The Crystal Structure of Sodium Pentafluoroperoxoniobate(V) Dihydrate, $Na_2[NbF_5(O_2)] \cdot 2H_2O$

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The crystals of $Na_2[NbF_5(O_2)]\cdot 2H_2O$ are ortho-rhombic, space group $C2cb$, with $a=6.890(1)$ Å, $b=10.263(2)$ Å, $c=10.361(3)$ Å, $V=732.7(3)$ Å^3 and $Z=4$. In all, 873 independent reflexions were registered on a SYNTEX single crystal X-ray diffractometer using Mo$K\alpha$ radiation. 708 reflexions were considered observed and used in the structure determination. Least-squares refinement of structural and thermal parameters yielded a final R-value of 0.035.

The pentafluoroperoxoniobate(V) ions are disordered in the crystal with two equally frequent, mutually perpendicular orientations. The anion has a pentagonal bipyramidal arrangement of ligands, the peroxo group and three fluorine atoms forming the pentagonal plane, while the other two fluorine atoms occupy the apical positions of the bipyramid. The niobium atom is situated in the pentagonal plane. The symmetry of the anion is almost C_{2v}. Average bond distances are: $Nb-O$ 1.95 Å, $O-O$ 1.46 Å and $Nb-F$ 1.95 Å.

As observed [1,2] during attempts to repeat the synthesis of the sodium fluoroperoxoniobates reported by Balke and Smith,[3] three crystallographically different compounds could be isolated, none of which had the stoichiometry given by these authors. According to crystal structure studies, they can be formulated as $Na_2[NbF_5(O_2)]\cdot H_2O$,[1,2] $Na_3[HF_2][NbF_5(O_2)]$,[1,4] and $Na_2[NbF_5(O_2)]\cdot 2H_2O$.[1] The last compound will be described in the present paper.

EXPERIMENTAL

Preparation and analysis. 0.08 mol sodium fluoride and 0.02 mol niobium(V) oxide were dissolved in 20 ml of 38 % hydrofluoric acid, and the solution was evaporated to dryness. Water was added and the solution evaporated. This procedure was repeated a couple of times in order to expel the excess of hydrofluoric acid.

The salt was then dissolved in hot water and allowed to crystallize by evaporation. A crystalline crust was obtained, which was dissolved in water, and hydrogen peroxide was added. On standing at room temperature, well-developed, bright yellow crystals were formed. After several days the crystals became opaque due to loss of water.

The sodium content was determined by atomic absorption spectroscopy using a Perkin Elmer 403 spectrometer. The peroxide content was determined by titration with a standardized permanganate solution. The amount of water was obtained by measuring the weight loss at 110 °C. (Found: Na 16.2; O_2^{2-} 10.6; H_2O 12.1. Calc. for $Na_2[NbF_5(O_2)]\cdot 2H_2O$: Na 15.23; O_2^{2-} 10.60; H_2O 11.93.)

The compound was investigated thermoanalytically up to 300 °C, using the universal microthermobalance Mettler TA1. The DTA curve indicated an endothermic reaction starting at room temperature with a maximum heat absorption at 105 °C and an exothermic one starting at 240 °C with a maximum heat evolution at 260 °C. The endothermic reaction was taken to be due to loss of water of crystallization (the TGA curve flattened out between 115 and 240 °C) and the exothermic one to loss of oxygen. (Mass loss found: H_2O 12.0; O_2 5.0. Calc. for $Na_2[NbF_5(O_2)]\cdot 2H_2O$: H_2O 11.93; O_2 5.30.)

An approximately 0.01 M solution of Na_2-$[NbF_5(O_2)]\cdot 2H_2O$ was titrated with 0.2000 M sodium hydroxide using a Radiometer PHM 64 pH meter. Neutralization required 3.86 mol sodium hydroxide per mol niobium.

X-Ray methods. X-Ray powder photographs were obtained by the Guinier-Hägg method using Cu$K\alpha$ radiation. $Pb(NO_3)_2$ ($a=7.8566$ Å at 21 °C) was used as an internal standard. Observed lines in the powder photograph are given in Table 1.

