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 β -K(VO₂)₂(PO₄)

Safa Ezzine Yahmed, Meriem Ayed, Mohamed Faouzi Zid* and Ahmed Driss

Laboratoire de Matériaux et Cristallographie, Faculté des Sciences de Tunis,
 Université de Tunis ElManar, 2092 Manar II Tunis, Tunisia
 Correspondence e-mail: faouzi.zid@fst.rnu.tn

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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{P}-\text{O}) = 0.002$ Å;
 R factor = 0.030; wR factor = 0.086; data-to-parameter ratio = 13.1.

A new vanadium oxide, potassium bis(dioxovanadyl) phosphate, β -K(VO₂)₂(PO₄), has been synthesized by a solid-state reaction. In the title compound, the [V₂PO₈] framework is built up from infinite pyramidal [V₂O₈]_∞ and [VPO₇]_∞ chains linked together by V—O—P bridges, leading to a three-dimensional framework which delimits two types of intersecting tunnels running along [100] and [010] in which the four unique K⁺ ions, showing coordination numbers of nine and ten, are located.

Related literature

For α -K(VO₂)₂(PO₄), see: Berrah *et al.* (1999). For background to the physico-chemical properties of related compounds, see: Daidouh *et al.* (1997); Pierini & Lombardo (2005). For details of structurally related compounds, see: Leclaire & Raveau (2006); Amoros & Le Bail (1992); Lii & Wang (1989); Daidouh *et al.* (1998); Benhamada *et al.* (1991). For the preparation, see: Ezzine *et al.* (2009). For bond-valence parameters, see: Brown & Altermatt (1985).

Experimental

Crystal data

K(VO₂)₂(PO₄)
 $M_r = 299.95$
 Triclinic, $P\bar{1}$

$a = 4.7438$ (8) Å
 $b = 13.889$ (2) Å
 $c = 21.201$ (3) Å

$\alpha = 70.89$ (2)°
 $\beta = 89.55$ (3)°
 $\gamma = 88.66$ (3)°
 $V = 1319.5$ (4) Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 3.71$ mm⁻¹
 $T = 298$ K
 $0.28 \times 0.18 \times 0.12$ mm

Data collection

Enraf-Nonius CAD-4
 diffractometer
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\text{min}} = 0.447$, $T_{\text{max}} = 0.668$
 7567 measured reflections

5673 independent reflections
 4828 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 2 standard reflections every 120 min
 intensity decay: 1.2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.086$
 $S = 1.08$
 5673 reflections

434 parameters
 $\Delta\rho_{\text{max}} = 0.80$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.50$ e Å⁻³

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2128).

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 β -K(VO₂)₂(PO₄)

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S1. Comment

Materials developed in the A—V—X—O systems (where A=Alkali, X= P or As) and having open mixed anionic frameworks (uni, bi, and three-dimensional) have interesting physical properties, especially catalytic (Pierini *et al.*, 2005) and ionic conductivity (Daidouh *et al.*, 1997). This has led to the synthesis, by a solid state reaction, of a new form of divanadium phosphate KV₂PO₈. The asymmetric unit, (Fig. 1) of the title compound, is built up from four double pyramidal units V₂O₉ (consisting of distorted vertices-sharing VO₅ trigonal-bipyramids encountered in A₆V₆P₆O₃₁ (A= Rb and K (Benhamada *et al.*, 1991; Leclaire & Raveau, 2006)) interconnected by corner-sharing PO₄ single tetrahedra. The junction of each kind of V₂O₉ unit by V—O—V bridges leads to two types of infinite chains with formula (V₂O₈)_∞. The first kind is parallel to the *b* axis where two successive pairs of pyramids have their apical oxygen atoms pointing towards the same direction (Fig. 2a). The second type is parallel to the *a* axis (Fig. 3) and two successive pairs of pyramids have their apical oxygen atoms pointing towards opposite directions. The compound can be described as a connection between the (V₂O₈)_∞ chains by vertex sharing of VO₅ pentahedra and PO₄ tetrahedra. Projections of the structure along the *a* and *b* axis show that the VO₅ and PO₄ polyhedra alternate along the *a* axis to form infinite chains (VPO₇)_∞ in which vanadium polyhedra share an oxygen with one neighboring chain, leading to double chains (V₂P₂O₁₂)_∞. Adjacent (V₂O₈)_∞ chains running along *b* are connected by double chains of vanadyl phosphate *via* corner sharing. The structure is depicted in (Fig. 4) and (Fig. 5) and can be denoted as a three-dimensional framework with two types of tunnels running along *a* and *b* axis where the monovalent cations K⁺ are located.

