

# The Crystal Structure of Potassium Fluorooxidiperoxovanadate(V), $K_2[VF_2O(O_2)_2]$

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The crystal structure of potassium fluorooxidiperoxovanadate(V),  $K_2[VF_2O(O_2)_2]$ , has been determined at room temperature by single-crystal X-ray methods. The compound crystallizes in the orthorhombic space group  $P2_12_12_1$  (No. 19) with  $a=6.180(1)$ ,  $b=11.054(3)$ ,  $c=7.995(2)$  Å,  $V=546.2(2)$  Å<sup>3</sup> and  $Z=4$ . The structure was solved by Patterson and Fourier methods and least-squares refinement gave a final  $R(F)$ -value of 0.031 for 1636 observed independent reflexions.

The ligands form a pentagonal pyramid around vanadium, the configuration being similar to that of  $[CrO(O_2)_2py]$ . The anions are disordered in the crystal and have two main orientations with occupation numbers 0.60(2) and 0.40(2), respectively. The positions of the vanadium atom in the differently orientated anions are identical within the limits of experimental errors. The apical oxygen atom positions differ by 0.26(1) Å causing the pentagonal planes to form an angle of 5.6° with each other. These planes are mutually twisted 126°.

Bond distances within the complex anions are:  $V=O$  1.620(5) and 1.605(7) Å,  $V-O_{\text{peroxo}}$  1.858(7)–1.930(5) Å,  $V-F$  1.906(3) and 1.900(5) Å and  $(O-O)_{\text{peroxo}}$  1.468(7)–1.505(6) Å.

Peroxovanadates have been studied kinetically (see Refs. 1–12 and references therein), thermochemically,<sup>13–15</sup> spectroscopically,<sup>16–24</sup> and, to a lesser extent, owing to their instability, structurally. Stabilization can be achieved by certain ligands which makes the corresponding peroxovanadate isolable and suitable for structural studies by crystallographic methods. Hitherto the structures of the following peroxovanadates

have been established:  $(NH_4)_4[O\{VO(O_2)_2\}_2]$ ,<sup>25</sup>  $NH_4[VO(O_2)_2(NH_3)]$ ,<sup>26</sup>  $(NH_4)_3[VO(O_2)_4]$ ,<sup>27</sup>  $NH_4[VO(O_2)(H_2O)dipic] \cdot xH_2O$ ,<sup>28</sup>  $K_3[VO(O_2)_2ox] \cdot H_2O$ ,<sup>29</sup> and, recently,  $NH_4[VO(O_2)_2bipy] \cdot 4H_2O$ ,<sup>30,31</sup>  $[VO(O_2)_2(pic)(bipy)] \cdot H_2O$ ,<sup>32</sup>  $(Hbipy)[H\{VO(O_2)_2bipy\}_2] \cdot xH_2O \cdot (6-x)H_2O$ ,<sup>33</sup> and  $(Hbipy)[VO(O_2)_2bipy] \cdot (3+x)H_2O \cdot (2-x)H_2O$ .<sup>34</sup> Thus, the structures of few peroxovanadates with purely inorganic ligands have been determined. Recently Jere *et al.* synthesized a new potassium fluoroperoxovanadate,  $K_2[VF_2O(O_2)_2]$ ,<sup>35</sup> and Chaudhuri *et al.* this and other alkali metals salts with the anion  $[VF_2O(O_2)_2]^{2-}$ .<sup>36</sup> It was, therefore, thought worthwhile to perform a structural investigation on the potassium salt, which we have found to be remarkably stable. It does not undergo any visible change for months at room temperature and not even for hours at 105 °C. For comparison we have undertaken synthetic and structural work on the ammonium fluoroperoxovanadate system. While the potassium salt can be kept in contact with its mother liquor for a very long time without change, the ammonium salt usually undergoes conversion within a few days. Apart from  $(NH_4)_2[VF_2O(O_2)_2]$ , one completely new compound, the seven-coordinated  $(NH_4)_3[VF_2O(O_2)_2]$ , was observed; it appears as light-yellow crystals. The crystal structure of the potassium salt is reported in this paper while those of  $(NH_4)_2[VF_2O(O_2)_2]$ <sup>37</sup> and  $(NH_4)_3[VF_2O(O_2)_2]$ <sup>38</sup> will be presented later.

