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Dipotassium aquapentafluorovanadate

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The title compound, $K_2[VF_5(H_2O)]$, was synthesized from potassium antimony tartrate, piperazine, V_2O_5 and HF under hydrothermal conditions. It is isostructural with $K_2[FeF_5(H_2O)]$ and contains polymeric anion chains held together by strong O—H···F bonds. Each V atom is coordinated to five terminal F atoms and the O atom of one water molecule. Pairs of O—H···F bonds are formed by two *cis*-related F atoms. Twofold axes run along the O—V—F axis of the V-centred otahedra.

Comment

Members of the K_2MF_6 series have been well studied and have been found to adopt structures similar to that of K_2PtCl_6, with virtually ideal octahedral arrangements of F atoms around the metal atoms (Wyckoff, 1951). For compounds of the $K_2MF_5(H_2O)$ series, previous researchers initially expected to obtain distinct $[MF_5(H_2O)]^{2-}$ anions in the crystal, deeming the water molecules to be part of the anion. However, studies of $K_2AlF_5(H_2O)$ (Wyckoff, 1951) and $K_2MnF_5(H_2O)$ (Edwards, 1971*a*) indicated that there are no separate $[MF_5(H_2O)]^{2-}$ anions; the water molecule is not part of the anion. The trivalent cation is octahedrally coordinated by bridging *trans* F atoms, thus forming polymeric MF_6 anion chains. In this paper, we report the hydrothermal synthesis and crystal structure of the title compound, $K_2[VF_5(H_2O)]$, which is found to be isostructural with $K_2[FeF_5(H_2O)]$ (Edwards, 1971*b*). In the title compound, separate $[MF_5(H_2O)]^{2-}$ anions do exist. The water molecule is part of the anion and participates in strong O—H···F bonds between two anions. The structural arrangement is shown in Fig. 1. The V atom is octahedrally coordinated to five F atoms and one water molecule. There are three terminal F atoms in each octahedron, with two others (F3) forming O—H···F bonds [O···F = 2.565 (3) Å and O—H···F = 170°] configured as kinked anion chains parallel to the *c* axis. The K^+ ions 'fill' the spaces between the anions. There are three types of V—F bonds. In the short V—F2 bond [1.9083 (10) Å], the F atom is *cis* with respect to the O atom, while in the long V—F1 bond [1.9538 (14) Å], the F atom is *trans* with respect to the O atom. The three terminal V—F bonds differ in length from the analogous Fe—F bonds in $K_2[FeF_5(H_2O)]$, while the V—F3 bond of 1.9250 (10) Å is intermediate in length between the V—F1 and V—F2 bonds because of the effect of the O—H···F bonds.

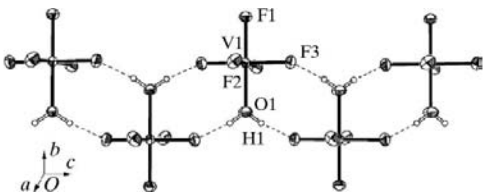

Figure 1
The structure of an anion chain in the title compound, viewed along the *a* axis, with displacement ellipsoids at the 50% probability level.

Unfortunately, no examples of distinct $[VF_6]^{3-}$ octahedra that are directly comparable to the title complex have been identified. However, $VF_3(H_2O)_3$ (Mootz & Schwarz, 1991) and $Na_3V_2(PO_4)_2F_3$ (Le Meins *et al.*, 1999), which have been characterized, have V—F bond lengths of 1.933 and 1.881 Å, respectively, which differ slightly from those reported here.