The bond valence sums (BVS) calculations using the empirical formula of Brown (Brown & Altermatt, 1985) assuming cations bonds give the values: P1(5.045), P2(4.991), P3(5.013), P4(5.027), V1(5.106), V2(5.066), V3(5.028), V4(5.127), V5(5.056), V6(5.081), V7(5.087), V8(5.115), K1(1.029), K2(0.991), K3(1.031) and K4(1.004) which verify coordination geometries and oxidation states for each atom.

The comparison of our material with those found in the literature reveals the presence of infinite chains (VPO₇)_∞, (V₂P₂O₁₂)_∞ and (V₂O₈)_∞ in the noncentrosymmetric compound with similar formulation α -KV₂PO₈ (Berrah *et al.*, 1999). However, two successive pairs of pyramids have their apical oxygen in a *trans*-position leading to the sequence "*cis-trans-trans-cis*" (Fig. 2b). This leads to eight-sided tunnels that are smaller than those encountered in our structure. (V₂O₈)_∞ chains similar to those found in our phase where two successive pairs of pyramids have their apical oxygen atoms pointing towards the same direction have already been observed for the ammonium hydrogenophosphate α -NH₄VO₂PO₃OH (Amoros & Le Bail, 1992). Materials K₃V₃As₂O₁₄ (Ezzine *et al.*, 2009) and A₂VP₂O₈ (A = Rb, Cs (Lii & Wang, 1989) and Na, Rb (Daidouh *et al.*, 1998)) also contain chains (VXO₇)_∞ (X= As or P) whose linkage form infinite layers of type (V₂As₂O₁₄)_∞ and (VP₂O₈)_∞. The junction of these chains along the three directions of the cell edges leads to three-dimensional structures with large tunnels for α -KV₂PO₈ and K₃V₃As₂O₁₄ compounds. As far as the series of A₂VP₂O₈ (A= Alkali) compounds is concerned, they form by P—O—P bridges two-dimensional structures, characterized by the presence of interlayer space where the cations are located.

S2. Experimental

In order to obtain a new phosphate isotypic with $K_3V_3As_2O_{14}$ (Ezzine *et al.*, 2009), KNO_3 (Fluka, 60415), NH_4VO_3 (Riedel-De Haën, 12739) and $NH_4H_2PO_4$ (Scharlau, AM0335) were first mixed in the molar ratio 3:3:2 and heated at 573 K to decompose the ammonium phosphate and the nitrate. In a second step, the resulting mixture was crushed then heated at 793 K for two days, cooled slowly (5°C/24 h) down to 743 K and finally quenched to room temperature. From the resulting product, well-shaped crystals of various sizes, of satisfactory quality for analysis by X-ray diffraction, were retrieved. A yellow ochre parallelepipedic crystal was chosen from the selection for the determination of cell parameters.

S3. Refinement

The electron density maximum and minimum in the remaining Fourier differences are acceptable and are located respectively at 0.88 Å from O25 and 0.73 Å from V2.

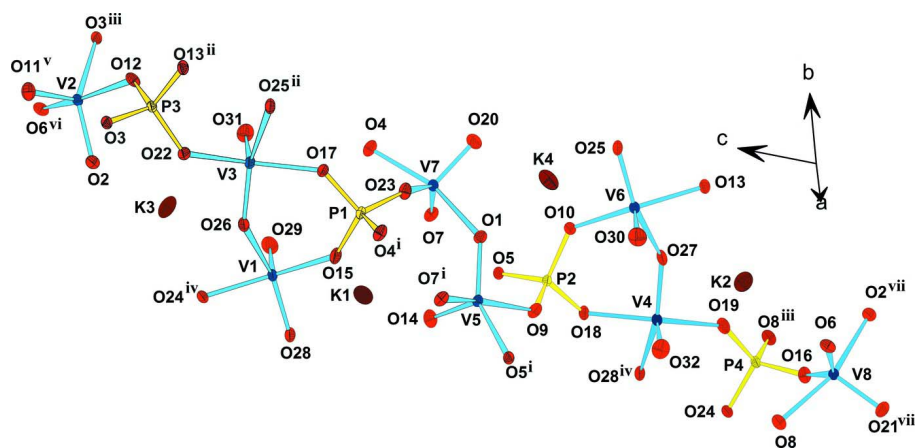


Figure 1

Asymmetric unit of β - $K(VO_2)_2(PO_4)$. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes:(i) $x+1, y, z$; (ii) $-x+1, -y+2, -z+1$; (iii) $x-1, y, z$; (iv) $-x+1, -y+1, -z+1$; (v) $x, y, z+1$; (vi) $x-1, y, z+1$; (vii) $x, y, z-1$.