## EXPERIMENTAL

**Preparation.** 0.46 g vanadium(V) oxide was mixed with 2.9 g potassium fluoride and the mixture was dissolved in 15 ml 15 % hydrogen peroxide. Unlike the procedure of Jere *et al.*<sup>35</sup> the solution was not warmed. After 15–18 h at room temperature light-yellow crystals were obtained. In most preparations they were almost hexagonal plates; these were multiple twins but single crystals could be cut out. The vanadium content was checked by precipitation as  $\text{AgVO}_3$  and the amount of peroxide by titration with a standardized potassium permanganate solution. (Found: V 22.4;  $\text{O}_2^-$  27.7. Calc. for  $\text{K}_2[\text{VFO}(\text{O}_2)_2]$ : V 22.33;  $\text{O}_2^-$  28.05.)

**X-Ray methods.** Intensity data were recorded at 18 °C with a SYNTEX P2<sub>1</sub> automatic four-circle single-crystal X-ray diffractometer using graphite-monochromatized  $\text{MoK}\alpha$  radiation and a single crystal with the dimensions  $0.16 \times 0.16 \times 0.19$  mm. The  $\omega$ - $2\theta$  scan method was used and the  $2\theta$  scan speed was allowed to vary between 2.3 and  $20^\circ \text{min}^{-1}$  depending on the intensity of the measured reflexion. Data were collected for  $2\theta \leq 80^\circ$ . A profile analysis based on the Lehmann-Larsen method<sup>39</sup> was applied to the 96-step profile collected for each reflexion. Two test reflexions measured after each forty-eighth reflexion showed no significant difference in intensity during the collection of the data.

A total of 1977 independent reflexions were measured. Of these, 1636 having  $I \geq 3\sigma(I)$  were used in the subsequent calculations. Corrections were made for Lorentz and polarization effects but not for absorption.

The unit cell parameters were determined from a least-squares fit of refined diffractometer setting angles for 15 reflexions.

Due to both the twinning and the disorder observed during the structure analysis, data were collected up to  $2\theta = 60^\circ$  for a second crystal obtained from a different preparation.

## CRYSTAL DATA

$\text{K}_2[\text{VFO}(\text{O}_2)_2]$  F.W. = 228.13

Space group  $P2_12_12_1$  (No. 19)

$a = 6.180(1) \text{ \AA}$ ,  $b = 11.054(3) \text{ \AA}$ ,  $c = 7.995(2) \text{ \AA}$ ,  
 $V = 546.2(2) \text{ \AA}^3$ ,  $Z = 4$ ,  $D_o = 2.74 \text{ g cm}^{-3}$ ,  $D_c = 2.77 \text{ g cm}^{-3}$ ,  $\mu(\text{MoK}\alpha) = 3.4 \text{ mm}^{-1}$ .

## STRUCTURE DETERMINATION

The structure was solved by Patterson and electron density calculations and refined by the least-squares method. During the structure analysis it soon became evident that the anion was disordered, indicated by an extra peak with half the height of an oxygen peak, unacceptably short peroxo oxygen distances and rather high thermal parameters for certain atoms. The last mentioned observation suggests unresolved disorder, in this case of the magnitude  $0.2 \text{ \AA}$ . A model with two orientations of the anion could be successfully refined. Full-matrix refinement of positional and anisotropic thermal parameters as well as occupation numbers for the fluorine and oxygen atoms reduced the  $R$ -value to 0.031 ( $R = \sum ||F_o| - |F_c|| / \sum |F_o|$ ). The weighting scheme used was that of Cruickshank:<sup>40</sup>  $w = (a + |F_o| + c|F_o|^2 + d|F_o|^3)^{-1}$  with  $a = 30$ ,  $c = 0.01$  and  $d = 0.002$ . The scattering factors for V, K, F, and O were taken from Ref. 41 as were the dispersion corrections.

