Bi^{3+}$ ions, differing from that ($u_a=0.403$) in $TlSbWO_6$.[8] From these results, it is not necessary to conclude that the $Bi^{3+}$ ions lie on the 32(e) sites and that they deviate slightly from the 16(d) sites. The selected interatomic distances and angles in $Bi_{1.34}CrNbO_6$ were calculated on the first assumption, and are shown in Table 4. These values are in reasonable accord with the ionic sizes of Shannon and Prewitt.[14] The O–Cr(Nb)–O angles are a measure of the distortion of the oxygen octahedra, since all of these angles are 90° in the case of a regular octahedron.

Table 4. Selected interatomic distances and angles for $Bi_{1.34}CrNbO_6$

| Distance | Å | Angle | Deg. |
|---|---|---|---|
| Bi–O | 2.56 | O–Bi–O | 63.5 |
| Cr(Nb)–O | 2.03 | O–Bi–O | 116.5 |
| O–O | 2.70 | O–Bi–O | 180.0 |
| O–O | 3.03 | O–Cr(Nb)–O | 83.3 |
| Bi–Bi | 3.70 | O–Cr(Nb)–O | 96.7 |

In order to further examine this defect pyrochlore phase, the values of the reliability factor $R$ for two series of proposed formulae, $Bi_{2-x}CrNbO_{7-3x/2}$ and $Bi_{2-x}Cr_{2-3x/2}Nb_{3x/2}O_6$, were calculated using the two parameters ($u=0.330$ and $B=4.18$ Å$^2$) determined previously. Figure 2 shows the relationship between $R$ and $x$. In both curves, the $R$ values are minima respectively when $x=2/3$, the $x$ value of which corresponds to the chemical formula of $Bi_{1.34}CrNbO_6$. The $R$ values rapidly increase with any deviation from this $x$ value. This result provides conclusive proof that the defect pyrochlore phase has the true composition, $Bi_{1.34}CrNbO_6$.

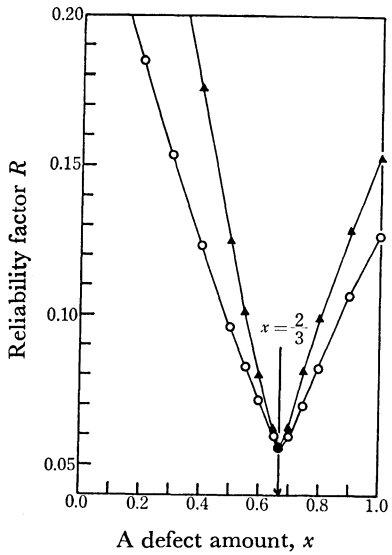


Fig. 2. Relation between $R$ and $x$ for two series of proposed formula.
○: $Bi_{2-x}CrNbO_{7-3x/2}$.
▲: $Bi_{2-x}Cr_{2-3x/2}Nb_{3x/2}O_6$.

*Dielectric Properties.* The six defect pyrochlores prepared had dielectric constants of *ca.* 30—110 at room temperature, as shown in Table 1, although no correction was made in regard to sample porosity. These values are relatively high in general, which fact renders them worth examining. The dielectric constants of $Nb^{5+}$-containing pyrochlores are considerably higher than those of $Ta^{5+}$- and $Sb^{5+}$-containing pyrochlores. Since these defect pyrochlores are of the cubic and centrosymmetric space group, the dielectric measurements were carried out in the temperature range from −150 to 20 °C. Figure 3 shows the temperature dependence of $\varepsilon$ and tan $\delta$ for $Bi_{1.34}CrNbO_6$ and $Bi_{1.34}FeNbO_6$. No dielectric anomaly was observed, and $\varepsilon$ and tan $\delta$ increased monotonically with increasing temperature. These defect pyrochlores are considered to be paraelectric in this temperature range. They have high electric resistivities which, although not