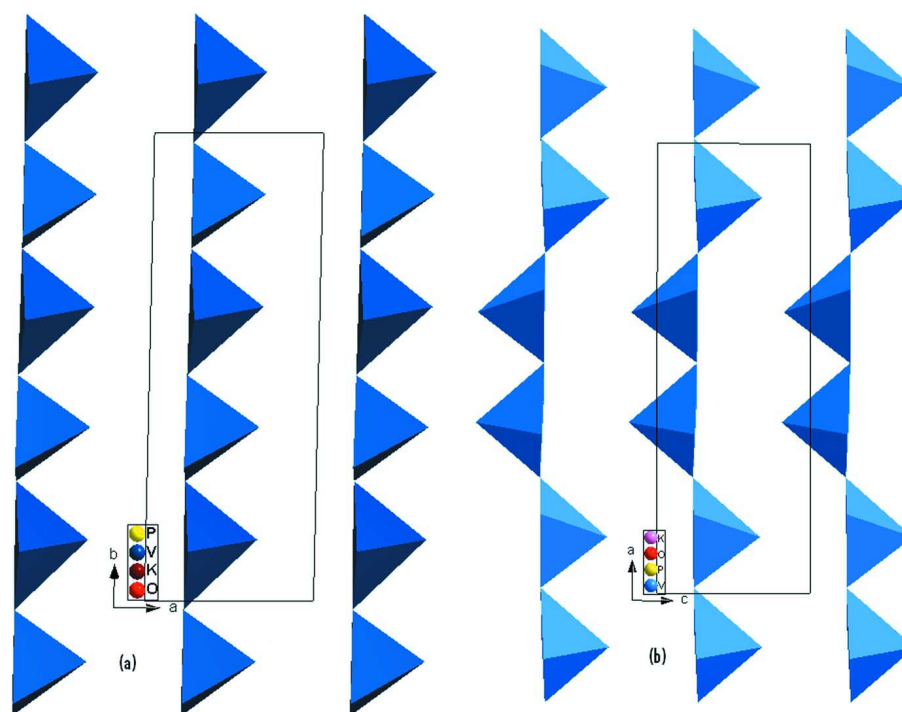


Figure 2

Representation of the first type of infinite chains $(V_2O_8)_\infty$ showing the disposition of apical oxygen atoms: **(a)** in *cis-cis* position in β - $K(VO_2)_2(PO_4)$. **(b)** in *cis-cis-trans-trans* position in α - $K(VO_2)_2(PO_4)$.