An electron density difference synthesis calculated after the final cycle of refinement showed no peak higher than  $0.47 \text{ e \AA}^{-3}$ .

Calculations were carried out on an IBM 3033 computer using the crystallographic programmes described in Ref. 42.

Lists of the structure factors and thermal parameters are available from the author on request.

## RESULTS AND DISCUSSION

The positional parameters, obtained in the last cycle of refinement, as well as  $U_{eq}$  are given in Table 1. 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 2 and coordination distances to the potassium ions in Table 3.

The crystals of  $\text{K}_2[\text{VFO}(\text{O}_2)_2]$  consist of potassium ions and fluorooxidiperoxovanadate(V), ions, held together by ionic forces. The anions are disorderly orientated in the crystals. Two main orientations, denoted A and B, with occupancies 0.60(2) and 0.40(2), respectively, were observed. The two anions have the same coordination geometry, a pentagonal pyramidal orientation of ligands of the type first met with for  $[\text{CrO}(\text{O}_2)_2\text{py}]$  by the author,<sup>43</sup> and also observed for  $\text{NH}_4[\text{VO}(\text{O}_2)_2\text{NH}_3]$ .<sup>26</sup> This is a

Table 1. Atomic fractional coordinates for  $K_2[VFO(O_2)_2]$ . Atoms marked with A and B have occupation numbers 0.60(2) and 0.40(2), respectively.  $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$ .

Atom	x	y	z	$U_{eq}/\text{\AA}^2$
V	0.27055(8)	0.17315(4)	0.21635(5)	0.01546(6)
K1	0.23232(13)	0.46890(6)	-0.00565(8)	0.0238(1)
K2	0.74384(17)	0.34726(8)	0.32550(12)	0.0344(2)
F(A)	0.2846(5)	0.3392(3)	0.2795(4)	0.0275(6)
O1(A)	0.4014(9)	0.2235(4)	0.0120(7)	0.0300(9)
O2(A)	0.3032(7)	0.1026(5)	0.0035(6)	0.0325(9)
O3(A)	0.0406(7)	0.0632(4)	0.2398(5)	0.0276(7)
O4(A)	0.0263(6)	0.1673(4)	0.3606(4)	0.0331(7)
O5(A)	0.4645(8)	0.1159(4)	0.3274(6)	0.0261(7)
F(B)	0.0152(8)	0.0859(4)	0.2711(7)	0.0283(9)
O1(B)	0.1614(10)	0.2880(6)	0.3694(7)	0.0334(11)
O2(B)	0.3395(7)	0.3400(5)	0.2638(7)	0.0209(9)
O3(B)	0.3882(13)	0.2030(5)	0.0031(8)	0.0253(11)
O4(B)	0.2450(11)	0.0965(6)	0.0105(9)	0.0307(12)
O5(B)	0.4614(12)	0.0985(6)	0.3052(9)	0.0280(12)

rather unusual geometry, most peroxometallates being seven-coordinated pentagonal bipyramids (see, e.g., Refs. 25, 30, 32–34, 44–48 and refs. therein). The equatorial planes are defined by the atoms O1–O4 and F; the maximum deviation from the respective plane is 0.039 Å in anion A and 0.051 Å in anion B, while the r.m.s. deviations are 0.027 and 0.034 Å, respectively. The plane defined by F, O5 and the midpoint between O2 and O3 form an angle of 90.6° with the equatorial plane in A and of 87.4° in B.