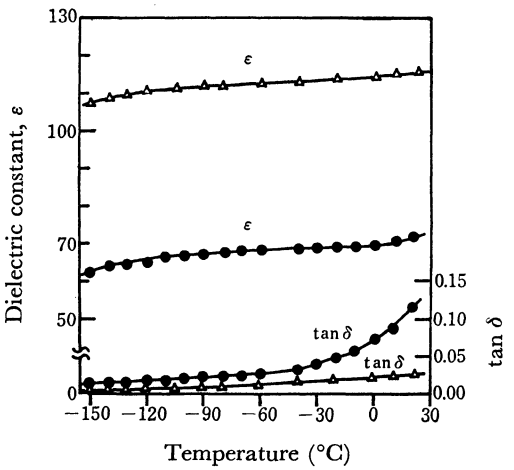


Fig. 3. Temperature dependence of $\varepsilon$ and tan $\delta$ for two defect pyrochlores.
●: $Bi_{1.34}CrNbO_6$. △: $Bi_{1.34}FeNbO_6$.

measured directly, were high enough to balance a capacitance bridge. There should be a little change even when transition metal ions are present in the ambivalent oxidation state, since the electrical resistivity is expected to be sensitive to the state.

## Discussion

The A cations in the pyrochlore structure are usually in eightfold coordination. In the case of $Bi_{1.34}CrNbO_6$, however, the $Bi^{3+}$ ions have a sixfold coordination with an equivalent distance of 2.56 Å, since the 8(b) sites are not occupied by oxygen ions. The $Cr^{3+}$ and $Nb^{5+}$ ions are bonded octahedrally to six oxygen ions at a distance of 2.03 Å. The positional $u$ parameter of 0.330 is within the range of 0.305 to 0.355 reported by Sleight[16] for twelve pyrochlores and is close to the values for rare-earth pyrochlores. The oxygen octahedra are slightly distorted because of the deviation from the 0.3125 value for a regular octahedron. The oxygen-oxygen distances in the octahedra are 2.70 and 3.03 Å. The octahedra form corners with other octahedra to form a three-dimensional framework, as shown in Fig. 4. The 8(b)-site vacancies are located at the

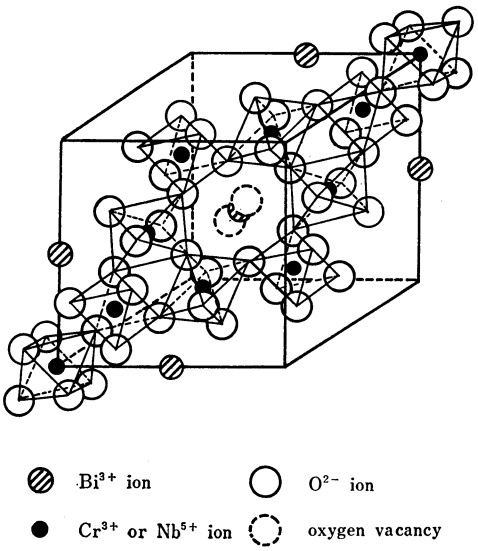


Fig. 4. Framework structure of defect pyrochlore $Bi_{1.34}CrNbO_6$.