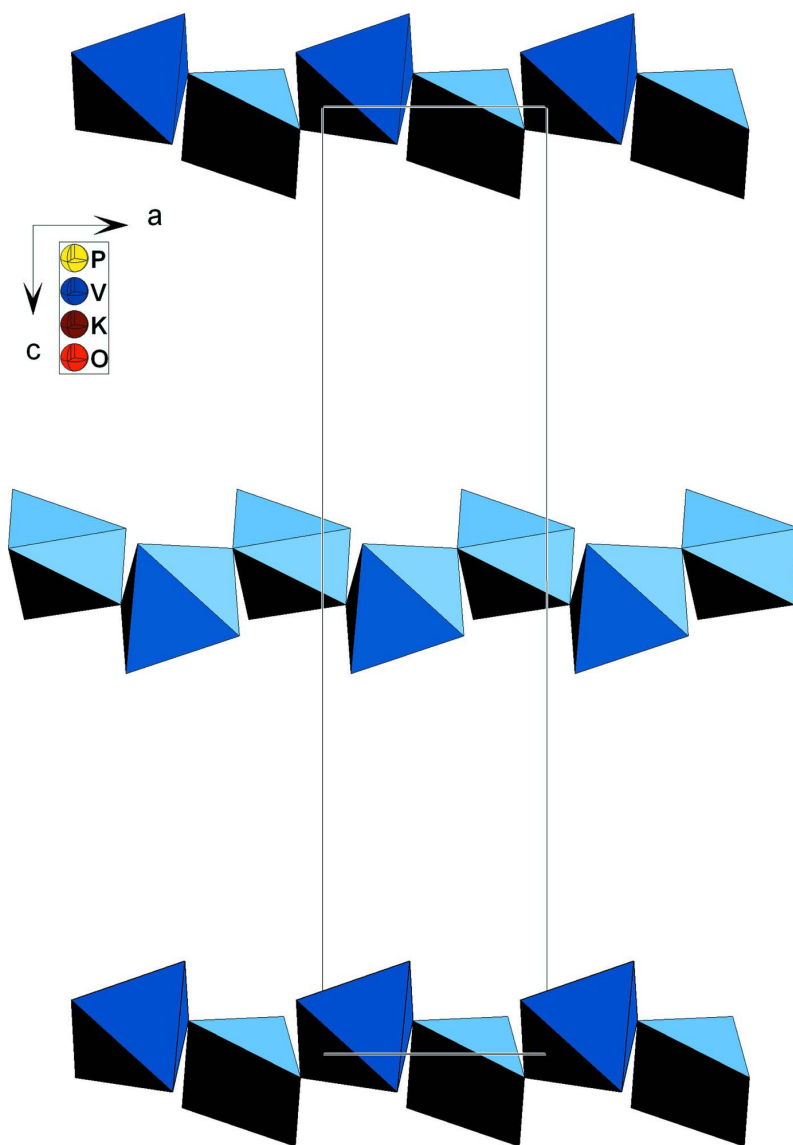


Figure 3
Representation of the second type of $(V_2O_8)_\infty$ chains, running along **a**.

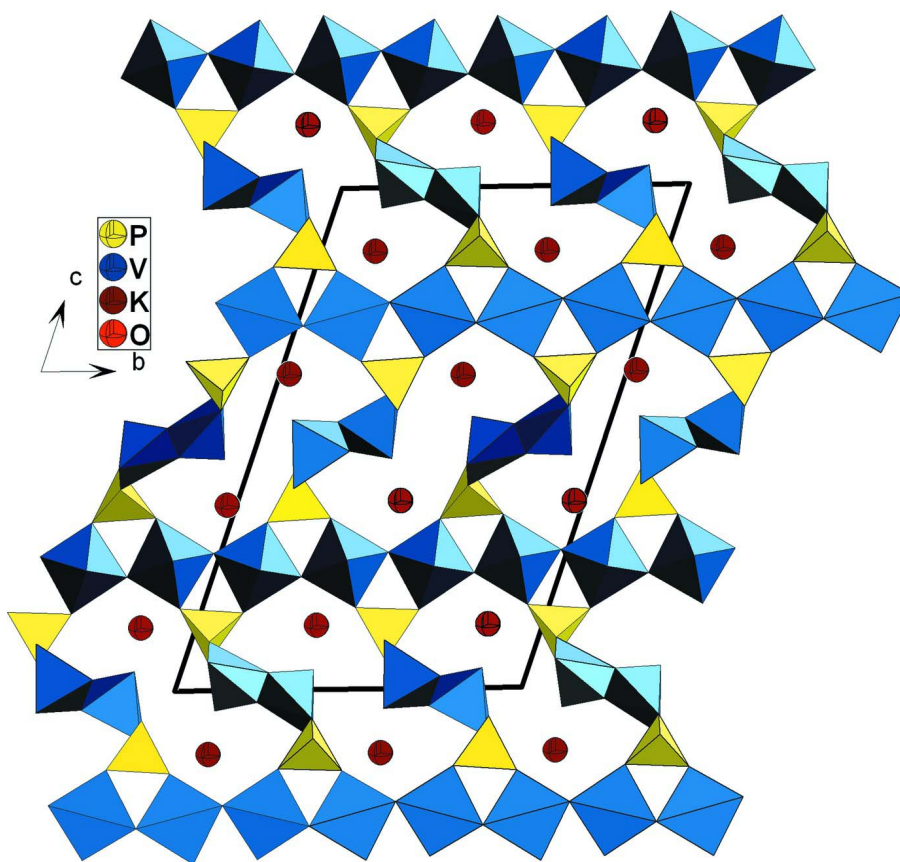


Figure 4

Projection of the structure of β -K(VO₂)₂(PO₄) along *a* showing S shaped tunnels.

