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The Crystal Structure of α-Sodium Hexafluorooxoniobate(V), α-$Na_3[NbF_6O]$

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Crystals of α-$Na_3[NbF_6O]$ are orthorhombic, space group $P2_12_12_1$ (No. 19), with $a=5.6481(4)$ Å, $b=5.7661(5)$ Å, $c=16.9340(13)$ Å and $Z=4$. The axial ratio is 1:1.0209:2.9982. Reflexion intensities were registered at room temperature with an automatic single-crystal X-ray diffractometer using Mo$K\alpha$ radiation. Least-squares refinement of structural and thermal parameters yielded a final R-value of 0.034 for 1173 observed reflexions.

Another orthorhombic phase with the approximate cell dimensions $a=5.60(1)$, $b=11.65(2)$, $c=8.46(1)$ Å and $V=552(3)$ Å ^{3} appeared in the system $NaF-Nb_2O_5-HF-H_2O$ after α-$Na_3[NbF_6O]$ had begun to separate on evaporation of the solvent. Within experimental errors the two phases have the same cell volumes.

The crystals contain sodium ions and hexafluorooxoniobate(V) ions. In the complex anion niobium is surrounded by a pentagonal bipyramidal arrangement of ligands with five fluorine atoms forming the equatorial plane and one fluorine atom and the oxygen atom occupying the apical positions. This is thus not in accord with the monocapped octahedral structure proposed for $[NbF_6O]^{3-}$ in the disordered cubic $K_3[NbF_6O]$.

Bond distances are: $Nb-F_{equatorial}$ 2.016–2.050(3) Å, $Nb-F_{apical}$ 2.084(3) Å and Nb=O 1.738(3) Å. Coordination distances to the sodium ions range from 2.221(3) Å to 2.677(4) Å.

About ten extremely weak reflexions, indicating the existence of a superstructure, were observed on the Weissenberg photographs taken in the initial stage; they were, however, too weak to be registered as significant reflexions by the diffractometer.

Based on the result of their structure investigation using powder diffraction data, Williams *et al.*[1] claim that the geometrical arrangement of the $[NbF_6O]^{3-}$ ion in $K_3[NbF_6O]$ is a capped octahedron, as was also deduced by Hampson *et al.*[2] for the $[ZrF_7]^{3-}$ ion in $(NH_4)_3[ZrF_7]$ and $K_3[ZrF_7]$. Since all these compounds crystallize in the cubic system, space group *Fm*3*m*, their structures are incompatible with ordered orientations of the anions. Zachariasen pointed out that $(NH_4)_3[ZrF_7]$ and $K_3[ZrF_7]$ are isostructural with the disordered cubic form of $K_3[UF_7]$.[3] The ordered tetragonal form of $K_3[UF_7]$ contains pentagonal bipyramidal $[UF_7]^{3-}$ ions.[3] Zachariasen, therefore, found it likely that the $[UF_7]^{3-}$ and $[ZrF_7]^{3-}$ ions in the cubic structures should exhibit the same geometry. The disorder of $(NH_4)_3[ZrF_7]$ has been further investigated by Hurst *et al.*,[4] who also proposed a model with a pentagonal bipyramidal geometry. The same situation was also observed by Stomberg *et al.* in $(NH_4)_3[TiF_5(O_2)]$.[5] This compound was later investigated by Massa *et al.*,[6] who preferred to describe the disorderly orientated anion as octahedral.

Besides the above-mentioned geometrical arrangements of ligands, the capped octahedron and the pentagonal bipyramid, there is a third possibility, the capped trigonal prism, exemplified by the $[ZrF_7]^{2-}$ ion in $K_2[ZrF_7]$.[7,8] The available structural data indicate that none of these three geometrical arrangements is particularly favoured unless a bias might be built into a polydentate ligand. Also, all three will, in general, have similar stability.

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As already pointed out by Baker *et al.*,[9] it would be of interest to determine the geometry of the $[NbF_6O]^{3-}$ ion by single-crystal studies. In connection with structural work on fluoroperoxoniobates performed at this department, mainly with sodium as cation, fluorooxoniobates are intermediates.[10–13] It, therefore, seemed natural to consider these. The structure of one of these, $(C_9H_8NO)_2[NbF_5O]\cdot 2H_2O$, containing a disorderly orientated octahedral anion, has already been published,[14] while that of α-$Na_3[NbF_6O]$ is reported in this paper.