centers of bismuth tetrahedra and the occupancy probability of $Bi^{3+}$ ions is 67% at the tetrahedral corners. The Bi-Bi distance was 3.70 Å and the bismuth ions are in close proximity to each other, being unscreened. In normal pyrochlores, the 8(b) sites are filled by oxygen ions and, therefore, the defect pyrochlore structure should be unstable because of the repulsion between the $Bi^{3+}$ ions, judging only from electrostatic considerations. No pyrochlore phases, however, were formed when the $Bi^{3+}$ ion was replaced by a $La^{3+}$ or $Y^{3+}$ ion as the A-site ion in the $A_{1.34}CrNbO_6$ and $A_2CrNbO_7$ forms. Occupancy by a $La^{3+}$ or $Y^{3+}$ ion appears to be unfavorable for the formation of such a defect pyrochlore structure, while occupancy by a $Bi^{3+}$ ion appears favorable. The ionic sizes of $La^{3+}$, $Y^{3+}$, and $Bi^{3+}$ ions are 1.18, 1.015, and 1.11 Å respectively,[14] and these ions are of suitable size for the A-site ion in the pyrochlore structure. Consequently, the essential nature of A-site ions must be considered in the formation of defect pyrochlores. That is to say, the $La^{3+}$ and $Y^{3+}$ ions are of ionic character and the $Bi^{3+}$ has a significant contribution of covalent character. Even though the Bi atom is completely ionized to the valence state of +3, the $Bi^{3+}$ ion has outer $6s^2$ core electrons which easily polarize the neighiboring oxygen ions and exhibit directional bonding. The remarkable arrangement of unscreened $Bi^{3+}$ ions in $Bi_{1.34}CrNbO_6$ has often been found for oxides containing an inert-pair ion, as in PbO, $Bi_2O_3$, and SnO.[17] The abnormal stereochemistry of these compounds is due to s-p hybridization. The trap-mediated metal-metal bond pointed out by Goodenough *et al.*[4] may also be responsible for the defect pyrochlore formation of $Bi_{1.34}CrNbO_6$. The 8(b)-vacancy sites act as traps for the outermost electrons of the $Bi^{3+}$ ions, and the 6s and 6p orbitals spread out into the vacancy sites, where considerable electron density is concentrated. The electrostatic repulsion between the $Bi^{3+}$ ions may be counteracted by the transfer of the outermost electrons to the vacancy sites, and the defect pyrochlore structure of $Bi_{1.34}CrNbO_6$ is stabilized by this Bi–Bi bonding. A 67% occupancy of $Bi^{3+}$ ions at the 16(d) sites is naturally determined from the electroneutrality condition, considering that the $Cr^{3+}$ and $Nb^{5+}$ ions contribute to the framework of the defect pyrochlore structure. The value itself exhibits no significant meaning in this case. However, the case appears to be different for $TlSbWO_6$ where the occupancy probability of $Tl^{1+}$ ions is only 50%. In the case of $TlSbWO_6$, the 32(e) positional parameter for the $Tl^{1+}$ ion has been determined to be $u_a=0.403$.[8] The 32(e) site lies on a straight line between the 16(d) and 8(b) sites. As shown in Fig. 1, the 16(d) and 8(b) sites are the corners and center of the cationic tetrahedron, respectively. The 32(e) site corresponds to the 16(d) site at $u_a=0.500$, and to the 8(b) site at $u_a=0.375$. Consequently, the Tl–Tl distance becomes smaller with decreasing $u_a$. In $TlSbWO_6$, the 32(e)-site occupancy of $Tl^{1+}$ ions, which deviates considerably from the 16(d) site, would enable the outermost electrons to overlap sufficiently at the vacancy sites despite the low occupancy probability. The circumstances under which the trap-mediated bond is formed may be severe because of the rather large Tl–Tl distance under the condition that the $Tl^{1+}$ ions occupy half of the 16(d) sites. For that reason, the assumption that the $Bi^{3+}$ ions occupy the 32(e) site with $u_a=0.495$ in $Bi_{1.34}CrNbO_6$ might be correct, although deviation from the 16(d) sites is slight. This slight deviation is probably due to the fact that the occupancy probability of A-site ions is higher in $Bi_{1.34}CrNbO_6$ than in $TlSbWO_6$. Thus, the $Bi^{3+}$ ions not only occupy open spaces in the framework, but also play an important role in the formation of defect pyrochlore structures. $Bi_{1.34}CrNbO_6$ and related compounds are rare examples of a defect pyrochlore type which has vacancies at the A sites and also at special oxygen sites.

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