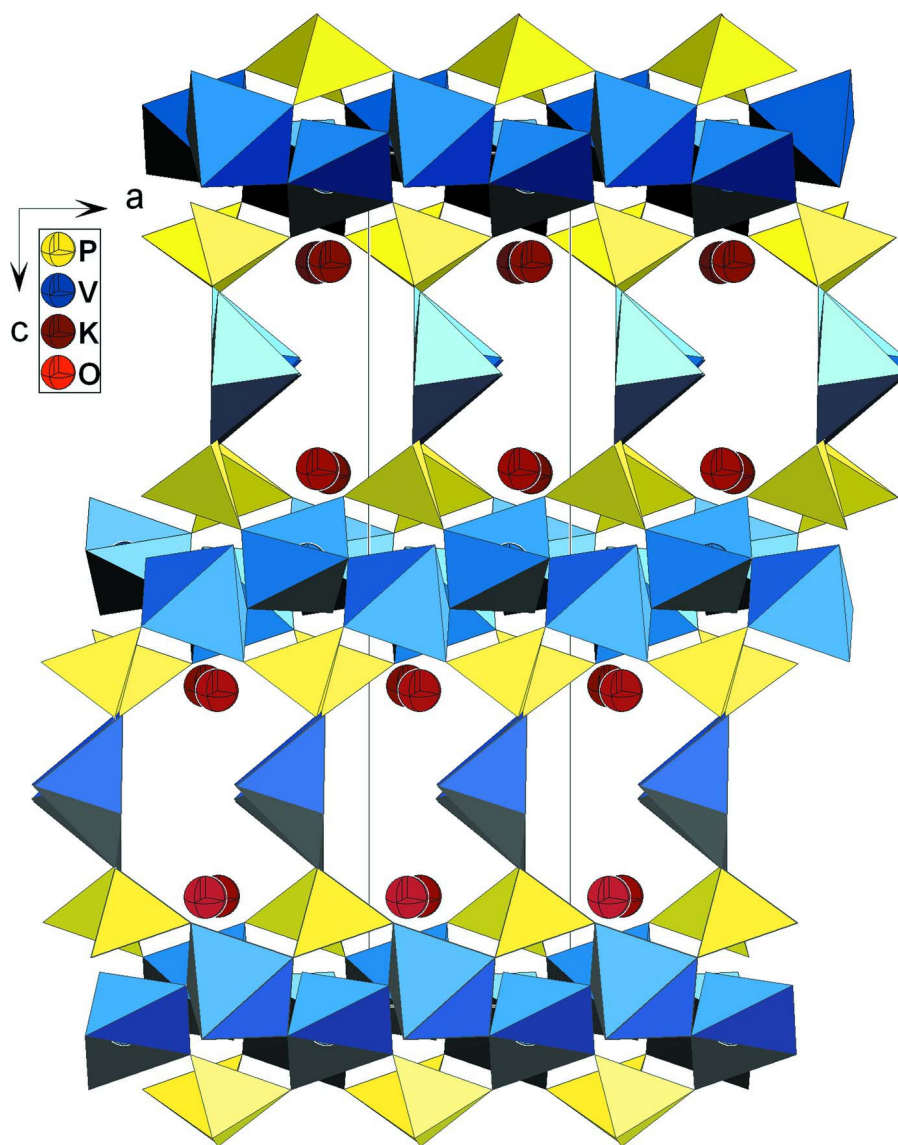


Figure 5

Projection of the structure of β -K(VO₂)₂(PO₄) along **b** showing 'Pacman' shaped tunnels.