EXPERIMENTAL

Preparation. 8 g sodium fluoride and 12 g niobium(V) oxide were dissolved in an excess of boiling 38 % hydrofluoric acid in a platinum crucible. Most of the hydrofluoric acid was expelled by repeated addition of water and subsequent evaporation to almost dryness. The final volume was adjusted to 200 ml. When half of the solvent had been evaporated at room temperature, a crystalline mass had begun to separate out. pH was then about 2. From this mass, crystals were picked out for the single-crystal X-ray work. Only a few could be used, most being multiple crystals. The rest, which proved to be a single phase (α), was separated from the solution and analysed. Further crystallization yielded at least one other phase (β) intergrown with the first one.

Analysis. Niobium was determined gravimetrically by precipitation as the hydroxide with ammonia and ignition to the oxide. Sodium was determined by flame emission spectroscopy. Fluoride was determined by potentiometric titration with lanthanum nitrate at pH 5.4 using a fluoride selective electrode (Radiometer fluoride selectrode^R F1052F). (Found: Nb 31.9; Na 22; F 39.3. Calc. for $Na_3[NbF_6O]$: Nb 31.8; Na 23.6; F 39.1).

X-Ray methods. X-Ray powder photographs were obtained by the Guinier-Hägg method (Cu$K\alpha_1$ radiation) using $Pb(NO_3)_2$ (a=7.8566 Å at 21 °C) as an internal standard.

Rotation photographs showed that reflexions with l odd were considerably weaker than those with l even. Weissenberg photographs with rotation about the c axis were registered for l=0–8, 10 and 12, using graphite-monochromatized Cu$K\alpha$ radiation. With a few exceptions the observed reflexions could be indexed according to an orthorhombic cell with a=5.65, b=5.77 and c=16.9 Å. Eleven clearly visible, very weak reflexions could be indexed only by doubling the a and b axes, thus indicating the existence of a superstructure. None of these reflexions (with indices 110, 101, 011, 112, 132, 312, 103, 303, 503, 114, 116 referring to the superstructure cell) was stronger than the weakest observed reflexion due to the smaller cell. Other crystals showed the same situation.

Intensity data were recorded at room temperature for the larger cell (a=11.307(4) Å, b=11.541(5) Å and c=16.919(4) Å, determined from a least-squares fit of refined diffractometer setting angles for 15 reflexions) with a SYNTEX $P2_1$ automatic four-circle single-crystal X-ray diffractometer using graphite-monochromatized Mo$K\alpha$ radiation and a crystal with the dimensions 0.15×0.15×0.26 mm. The $\theta-2\theta$ method was used and the 2θ scan speed was allowed to vary between 3 and 20 °/min, depending on the intensity of the measured reflexion. Data were collected for $2\theta \leq 78°$. Three test reflexions, measured after each forty-seventh reflexion, showed no significant difference in intensity during the data collection. A profile analysis based on the Lehmann-Larsen method [15] was applied to the 96-step profile collected for each reflexion.

Of the 6580 measured, independent reflexions 1173, having $I_o \geq 3\sigma(I_o)$, were regarded as being observed and were used in the subsequent calculations. All observed reflexions could be indexed according to the smaller cell. Thus, none of the above-mentioned very weak, photographically registered reflexions was strong enough to be registered by the diffractometer. The structure solution is, therefore, based upon the smaller cell. The intensities were corrected for Lorentz, polarization and absorption effects. For the absorption correction the crystal was divided into a 6×6×6 grid. The transmission factor varied between 0.60 and 0.65.

The unit cell parameters were determined by a least-squares fit of observed and calculated $\sin^2\theta$-values for 57 unequivocally indexed reflexions obtained from two independent powder photographs.

Preliminary crystallographic data for the β-phase, which appeared on further crystallization (*vide supra*), were obtained from rotation and Weissenberg photographs. These indicated an orthorhombic cell with the approximate cell dimensions a=5.60(1), b=11.65(2), c=8.46(1) Å and V=552(3) Å^3. The crystal used was intergrown with the α-phase, the respective axes being parallel (*i.e.* $a_\alpha \| a_\beta$, etc.), as far as could be judged from the photographs. The axial ratio for the β-phase is 1:2.080:1.511 and the ratios $a_\alpha:a_\beta$, $b_\alpha:b_\beta$, and $c_\alpha:c_\beta$ are 0.991, 2.020 and 0.500, respectively. The cell volumes, V_α=551.50(8) Å^3 and V_β=552(3) Å^3, do not differ significantly.