In $(NH_4)_2[VF_4O]$ (Bukovec & Golic, 1980), the presence of ammonium causes the terminal V—F bond lengths to be of unequal lengths. The difference between the lengths of the two bridging V—F bonds is that one is *cis* and the other is *trans* with respect to the O atom. Waltersson (1979) discussed different metal–ligand effects and considered that the *trans* effect plays an important role in differences in the V—F bond length. We therefore suspect that the *trans* effect plays an appreciable role in governing the V—F bond lengths in $K_2[VF_5(H_2O)]$

In the V-centred octahedra, the V—O bond lengths [2.066 (2) Å] are similar to the Fe—O bond length in $K_2[FeF_5(H_2O)]$ but longer than the V—O bond length in $VF_3(H_2O)_3$ [1.985 (4) Å]. Edwards (1971*b*) assessed the effect of the water molecule in $K_2[FeF_5(H_2O)]$ on the five-coordinate species and concluded that the water molecule has an appreciable effect on the ligand$_{\text{apical}}$–*M*–ligand$_{\text{basal}}$ bond angles.

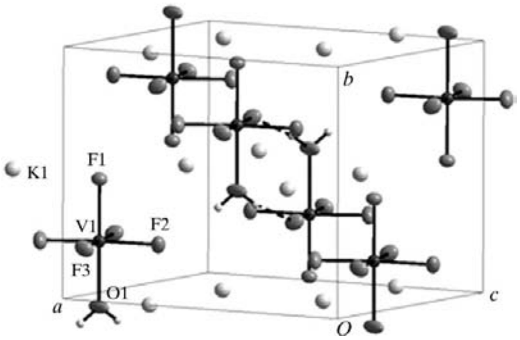

Figure 2
The crystal packing of the title compound, with displacement ellipsoids at the 50% probability level.

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In the title compound, although the V—O bond length is so long that the V—O bond order is feeble, the V—O bond affects the ligand–*M*–ligand angles, which differ from those in $VF_3(H_2O)_3$ (the $F_{apical}—V—F_{basal}$, F—V—O and O—V—F angles are 92.9, 88.5 and 177.9°, respectively). The F—V—F and F—V—O angles of the title compound are listed in Table 1.

The structural arrangement of the chain is shown in Fig. 2. Two $[VF_5(H_2O)]^{2-}$ anions have *C*2 symmetry; two F3 atoms that are *cis* with respect to the O atom form the above-mentioned O—H···F bonds.

Experimental

The title compound was prepared hydrothermally from a mixture of V_2O_5 (0.5 mmol), potassium antimony tartrate (1 mmol), pipe (piperazine; 1 *M*, 2 ml), HF (10 mmol) and H_2O (6 ml), with a V–K–pipe–HF–H_2O ratio of 1:1:2:10:555 and a total volume of 10 ml. All compounds were of AR grade and were used without further purification. The mixture was stirred for about 30 min and then transferred to a 23 ml polytetrafluoroethylene (PTFE) bottle, which was heated to 423 K for a period of 2 d. Light-green crystals of the title compound were obtained in ~50% yield.

Crystal data

$K_2[VF_5(H_2O)]$
$M_r = 242.16$
Monoclinic, *C*2/*c*
a = 9.7328 (10) Å
b = 7.9105 (6) Å
c = 7.9803 (7) Å
β = 96.578 (4)°
V = 610.37 (9) Å^3
Z = 4

$D_x = 2.635$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 25 reflections
θ = 12–18°
μ = 3.01 mm^{-1}
T = 293 (2) K
Block, green
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*TEXRAY*; Molecular Structure Corporation, 1999)
$T_{min} = 0.537$, $T_{max} = 0.548$

701 measured reflections
701 independent reflections
651 reflections with $I > 2\sigma(I)$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 10$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.054$
S = 1.11
701 reflections
44 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0296P)^2 + 0.3825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected bond angles (°).

$F2^i—V1—F2$	177.35 (7)	$F2—V1—F1$	91.33 (3)
$F2^i—V1—F3$	90.58 (5)	$F3—V1—F1$	89.90 (3)
$F2—V1—F3$	89.43 (5)	$F2—V1—O1$	88.67 (3)
$F3—V1—F3^i$	179.80 (7)	$F3—V1—O1$	90.10 (3)

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

The H atoms were fixed at positions found from difference Fourier maps.

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1993); software used to prepare material for publication: *SHELXL*97-2 (Sheldrick, 1997).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: IZ1034). Services for accessing these data are described at the back of the journal.

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