The disorder of the anions A and B can be described in the following way. The central atom position of A could not be distinguished from that of B. The equatorial pentagonal planes form an angle of 5.6° with one another, and the pentagons are mutually twisted 126° (Fig. 3). As found by the author disordered fluoroperoxometallates are not uncommon. The same type of disorder was observed for the anion in  $(C_{12}H_{10}N_2)[NbF_5(O_2)]$ ,<sup>48</sup> but is different from that met with in  $Na_2[NbF_5(O_2)] \cdot 2H_2O$ ,<sup>47</sup>  $(NH_4)_3[TiF_5(O_2)]$ ,<sup>49, 50</sup> and  $(C_9H_8NO)_2[NbF_5(O_2)] \cdot 3H_2O$ .<sup>51</sup>

The equatorial V–O and V–F distances indicate them to be normal single bonds, lying in the range 1.858(7)–1.930(5) Å, while the V=O<sub>apical</sub> bonds are double bonds, being 1.620(5) and 1.605(7) Å (see Table 5 in Ref. 32). The peroxo oxygen bond distances, 1.468(7)–1.505(6) Å, compare well with other observations (see Table

7 in Ref. 25, Table 5 in Ref. 47 and Table 5 in Ref. 32).

Table 2. Bond distances (Å) and angles (°) in  $K_2[VFO(O_2)_2]$ .

	A	B
V–F	1.906(3)	1.900(5)
V–O1	1.906(6)	1.888(6)
V–O2	1.882(5)	1.930(5)
V–O3	1.880(4)	1.882(7)
V–O4	1.901(4)	1.858(7)
V–O5	1.620(5)	1.605(7)
O1–O2	1.468(7)	1.501(8)
O3–O4	1.505(6)	1.474(9)
F–V–O1	85.8(2)	84.0(2)
F–V–O2	129.3(2)	128.5(2)
F–V–O3	129.1(2)	128.2(3)
F–V–O4	84.7(2)	84.4(3)
F–V–O5	101.4(2)	104.3(3)
O1–V–O2	45.6(2)	46.3(2)
O1–V–O3	126.5(2)	127.4(3)
O1–V–O4	150.1(2)	148.3(3)
O1–V–O5	105.6(2)	108.8(3)
O2–V–O3	84.5(2)	85.7(2)
O2–V–O4	128.3(2)	129.0(3)
O2–V–O5	104.7(2)	104.0(3)
O3–V–O4	46.9(2)	46.4(3)
O3–V–O5	104.6(2)	102.0(3)
O4–V–O5	104.0(2)	102.7(3)

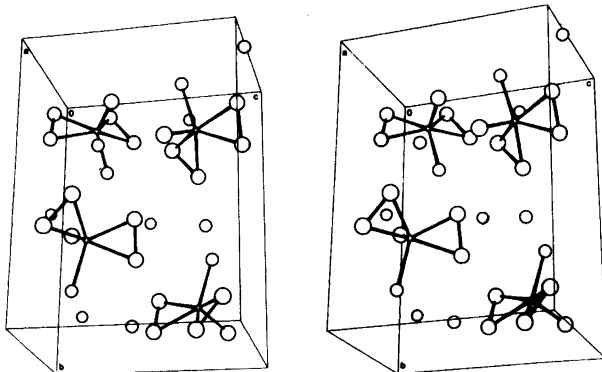


Fig. 1. Stereoscopic drawing of the unit cell of  $K_2[VFO(O_2)_2]$  with the anions in orientation A.

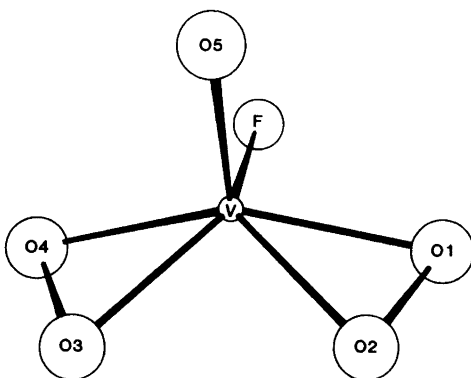


Fig. 2. The anion  $[VFO(O_2)_2]^{2-}$ .