The above evidence supports the view that the two phases are isostoichiometrical. Since the α-phase could be obtained pure, the single-crystal study was performed on that phase.

CRYSTAL DATA

α-Sodium hexafluorooxoniobate(V), α-$Na_3[NbF_6O]$; *F.W.* =291.87
Space group $P2_12_12_1$ (No. 19)
$a=5.6481(4)$ Å, $b=5.7661(5)$ Å, $c=16.9340(13)$ Å, $V=551.50(8)$ Å^3, $Z=4$, $D_o=3.53$ g cm^{-3}, $D_c=3.52$ g cm^{-3}, $\mu(\text{Mo}K\alpha)=2.38$ mm^{-1}, $\lambda(\text{Mo}K\alpha)=0.7107$ Å, $\lambda(\text{Cu}K\alpha_1)=1.54051$ Å.

A list of observed lines in the powder photograph of α-$Na_3[NbF_6O]$, giving *hkl*, d_o, d_c and I_o, as well as lists of observed and calculated structure factors and anisotropic thermal parameters are available from the author upon request.

STRUCTURE DETERMINATION

As none of the few very weak reflexions indicating a supercell, observed on the Weissenberg photographs, was strong enough to be registered as significant by the diffractometer, the structure determination is based on the smaller cell.

The positions of the niobium atom and two sodium atoms were obtained from the Patterson function and the third sodium atom from the first electron density map. Since these atoms have rather special positions, a lot of false symmetry was introduced in the subsequent electron density map. A tedious trial method in identifying this map led to a plausible structure, which could be successfully refined.

Full-matrix least-squares refinement of an overall scale factor and positional and isotropic thermal parameters for all atoms yielded an *R*-value of 0.057 ($R=\Sigma||F_o|-|F_c||/\Sigma|F_o|$). When anisotropic thermal parameters were introduced for all atoms, the *R*-value was reduced to 0.034. The weighting scheme used was that of Cruickshank:[16] $w=(a+|F_o|+c|F_o|^2+d|F_o|^3)^{-1}$ with $a=15$, $c=0.01$ and $d=0.001$. The scattering factors for Nb, Na^+, F, and O were taken from Ref. 17 as were the dispersion corrections.

The largest peak in a difference synthesis calculated after the final cycle of refinement, appeared at 0.8 Å from niobium with a peak height of 2 e/Å^3. No other peak had a height exceeding 0.7 e/Å^3.

Calculations were carried out on an IBM 3033 computer using the crystallographic programmes described by Lindgren.[18]

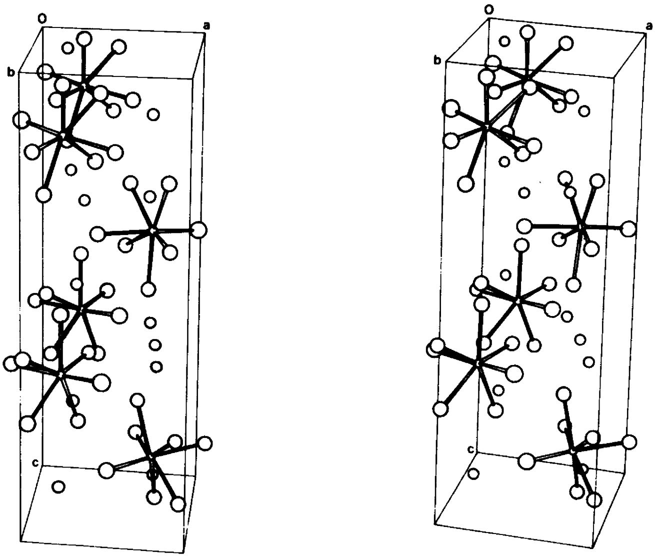

Fig. 1. Stereoscopic drawing of the unit cell of α-$Na_3[NbF_6O]$.