Table 1. Observed lines in the powder photograph of $Na_2[NbF_5(O_2)]\cdot 2H_2O$ at room temperature registered with $CuK\alpha$ radiation, using lead nitrate ($a = 7.8566$ Å) as internal standard.

d_o	d_c	h k l	I_o	d_o	d_c	h k l	I_o
5.172	5.173	0 0 2	m	1.6308	1.6310	4 2 0	m
5.126	5.124	0 2 0	m	1.6209	1.6220	0 6 2	m
5.002	5.001	1 1 1	s	1.6088	1.6090	2 4 4	m
4.589	4.592	0 2 1	w	1.5554	1.5555	4 2 2	m
3.834	3.835	1 1 2	w	1.5412	1.5416	2 0 6	m
3.640	3.640	0 2 2	s	1.5279	1.5291	2 6 0	m
3.440	3.441	2 0 0	m	1.5193	1.5195	3 1 5	m
2.9523	2.9525	1 1 3	vs	1.5114	1.5120	3 5 1	m
2.9332	2.9342	1 3 1	s	1.4756	1.4763	2 2 6	w
2.8642	2.8650	2 0 2	m	1.4666	1.4671	2 6 2	w
2.8583	{2.8610 / 2.8567}	{0 2 3 / 2 2 0}	m	1.4312	1.4309	1 1 7	w
				1.4284	1.4284	4 4 0	w
2.7527	2.7537	2 2 1	w	1.4242	1.4247	1 5 5	m
2.6319	2.6336	1 3 2	w	1.4184	1.4185	1 7 1	vw
2.5854	2.5866	0 0 4	m	1.4013	1.4013	3 3 5	w
2.5618	2.5621	0 4 0	m	1.3971	1.3974	3 5 3	w
2.4993	2.5007	2 2 2	w	1.3767	1.3768	4 4 2	w
2.3564	2.3563	1 1 4	vvw	1.3524	1.3525	5 1 1	w
2.3084	2.3090	0 2 4	m	1.3222	1.3225	1 7 3	vw
2.2954	2.2959	0 4 2	m	1.3207	1.3209	2 4 6	m
2.2002	2.2000	2 2 3	vvw	1.3165	1.3168	2 6 4	m
2.1870	2.1880	3 1 1	vs	1.2930	1.2933	0 0 8	w
2.0678	2.0675	2 0 4	vw	1.2808	1.2811	0 8 0	w
2.0540	{2.0551 / 2.0545}	{2 4 0 / 3 1 2}	w	1.2682	1.2685	5 1 3	w
				1.2669	1.2671	5 3 1	w
2.0170	2.0156	2 4 1	vvw	1.2335	1.2334	3 1 7	vw
1.9450	1.9456	1 1 5	s	1.2294	1.2294	3 5 5	w
1.9170	1.9174	2 2 4	m	1.2257	1.2255	3 7 1	vw
1.9088	1.9099	2 4 2	m	1.2183	1.2180	4 0 6	vw
1.8769	1.8777	3 1 3	s	1.2136	1.2135	0 6 6	vw
1.8722	1.8730	3 3 1	m	1.2121	1.2122	4 6 0	vw
1.8361	1.8365	1 5 2	vw	1.2104	1.2106	2 0 8	vvw
1.8203	1.8203	0 4 4	m	1.1853	1.1849	4 2 6	m
1.7871	1.7871	3 3 2	vvw	1.1810	1.1811	1 5 7	m
1.7243	1.7244	0 0 6	m	1.1776	1.1775	1 7 5	m
1.7198	1.7205	4 0 0	m	1.1697	1.1695	2 8 2	vvw
1.7136	1.7141	1 3 5	m	1.1622	1.1620	3 7 3	m
1.7068	{1.7081 / 1.7070}	{0 6 0 / 1 5 3}	m	1.1546	1.1545	0 4 8	vvw
				1.1479	1.1480	0 8 4	vvw
1.6919	1.6927	3 1 4	vvw	1.1450	1.1448	5 3 4	m
1.6843	1.6853	0 6 1	vvw	1.1395	1.1389	5 1 5	m
1.6506	1.6508	1 1 6	vvw	1.1179	1.1177	0 6 7	vw
1.6335	1.6343	0 2 6	m	1.1013	1.1017	1 7 6	vw