The vanadium atom is displaced 0.457 and 0.471 Å from the respective equatorial planes towards the apical oxygen atom. These are close to the values 0.46 and 0.51 Å observed for  $NH_4[VO(O_2)_2NH_3]^{26}$  and the type structure  $[CrO(O_2)_2py]^{43}$  respectively. In seven-coordinated pentagonal bipyramidal peroxometallates with one  $M=O_{apical}$  and one  $M-X_{apical}$  bond the displacement of the metal atom from the equatorial plane is less, being 0.18–0.43 Å (see Table 5 in Ref. 32 and Table 6 in Ref. 25), while, as expected, there is hardly any displacement with two identical  $M-X_{apical}$  bonds (see Table 6 in Ref. 25 and Refs. 45–48).

The potassium ions are surrounded by seven to

Table 3. Coordination distances (Å) to the potassium ions in  $K_2[VFO(O_2)_2]$ .

K1...O3(A)	$(\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.694(4)	K1...O2(B)	$(x, y, z)$	2.666(5)
F(A)	$(x, y, z)$	2.712(3)	F(B)	$(\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z)$	2.744(5)
F(A)	$(\frac{1}{2}-x, 1-y, -\frac{1}{2}+z)$	2.731(3)	F(B)	$(\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.815(5)
O2(A)	$(-\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.767(5)	O2(B)	$(\frac{1}{2}-x, 1-y, -\frac{1}{2}+z)$	2.838(5)
O5(A)	$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$	2.860(5)	O3(B)	$(-\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.852(7)
O3(A)	$(\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z)$	2.907(4)	O5(B)	$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$	2.864(7)
O1(A)	$(x, y, z)$	2.911(4)	O1(B)	$(\frac{1}{2}-x, 1-y, -\frac{1}{2}+z)$	2.941(7)
O1(A)	$(-\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.950(5)	O5(B)	$(-\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	3.015(7)
O4(A)	$(\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z)$	2.951(4)	O3(B)	$(x, y, z)$	3.094(6)
K2...O4(A)	$(1+x, y, z)$	2.662(4)	O4(B)	$(-\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	3.097(7)
O2(A)	$(\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.713(5)	K2...O2(B)	$(x, y, z)$	2.549(5)
O3(A)	$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$	2.783(4)	O1(B)	$(1+x, y, z)$	2.685(6)
O4(A)	$(\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$	2.851(4)	O4(B)	$(\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.758(7)
F(A)	$(x, y, z)$	2.863(4)	O3(B)	$(\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.830(7)
O1(A)	$(\frac{1}{2}+x, \frac{1}{2}-y, \bar{z})$	2.973(6)	O1(B)	$(\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$	2.906(6)
O5(A)	$(x, y, z)$	3.086(5)	O4(B)	$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$	3.052(7)
O5(A)	$(\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$	3.119(5)	F(B)	$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$	3.126(5)
O2(A)	$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$	3.150(5)			

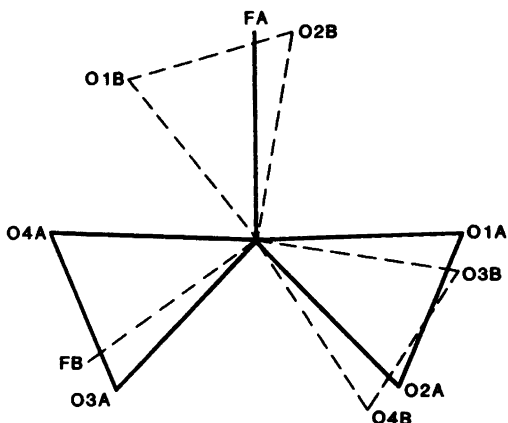


Fig. 3. The relative orientation of the equatorial planes in the anions A and B. These planes are inclined at  $5.6^\circ$  to each other.

ten nearest fluorine and oxygen atoms at distances ranging from 2.549(5) to 3.150(5) Å, the shortest value being observed for the smallest coordination number. In  $K_3[HF_2][TaF_5(O_2)]$  values ranging from 2.584(7) to 3.124(10) Å were observed.<sup>52</sup> These distances may be compared with the radii sum of 2.7 Å.