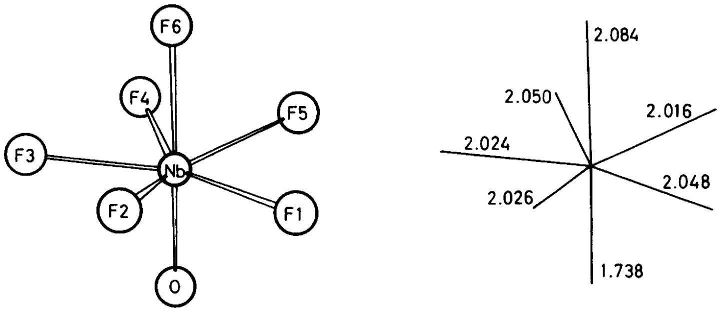

Fig. 2. The ion $[NbF_6O]^{3-}$.

RESULTS AND DISCUSSION

The positional and thermal parameters obtained in the last refinement cycle are given in Table 1. A stereoscopic drawing of the unit cell is shown in Fig. 1 and the anion in Fig. 2. Bond distances and angles are given in Table 2 and coordination distances to the sodium ions in Table 3.

The crystals of α-sodium hexafluorooxoniobate(V), α-$Na_3[NbF_6O]$, are composed of sodium ions and hexafluorooxoniobate(V) ions, held together by ionic forces.

Niobium is seven-coordinated to six fluorine atoms and one oxygen atom. The arrangement of the ligands in $[NbF_6O]^{3-}$ is somewhat distorted pentagonal bipyramidal with five fluorine atoms, F1–F5, forming the pentagonal plane and one fluorine atom, F6, and one oxygen atom occupying the apical positions. This investigation thus contradicts the monocapped octahedral configuration proposed by Williams *et al.* based on a structure determination of $K_3[NbF_6O]$.[1] The distances from the least-squares plane through F1–F5 to these atoms and to Nb, F6 and O are in order 0.163, −0.124, 0.040, 0.069, −0.148, 0.187, −1.893 and 1.926 Å, respectively. The largest observed deviation from the 72° angle between neighbouring Nb–F bonds in the pentagonal plane, expected for full C_5 symmetry, is 1.2°. The geometry of the $[NbF_6O]^{3-}$ ion is, therefore, completely different from that of the $[NbF_7]^{2-}$ ion observed in $K_2[NbF_7]$.[8] The latter is described as a distorted monocapped trigonal prism. Seven-coordinated complexes are considered to be stereochemically nonrigid, since, according to theory, interconvertions between the three geometrical arrangements of ligands can occur without difficulty. One might, therefore, expect considerable distortions in such systems, $[NbF_7]^{2-}$ being one example. In $[NbF_6O]^{3-}$ the five equatorial atoms deviate significantly (by 20–60σ) from coplanarity. This may be compared with the situation in seven-coordinated transition metal peroxo compounds, the five equatorial atoms

Table 1. Positional and thermal parameters for α-$Na_3[NbF_6O]$. $U_{eq}=\frac{1}{3}(U_{11}+U_{22}+U_{33})$.

Atom	x	y	z	$U_{eq}/Å^2$
Nb	0.25348(6)	−0.00913(4)	0.12488(2)	0.0101(1)
Na1	0.2638(4)	0.0124(3)	0.3749(1)	0.0204(4)
Na2	0.2371(3)	0.4688(3)	0.2562(1)	0.0195(3)
Na3	0.2198(4)	0.4908(3)	−0.0034(1)	0.0239(4)
F1	0.1566(5)	0.0768(5)	0.2377(2)	0.0208(5)
F2	0.1243(5)	0.3187(4)	0.1235(1)	0.0226(5)
F3	0.2693(6)	0.1017(4)	0.0116(1)	0.0239(5)
F4	0.4459(5)	−0.2436(5)	0.0604(2)	0.0206(5)
F5	0.4193(5)	−0.2289(4)	0.1992(1)	0.0233(5)
F6	0.5780(5)	0.1608(4)	0.1341(1)	0.0199(5)
O	−0.0098(5)	−0.1608(5)	0.1123(2)	0.0188(6)

Table 2. Bond distances and angles in α-$Na_3[NaF_6O]$.