For the single crystal work, reflexion intensities were measured with a SYNTEX $P2_1$ automatic four-circle diffractometer at room temperature. A crystal with the dimensions $0.35 \times 0.35 \times 0.20$ mm was used. The X-ray intensities were measured by the $\omega - 2\theta$ scan technique, using graphite-mono-chromatized $MoK\alpha$ radiation ($2\theta \leq 70°$). One reflexion measured after each twenty-sixth reflexion showed no significant difference in intensity during the data collection. The 2θ scan speed varied between 1.5 and 6°/min. A profile analysis based on the Lehmann-Larsen method [5] was applied to the 96-

step profile collected for each reflexion. A total of 873 independent reflexions were measured. Of these, 708, for which $I_0 \geq 3\sigma(I_0)$, were regarded as being observed and were used in the subsequent calculations. The intensities were corrected for Lorentz, polarization, absorption and extinction effects. The unit cell parameters were determined from a least-squares fit of refined diffractometer setting angles for 15 reflexions.

Computing methods. A set of crystallographic programs described in Ref. 6 was used. The atomic scattering factors for Na$^+$, Nb, F and O were taken from the *International Tables for X-Ray Crystallography, Vol. III*, as was the dispersion correction applied to Nb.

CRYSTAL DATA

Sodium pentafluoroperoxoniobate(V) dihydrate, Na$_2$[NbF$_5$(O$_2$)]·2H$_2$O, $M_r = 301.92$.

Systematic absences hkl for $h + k = 2n + 1$, $h0l$ for
$\qquad\qquad\qquad l = 2n + 1$, $hk0$ for $k = 2n + 1$
Space group $C2cb$ (No. 41; non-standard setting)
General positions $(000, \frac{1}{2}\frac{1}{2}0) +$
$\qquad (x,y,z)$, $(x,\bar{y},\bar{z})$, $(x,\frac{1}{2} + y,\frac{1}{2} - z)$,
$\qquad (x,\frac{1}{2} - y,\frac{1}{2} + z)$
$a = 6.890(1)$ Å, $b = 10.263(2)$ Å, $c = 10.361(3)$ Å,
$V = 732.7(3)$ Å^3, $Z = 4$, $D_c = 2.74$ g cm^{-3},
$\mu(\text{Mo}K\alpha) = 18.1$ cm^{-1}.

A list of observed and calculated structure factors is available from the author upon request.

STRUCTURE DETERMINATION

The main features of the structure were originally obtained from film data by applying Patterson and successive electron density calculations.[7] It became obvious that a peak at (0.3,0,0), extending in both the y and z directions as observed in the Patterson function as well as on the electron density map, could be interpreted as a peroxo group statistically oriented in the y and z directions. With diffractometer data this disorder was more carefully interpreted. Moreover, a model with the complex and its mirror image in the yz-plane was tried. The results from the refinement of occupation numbers for the peroxo group and the fluorine atoms indicated disorder only in the orientation of the peroxo group. The atomic arrangement indicates a pseudo tetragonal structure, also revealed by the observation that $|F(hkl)|$ approximately equals $|F(hlk)|$ for a large number of reflexions.

The final cycles of refinement were performed with a full-matrix least-squares program, anisotropic thermal parameters being included for all non-hydrogen atoms. The observed structure factors were weighted according to Cruickshank,[8] $w = (a + |F_o| + c|F_o|^2 + d|F_o|^3)^{-1}$, with $a = 10.0$, $c = 0.02$ and $d = 0.0$. A final R-value of 0.035 was obtained. Atomic coordinates are listed in Table 2. A final electron density difference map showed no anomalies.
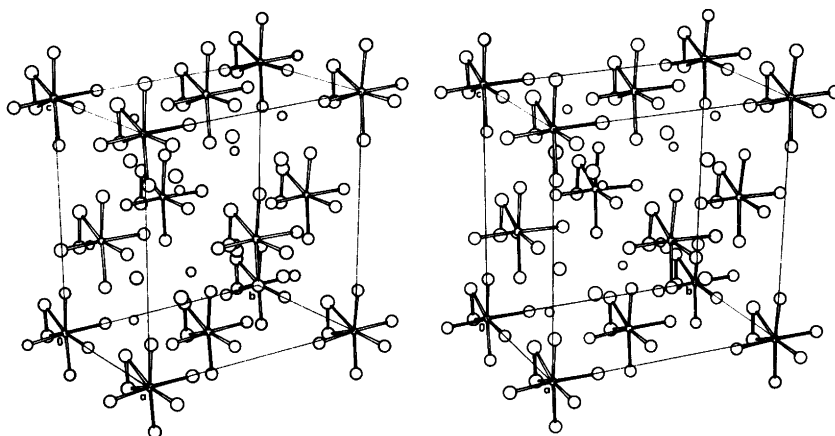