Potassium bis(dioxovanadyl) phosphate

Crystal data

K(VO₂)₂(PO₄)
 $M_r = 299.95$
 Triclinic, $P\bar{1}$
 Hall symbol: $-P\ 1$
 $a = 4.7438\ (8)\ \text{\AA}$
 $b = 13.889\ (2)\ \text{\AA}$
 $c = 21.201\ (3)\ \text{\AA}$
 $\alpha = 70.89\ (2)^\circ$
 $\beta = 89.55\ (3)^\circ$
 $\gamma = 88.66\ (3)^\circ$
 $V = 1319.5\ (4)\ \text{\AA}^3$

$Z = 8$
 $F(000) = 1152$
 $D_x = 3.020\ \text{Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$
 Cell parameters from 25 reflections
 $\theta = 10\text{--}16^\circ$
 $\mu = 3.71\ \text{mm}^{-1}$
 $T = 298\ \text{K}$
 Prism, yellow ochre
 $0.28 \times 0.18 \times 0.12\ \text{mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.447$, $T_{\max} = 0.668$

7567 measured reflections

5673 independent reflections

4828 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -6 \rightarrow 1$

$k = -17 \rightarrow 17$

$l = -27 \rightarrow 27$

2 standard reflections every 120 min

intensity decay: 1.2%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.086$

$S = 1.08$

5673 reflections

434 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 1.4475P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.80 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.50 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0177 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.81894 (16)	0.82998 (6)	0.62044 (4)	0.00927 (16)
P2	0.30877 (16)	0.66561 (6)	0.38286 (4)	0.00846 (16)
P3	0.69764 (16)	0.95394 (6)	0.88110 (4)	0.00847 (16)
P4	0.18695 (16)	0.55208 (6)	0.11864 (4)	0.00897 (16)
V1	0.67895 (11)	0.62518 (4)	0.74349 (2)	0.01025 (12)
V2	0.20740 (11)	0.84522 (4)	0.97612 (2)	0.00914 (12)
V3	0.67812 (11)	0.86795 (4)	0.75923 (3)	0.01073 (12)
V4	0.33078 (11)	0.63012 (4)	0.24341 (3)	0.01087 (12)
V5	0.79712 (10)	0.68009 (4)	0.47752 (2)	0.00898 (12)
V6	0.32962 (11)	0.87164 (4)	0.25925 (3)	0.01096 (12)
V7	0.30088 (11)	0.82752 (4)	0.52064 (2)	0.00904 (12)
V8	0.70261 (11)	0.65479 (4)	0.01947 (2)	0.00902 (12)
K1	0.19194 (17)	0.53056 (6)	0.62537 (4)	0.02600 (18)
K2	0.80918 (17)	0.84200 (7)	0.12683 (4)	0.02642 (18)
K3	0.25658 (18)	0.65377 (7)	0.87178 (4)	0.02692 (19)