Distance/Å		Angle/°	
Nb–F1	2.048(3)	F2–Nb–F3	73.1(1)
Nb–F2	2.026(2)	F2–Nb–F4	143.1(1)
Nb–F3	2.024(2)	F2–Nb–F5	139.6(1)
Nb–F4	2.050(3)	F3–Nb–F6	83.1(1)
Nb–F5	2.016(3)	F2–Nb–O	99.2(1)
Nb–F6	2.084(3)	F3–Nb–F4	71.3(1)
Nb–O	1.738(3)	F3–Nb–F5	140.3(1)
Angle/°		F3–Nb–F6	83.3(1)
		F3–Nb–O	94.6(1)
F1–Nb–F2	71.9(1)	F4–Nb–F5	70.8(1)
F1–Nb–F3	145.0(1)	F4–Nb–F6	83.3(1)
F1–Nb–F4	143.0(1)	F4–Nb–O	93.2(1)
F1–Nb–F5	72.2(1)	F5–Nb–F6	80.8(1)
F1–Nb–F6	92.9(1)	F5–Nb–O	99.1(1)
F1–Nb–O	90.4(1)	F6–Nb–O	176.4(1)

Table 3. Cation environment in α-$Na_3[NbF_6O]$.

Distance/Å		Distance/Å	
Na1···F1	2.429(3)	Na2···F5^{iii}	2.374(3)
Na1···F2^{i}	2.461(3)	Na2···F6^{iii}	2.401(3)
Na1···F3^{ii}	2.414(3)	Na2···O$_{v}$	2.677(4)
Na1···F4^{iii}	2.423(3)	Na3···F2	2.427(3)
Na1···F5^{iii}	2.646(3)	Na3···F3	2.275(3)
Na1···F6^{iv}	2.221(3)	Na3···F3^{vii}	2.603(4)
Na1···O^{v}	2.378(3)	Na3···F4^{vi}	2.269(3)
Na2···F1^{v}	2.312(3)	Na3···F4^{vii}	2.334(3)
Na2···F1	2.327(3)	Na3···F6^{vii}	2.510(3)
Na2···F2	2.492(3)	Na3···O^{viii}	2.587(4)
Na2···F5^{vi}	2.243(3)		

Symmetry codes

i	$(\bar{x},-\frac{1}{2}+y,\frac{1}{2}-z)$	v	$(\bar{x},\frac{1}{2}+y,\frac{1}{2}-z)$
ii	$(\frac{1}{2}-x,\bar{y},\frac{1}{2}+z)$	vi	$(x,1+y,z)$
iii	$(1-x,\frac{1}{2}+y,\frac{1}{2}-z)$	vii	$(-\frac{1}{2}+x,\frac{1}{2}-y,\bar{z})$
iv	$(1-x,-\frac{1}{2}+y,\frac{1}{2}-z)$	viii	$(\frac{1}{2}+x,\frac{1}{2}-y,\bar{z})$

being much nearer to coplanarity. The bidentate peroxo group thus seems to stabilize the pentagonal bipyramidal geometry.

The niobium atom is displaced 0.19 Å from the equatorial plane towards the oxygen atom. The effect of this is that the distances between the apical positions to the equatorial plane deviate by only 0.033 Å. Such displacements are often observed in pentagonally bipyramidal configurations when the apical atoms are different,or are differently coordinated, numerous examples being found among transition metal peroxo complexes exhibiting this geometry with displacements ranging from 0.2 to 0.4 Å (see Table 6 in Ref. 19). In pentagonal pyramidal complexes the displacement may be as large as 0.5 Å, examples being $[CrO(O_2)_2py]$[20] and $[VO(O_2)_2(NH_3)]^-$.[21]

The Nb–F$_{\text{equatorial}}$ bond distances, 2.016–2.050 Å, are somewhat shorter than the Nb–F$_{\text{apical}}$ bond distance, 2.084 Å. This is contrary to the observations made for fluoroperoxometallates with both apical positions being occupied by fluorine atoms. In these cases the average Nb–F$_{\text{equatorial}}$ bond distances are