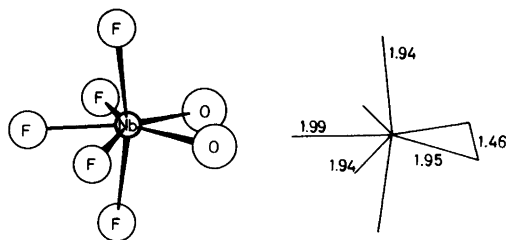*Fig. 1.* Stereoscopic drawing of the unit cell of Na$_2$[NbF$_5$(O$_2$)]·2H$_2$O.

Fig. 2. The ion $[NbF_5(O_2)]^{2-}$.

RESULTS AND DISCUSSION

The content of the unit cell is shown in Fig. 1 and the anion in Fig. 2. Bond distances and angles are given in Table 3 and coordination distances to the sodium ion in Table 4.

The crystals of sodium pentafluoroperoxoniobate-(V) dihydrate consist of sodium ions, pentafluoro-peroxoniobate(V) ions and water of crystallization. There are four formula units in the crystallographic

cell. The crystal structure is disordered; the anions have one or the other of two equally frequent, mutually perpendicular orientations (by rotating the anion from one position through 90° about the a-axis, the other orientation is obtained). In Fig. 1 only one of the two observed orientations of the anions is depicted.

In $Na_2[NbF_5(O_2)] \cdot 2H_2O$ the $[NbF_5(O_2)]^{2-}$ ion has the pentagonal bipyramidal arrangement of ligands observed in $Na_2[NbF_5(O_2)] \cdot H_2O$,[2] $Na_3[HF_2][NbF_5(O_2)]$,[4] $K_3[HF_2][TaF_5(O_2)]$[9,22] and $K_6[TaF_5(O_2)][O\{TaF_4(O_2)\}_2] \cdot H_2O$[10] as well as in several other transition metal peroxo complexes (see, *e.g.*, Refs. 11–13). The consistency of this geometry for the pentafluoromonoperoxometallates is evident from Table 5, listing interatomic distances in some compounds of this type. This also supports the interpretation of the disordered structure of $(NH_4)_3[TiF_5(O_2)]$ made by Stomberg *et al.*[14] The two crystallographically different anions are denoted I and II, respectively. I consists of Nb, F1, F2, F1′,

Table 2a. Atomic coordinates, expressed as fractions of the cell edges, for $Na_2[NbF_5(O_2)] \cdot 2H_2O$. Space group $C2cb$, $Z=4$. The numbers in parentheses are the standard deviations. The extinction coefficient is $0.60(3) \times 10^4$.

Atom	Position	x	y	z
Nb	4a	0 ^{a}	0	0
Na	8b	0.0070 (18)	0.1345 (2)	0.3606 (2)
F1	8b	0.0292 (13)	−0.0022 (3)	0.1858 (4)
F2	4a	0.2884 (10)	0	0
F3	8b	0.0183 (17)	0.1884 (3)	0.0047 (2)
O1	8b ^{b}	−0.2584 (17)	−0.0039 (8)	0.0726 (13)
O2	8b ^{b}	−0.2657 (20)	0.0687 (15)	−0.0024 (9)
O3	8b	0.2499 (26)	0.2581 (4)	0.2411 (10)

^{a} Defines origo. ^{b} Half-filled.