Due to the disorder, data were collected for a second crystal obtained from a different preparation (but only up to  $2\theta=60^\circ$ ). Least-squares refinement led to the same result, including the occupation numbers, within the limits of experimental errors.

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**Note added in proof.** The crystal structures of  $(NH_4)_2[VFO(O_2)_2]$ <sup>37</sup> and  $(NH_4)_3[VF_2O(O_2)_2]$ <sup>38</sup> have been solved and refined to the  $R(F)$ -values 0.035 and 0.027, respectively.  $(NH_4)_2[VFO(O_2)_2]$  crystallizes in the monoclinic space group  $P2_1/c$  (No. 14) with  $a=6.123(2)$  Å,  $b=11.319(4)$  Å,  $c=8.076(2)$  Å,  $\beta=89.95(3)^\circ$  and  $Z=4$ . There is a profound difference between the structures of  $(NH_4)_2[VFO(O_2)_2]$  and  $K_2[VFO(O_2)_2]$  although the stoichiometry and cell dimensions might indicate isomorphism. While the anions in the potassium salt are discrete and have pentagonal-pyramidal configuration (C.N. 6), the  $VFO(O_2)_2^{2-}$  units in the ammonium compound are linked

together through the oxide oxygen atom to form  $\dots V=O\dots V=O$  chains extended along  $c$ . The vanadium-oxygen distances are 1.613(1) and 2.505(1) Å, respectively, the vanadium atoms thus being seven-coordinated with a pentagonal-bipyramidal ligand configuration, as in  $(NH_4)_4[O\{VO(O_2)_2\}_2]$ .<sup>25</sup> The two *trans*  $V-O_{\text{peroxo}}$  bond distances, 1.903(1) and 1.905(1) Å, are significantly longer than the two *cis*  $V-O_{\text{peroxo}}$  bond distances, 1.874(1) and 1.880(2) Å. The  $V-F$  bond is 1.929(1) Å, the  $O-O$  bonds are 1.460(2) and 1.462(2) Å, and the vanadium atom is displaced 0.364 Å from the pentagonal plane towards the double-bonded oxygen atom.

Crystals of  $(NH_4)_3[VF_2O(O_2)_2]$  are orthorhombic, space group  $P2_12_12_1$  (No. 19) with  $a=6.866(2)$  Å,  $b=8.365(1)$  Å,  $c=13.264(2)$  Å and  $Z=4$ . They contain discrete, seven-coordinated pentagonal-bipyramidal  $[VF_2O(O_2)_2]^{3-}$  anions with the double-bonded oxygen atom and one fluorine atom occupying the apical positions.

Bond distances:  $V=O$  1.609(2) Å, *trans*  $V-O_{\text{peroxo}}$  1.921(2) and 1.927(2) Å, *cis*  $V-O_{\text{peroxo}}$  1.890(2) and 1.887(2) Å,  $V-F_{\text{equatorial}}$  1.958(1) Å,  $V-F_{\text{apical}}$  2.306(1) Å and  $(O-O)_{\text{peroxo}}$  1.462(2) and 1.466(2) Å. The vanadium atom is displaced 0.309 Å from the equatorial plane towards the double-bonded oxygen atom. There is, therefore, a substantial change in coordination in going from  $K_2[VFO(O_2)_2]$  via  $(NH_4)_2[VFO(O_2)_2]$  to  $(NH_4)_3[VF_2O(O_2)_2]$ .

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