2.00 Å and the Nb$-$F$_{\text{apical}}$ ones are 1.92 Å. In the seven-coordinate monocapped trigonal prismatic $[NbF_7]^{2-}$ ion the Nb$-$F bonds range between 1.911 and 1.971 Å, which is normal for Nb$-$F single bond distances in seven-coordinate fluoroniobates. In $[NbF_6O]^{3-}$, the Nb$-$F$_{\text{apical}}$ bond distance, 2.084 Å, is thus considerably longer. This seems to be due to the presence of the oxygen atom, the Nb=O bond being a double bond, as can be judged from the short Nb=O bond distance of 1.738 Å. In seven-coordinate complexes with a pentagonal bipyramidal geometry and with an Me=O$_{\text{apical}}$ bond, the opposite Me$-$L$_{\text{apical}}$ bond distance is significantly longer than a normal single bond, examples being $[CrO(O_2)_2bipy]$,[22] $[CrO(O_2)_2phen]$,[23] $K_2[O\{MoO(O_2)_2(H_2O)\}_2]\cdot 2H_2O$,[24] $K_2[MoO(O_2)_2(C_2O_4)]$,[25] $(NH_4)_4[O\{VO(O_2)_2\}_2]$,[19] $K_6[Mo_7O_{22}(O_2)_2\cdot 8H_2O$,[26,27] $(NH_4)[VO(O_2)(H_2O\{C_5H_3M(COO)_2\}]\cdot xH_2O$,[28] $K_3[VO(O_2)_2(C_2O_4)]\cdot H_2O$,[29] and $K_2[O\{WO(O_2)_2(H_2O)\}_2]\cdot 2H_2O$.[30] In the six-coordinate, tetragonally bipyramidal complex $[NbF_5O]^{2-}$ the Nb$-$F$_{\text{trans}}$ (trans corresponds here to apical) bond distance is 2.06 Å in $K_2[NbF_5O]$,[31] *i.e.* about the same as in the $[NbF_6O]^{3-}$ ion (2.084 Å), while it is considerably longer, being 2.21 Å, in $(N_2H_6)[NbF_5O]\cdot H_2O$.[32] In both $(C_9H_8NO)_2[NbF_5O]\cdot 2H_2O$ [14] and Li_2NbF_5O [33] the $[NbF_5O]^{2-}$ ions are disordered, the average Nb$-$(F,O) distances being 1.92(1) Å and 1.95 Å, respectively. That the Nb$-$F$_{\text{equatorial}}$ bond distances are persistently significantly longer in the pentagonal planes, being 1.97$-$2.05 Å,[10-13] than in the tetragonal ones, lying in the range of 1.84$-$1.93 Å for a number of cases,[31,32,34] is understandable on purely geometrical grounds, the repulsive effect being larger in the more crowded pentagonal case.

The Nb=O distance in $[NbF_6O]^{3-}$ is 1.738(3) Å and compares well with the value 1.71±0.02 Å, which is the average Nb=O double-bond distance calculated from data for $(N_2H_6)[NbF_5O]\cdot H_2O$,[32] $K_2[NbF_5O]$,[31] $[NbCl_2O(OC_2H_5)(C_{10}H_8N_2)]$,[35] $[(C_6H_5)_4As]_2[Nb(NCS)_5O]$,[35] and $(NH_4)_3[Nb(C_2O_4)_3O]\cdot H_2O$.[36,37]

The observed coordination distances between the sodium and fluoride ions range from 2.221 to 2.646 Å; this is to be compared with the radii sum of 2.31 Å. The shortest Na···F interaction involves the fluorine atom with the longest coordination distance to the niobium atom. The coordination distances between the sodium atoms and the oxygen atom lie between 2.378 and 2.677 Å; in this case the radii sum is 2.35 Å.

In the system $NaF-Nb_2O_5-HF-H_2O_2-H_2O$ three distinct crystalline phases were isolated and identified by Stomberg.[10-12] They all appeared during the same crystallization experiment on total evaporation of the solvent. Their structures were determined and their formulae are $Na_2[NbF_5(O_2)]\cdot H_2O$,[10] $Na_3[HF_2][NbF_5(O_2)]$,[11] and $Na_2[NbF_5(O_2)]\cdot 2H_2O$.[12] They all contain the anion $[NbF_5(O_2)]^{2-}$. The situation might be similar in the system $NaF-Nb_2O_5-HF-H_2O$. Two phases have hitherto been observed, namely the α-phase, whose structure is described in this paper, and another phase, called the β-phase. The latter has the same cell volume as the α-phase well within experimental errors, and there is a simple ratio between the corresponding cell edges. The β-phase appeared intergrown with the α-phase with the respective axes parallel to each other. The above evidence might indicate identical stoichiometry.

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