Table 2b. Anisotropic thermal parameters U_{ij} for $Na_2[NbF_5(O_2)] \cdot 2H_2O$. The expression used is $\exp[-2\pi^2(U_{11}h^2a^{*2} + \cdots + 2U_{23}klb^*c^*)]$.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Nb	0.0105(2)	0.0129(2)	0.0118(2)	0	0	−0.0010(1)
Na	0.043(1)	0.032(1)	0.030(1)	−0.002(3)	0.002(3)	−0.007(1)
F1	0.076(6)	0.031(2)	0.015(1)	0.012(3)	−0.007(3)	−0.002(1)
F2	0.012(3)	0.045(5)	0.073(7)	0	0	−0.027(4)
F3	0.073(4)	0.016(1)	0.027(2)	−0.008(4)	−0.003(3)	0.001(1)
O1	0.016(4)	0.035(5)	0.038(5)	0.001(4)	0.007(4)	−0.006(4)
O2	0.021(5)	0.047(7)	0.045(7)	0.012(5)	0.002(4)	−0.004(4)
O3	0.047(3)	0.033(3)	0.060(4)	−0.012(4)	0.025(3)	−0.012(3)

Table 3. Bond distances and angles in Na_2-$[NbF_5(O_2)] \cdot 2H_2O$. A prime indicates a symmetry related atom within the anion. O1 and O2 belong to different anions.

	Distance/Å			Angle/°
Nb — F1	1.936 (4)	F1 — Nb — F1′		168.1 (5)
F2	1.987 (7)		F2	84.0 (3)
F3	1.938 (4)		F3	88.9 (1)
O1	1.933 (12)		F3′	90.4 (1)
O2	1.962 (14)		O1	73.1 (5)
O1 — O1′	1.506 (26)		O1′	118.9 (5)
O2 — O2′	1.411 (31)		O2	96.5 (4)
			O2′	94.6 (4)
		F2 — Nb — F3		86.3 (3)
			O1	157.1 (4)
			O2	158.9 (4)
		F3 — Nb — F3′		172.5 (7)
			O1	94.1 (4)
			O1′	92.8 (4)
			O2	72.7 (6)
			O2′	114.8 (6)
		O1 — Nb — O1′		45.8 (8)
		O2 — Nb — O2′		42.2 (9)

O1′, O1, F3 and F3′ and II of Nb, F3, F2, F3′, O2′, O2, F1 and F1′. The peroxo oxygen atoms O1 and O2 are well resolved, while the positions of the fluorine atoms, except F2 which besides Nb lies on the two-fold axis, are by necessity merely average ones. This is reflected in the observed bond distances and angles involving the fluorine atoms as well as larger thermal motions perpendicular to the Nb — F bond directions.

The distances from the least-squares plane A through F1, F2, F1′, O1′ and O1 to F1, O1 and F3 in I are -0.011, 0.027 and -1.934 Å, respectively. Due to symmetry F2 and Nb lie in this plane. The

Table 4. Coordination distances to the sodium ions. O1 and O2 are the two alternative peroxide oxygen atoms.

	Distance/Å
Na···O2($\frac{1}{2}+x, y, \frac{1}{2}-z$)	2.251 (15)
O1($\frac{1}{2}+x, y, \frac{1}{2}-z$)	2.260 (14)
F1(x, y, z)	2.296 (4)
F3($x, \frac{1}{2}-y, \frac{1}{2}+z$)	2.354 (4)
O3($-\frac{1}{2}+x, y, \frac{1}{2}-z$)	2.420 (17)
O3(x, y, z)	2.439 (16)
F2($-\frac{1}{2}+x, y, \frac{1}{2}-z$)	2.502 (9)

distances from the corresponding least-squares plane B in II through F3, F2, F3′, O2′ and O2 to F3, O2 and F1 are -0.014, 0.038 and -1.925 Å, respectively. F2 and Nb lie in this plane also. The plane through F2, F3, F3′ and the midpoint between O1 and O1′ makes an angle of 89.5° with plane A. The corresponding plane through F2, F1, F1′ and the midpoint between O2 and O2′ makes an angle of 89.6° with plane B. Hence, the point symmetry of the anion is C_{2v} within the experimental error.

In $Na_2[NbF_5(O_2)] \cdot H_2O^2$ and $Na_3[HF_2]$-$[NbF_5(O_2)]^4$ the $Nb - F_{equatorial}$ bond distances $1.976 - 2.037$ Å and $1.975 - 2.019$ Å are somewhat longer than the $Nb - F_{apical}$ ones, being $1.906 - 1.938$ Å and $1.906 - 1.936$ Å, respectively. In $Na_2[NbF_5(O_2)] \cdot 2H_2O$ the equatorial plane takes one or the other of two equally frequent, mutually perpendicular directions. Only one of the fluorine atoms, F2, is situated in the pentagonal plane in either orientation; this $Nb - F_{equatorial}$ bond distance is 1.987 Å, in harmony with the observations in the other two fluoroperoxoniobates mentioned above. The other fluorine atoms, F1, F1′ and F3, F3′, respectively, are apically situated in one half of the complexes in the crystal and equatorially in the other half. The observed Nb — F distances, 1.936 and 1.938 Å, respectively, are therefore only average values. A possible explanation to the differences between the $Nb - F_{apical}$ and $Nb - F_{equatorial}$ bond distances is given in Ref. 2.

The Nb — O distances, 1.933 and 1.962 Å, are normal Nb — O single bond values, observed distances in $Na_2[NbF_5(O_2)] \cdot H_2O$ being 1.928 Å, in $Na_3[HF_2][NbF_5(O_2)]$ 1.924 and 1.940 Å and in $(C_9H_8NO)_2[NbF_5(O_2)] \cdot 3H_2O$ $1.91 - 1.95$ Å.[2,4,13] Additional Nb — F and Nb — O distances can be found in Ref. 15 and references therein.

The average distance between the oxygen atoms in the peroxo groups, 1.46 Å, compares well with other observations. In $Na_2[NbF_5(O_2)] \cdot H_2O$ and $Na_3[HF_2][NbF_5(O_2)]$ the O — O distances are 1.476(7) and 1.481(4) (for further details see Table 7 in Ref. 16). The large standard deviation, 0.03 Å, in the two observed O — O distances in the present work is due to the disorder.

The sodium ion is surrounded by seven close neighbours. The observed coordination distances between the sodium ion and the fluorine and oxygen atoms range between 2.251 and 2.502 Å, which are quite normal values.

The shortest sodium to water oxygen distance is 2.420 Å, which is a normal value (see, *e.g.*, Refs. 17

Table 5. Comparison between the lengths of the edges (in Å) of the pentagonal bipyramidal coordination polyhedron in some pentafluoromono-peroxometallates. The letters refer to the edges of the polyhedron as defined in Fig. 3.

Edge	$Na_2[NbF_5(O_2)]\cdot H_2O$ Ref. 2	$Na_3[HF_2][NbF_5(O_2)]$ Ref. 4	$Na_2[NbF_5(O_2)]\cdot 2H_2O$ this paper[a] I	II	$K_3[HF_2][TaF_5(O_2)]$ Ref. 9	$Na_2[NbF_5(O_2)]$ Ref. 22[b]	$K_6[TaF_5(O_2)]-[O\{TaF_4(O_2)\}_2]\cdot H_2O$ Ref. 10
a	2.527(4)	2.531(3)	2.63(1)	2.68(1)	2.6(1)	2.52(2)	2.48(1)
b	2.527(4)	2.533(3)	2.63(1)	2.68(1)	2.41(1)	2.52(2)	2.48(1)
c	2.496(4)	2.486(3)	2.30(2)	2.31(2)	2.6(2)	2.47(2)	2.51(2)
d	1.476(7)	1.481(4)	1.51(3)	1.41(3)	1.6(2)	1.48(3)	1.39(2)
e	2.496(5)	2.496(3)	2.30(2)	2.31(2)	2.1(1)	2.47(2)	2.51(2)
f	2.696(4)	2.640(3)	2.75(1)	2.71(1)	3.1(1)	2.69(2)	2.65(1)
g	2.644(6)	2.632(3)	2.68(1)	2.63(1)	2.6(1)	2.63(2)	2.56(2)
h	2.696(4)	2.853(3)	2.71(1)	2.75(1)	2.3(1)	2.69(2)	2.65(1)
i	2.905(6)	2.792(3)	2.83(1)	2.86(1)	3.1(1)	2.91(2)	2.88(2)
j	2.905(6)	2.791(3)	2.80(1)	2.91(1)	3.2(1)	2.91(2)	2.88(2)
k	2.744(4)	2.755(3)	2.71(1)	2.75(1)	2.4(1)	2.71(2)	2.76(2)
l	2.588(5)	2.680(3)	2.68(1)	2.63(1)	2.5(1)	2.58(2)	2.69(2)
m	2.744(4)	2.686(3)	2.75(1)	2.71(1)	2.9(1)	2.71(2)	2.76(2)
n	2.851(6)	2.922(3)	2.80(1)	2.91(1)	2.8(1)	2.81(2)	2.81(2)
o	2.851(6)	2.843(3)	2.83(1)	2.86(1)	2.7(1)	2.81(2)	2.81(2)

[a] The structure is disordered. [b] Preliminary values due to a reinvestigation of the structure at -100 °C with a SYNTEX $P2_1$ diffractometer.[22]
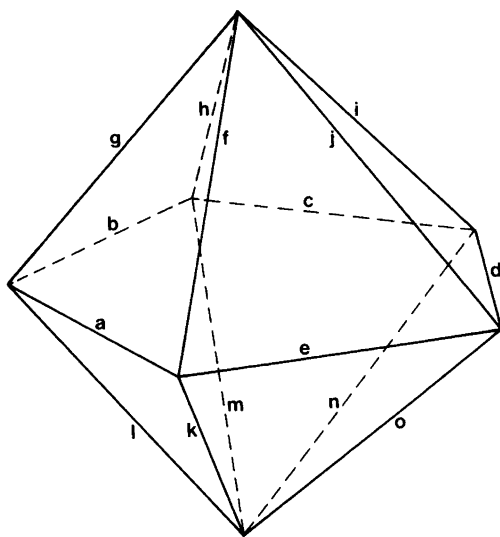

Fig. 3. Coordination polyhedron of pentafluoro-monoperoxometallates (see Table 5).

and 18). The shortest distance between the water oxygen and the neighbouring fluorine and oxygen atoms is 2.99 Å, indicating no significant hydrogen bonding.

A 0.01 M water solution of $Na_2[NbF_5(O_2)]\cdot 2H_2O$ has a pH value of about 3. The pentafluoroperoxo-niobate(V) ion is therefore appreciably hydrolyzed. Titration of such a solution with standardized sodium hydroxide indicated that 4 mol OH^- per mol Nb was required to reach the equivalence point. Sieverts *et al.* have found that if a concentrated solution of potassium tetraperoxoniobate(V) is titrated with an amount of sulfuric acid equivalent to the potassium content of the salt, precipitation of the 'peracid' $HNbO_4\cdot xH_2O$ will occur.[19] This may be formulated

$$[Nb(O_2)_4]^{3-} + 3H^+ + (2+x)H_2O \rightarrow$$
$$HNbO_4\cdot xH_2O + 3H_2O_2$$
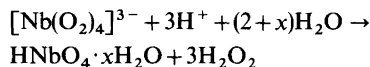
Moreover, the potassium salt $KNbO_4\cdot 7H_2O$ of this peroxoacid has been reported by Grigoréva *et al.*;[20] the compound has also been studied spectrophotometrically by Griffith.[21] In view of these observations the monoperoxoniobate(V) ion may be formulated $NbO_2(O_2)^-$, neglecting co-ordinated water molecules. The neutralization of the pentafluoroperoxoniobate(V) ion is therefore in accordance with the reaction
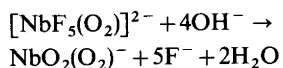
$$[NbF_5(O_2)]^{2-} + 4OH^- \rightarrow$$
$$NbO_2(O_2)^- + 5F^- + 2H_2O$$

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