K4	0.74706 (18)	0.97341 (6)	0.36973 (4)	0.02608 (19)
O1	0.6015 (5)	0.78815 (16)	0.46573 (11)	0.0130 (4)
O2	0.4024 (4)	0.74851 (16)	0.96467 (11)	0.0132 (4)
O3	0.8822 (4)	0.93237 (16)	0.94288 (10)	0.0116 (4)
O4	0.1257 (4)	0.85042 (17)	0.59847 (11)	0.0146 (4)
O5	0.1243 (4)	0.62663 (16)	0.44482 (10)	0.0113 (4)
O6	0.9010 (4)	0.75197 (16)	0.02434 (11)	0.0135 (4)
O7	0.1026 (5)	0.72520 (17)	0.52599 (11)	0.0135 (4)
O8	0.8785 (4)	0.55572 (16)	0.09744 (11)	0.0145 (5)
O9	0.6199 (5)	0.65051 (17)	0.40334 (11)	0.0146 (4)
O10	0.2347 (5)	0.77905 (16)	0.34598 (11)	0.0143 (5)
O11	0.3322 (5)	0.86573 (18)	0.03997 (11)	0.0184 (5)
O12	0.3868 (4)	0.94798 (16)	0.90171 (11)	0.0136 (4)
O13	0.2543 (5)	0.93608 (16)	0.16436 (11)	0.0129 (4)
O14	0.6717 (5)	0.59606 (18)	0.54165 (11)	0.0177 (5)
O15	0.7832 (6)	0.71576 (17)	0.65851 (11)	0.0201 (5)
O16	0.3734 (4)	0.57915 (17)	0.05664 (11)	0.0141 (4)
O17	0.7492 (5)	0.89548 (16)	0.66473 (11)	0.0131 (4)
O18	0.2579 (5)	0.59998 (16)	0.33811 (11)	0.0126 (4)
O19	0.2327 (6)	0.62756 (17)	0.15666 (11)	0.0196 (5)
O20	0.1780 (5)	0.92122 (18)	0.46105 (12)	0.0195 (5)
O21	0.8267 (5)	0.62017 (18)	0.96030 (12)	0.0184 (5)
O22	0.7748 (5)	0.87717 (16)	0.84469 (11)	0.0143 (5)
O23	0.6303 (5)	0.86329 (17)	0.55904 (11)	0.0143 (4)
O24	0.2538 (5)	0.44138 (16)	0.16220 (11)	0.0131 (4)
O25	0.2320 (5)	0.98015 (16)	0.27381 (11)	0.0174 (5)
O26	0.7750 (5)	0.74426 (16)	0.77524 (11)	0.0163 (5)
O27	0.2304 (5)	0.75295 (16)	0.22901 (11)	0.0175 (5)
O28	0.7767 (5)	0.51742 (16)	0.72784 (11)	0.0166 (5)
O29	0.3424 (5)	0.62895 (19)	0.74222 (12)	0.0219 (5)
O30	0.6654 (5)	0.8693 (2)	0.25991 (13)	0.0236 (5)
O31	0.3413 (5)	0.86967 (19)	0.76068 (12)	0.0215 (5)
O32	0.6666 (5)	0.6285 (2)	0.24292 (13)	0.0231 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0107 (4)	0.0107 (3)	0.0072 (3)	-0.0017 (3)	0.0015 (3)	-0.0039 (3)
P2	0.0090 (4)	0.0101 (3)	0.0066 (3)	-0.0001 (3)	-0.0001 (3)	-0.0032 (3)
P3	0.0086 (4)	0.0090 (3)	0.0070 (3)	-0.0001 (3)	0.0002 (3)	-0.0016 (3)
P4	0.0105 (4)	0.0087 (3)	0.0069 (3)	-0.0003 (3)	-0.0015 (3)	-0.0013 (3)
V1	0.0148 (3)	0.0087 (2)	0.0071 (2)	-0.00055 (19)	-0.00053 (19)	-0.00236 (19)
V2	0.0078 (2)	0.0107 (2)	0.0082 (2)	0.00032 (18)	-0.00125 (18)	-0.00212 (19)
V3	0.0153 (3)	0.0088 (2)	0.0081 (2)	-0.00139 (19)	0.00208 (19)	-0.00273 (19)
V4	0.0157 (3)	0.0089 (2)	0.0081 (2)	-0.00129 (19)	-0.0007 (2)	-0.00267 (19)
V5	0.0072 (2)	0.0115 (2)	0.0083 (2)	-0.00063 (18)	0.00118 (18)	-0.00344 (19)
V6	0.0161 (3)	0.0088 (2)	0.0082 (2)	-0.00082 (19)	-0.0007 (2)	-0.00290 (19)
V7	0.0077 (2)	0.0114 (2)	0.0083 (2)	0.00012 (18)	-0.00159 (18)	-0.00353 (19)

V8	0.0081 (2)	0.0101 (2)	0.0083 (2)	-0.00074 (18)	0.00148 (19)	-0.00222 (19)
K1	0.0215 (4)	0.0238 (4)	0.0267 (4)	-0.0012 (3)	0.0034 (3)	-0.0002 (3)
K2	0.0228 (4)	0.0352 (4)	0.0265 (4)	0.0008 (3)	-0.0044 (3)	-0.0173 (4)
K3	0.0252 (4)	0.0351 (4)	0.0268 (4)	-0.0088 (3)	0.0098 (3)	-0.0186 (4)
K4	0.0262 (4)	0.0217 (4)	0.0252 (4)	-0.0035 (3)	-0.0088 (3)	-0.0003 (3)
O1	0.0112 (10)	0.0162 (11)	0.0130 (10)	0.0018 (8)	-0.0012 (8)	-0.0069 (9)
O2	0.0106 (10)	0.0134 (10)	0.0139 (11)	-0.0001 (8)	0.0009 (8)	-0.0023 (8)
O3	0.0109 (10)	0.0149 (10)	0.0085 (10)	0.0027 (8)	-0.0021 (8)	-0.0032 (8)
O4	0.0077 (10)	0.0244 (12)	0.0149 (11)	-0.0013 (9)	0.0013 (8)	-0.0110 (9)
O5	0.0090 (10)	0.0150 (10)	0.0085 (10)	0.0021 (8)	0.0012 (8)	-0.0024 (8)
O6	0.0092 (10)	0.0120 (10)	0.0169 (11)	-0.0007 (8)	-0.0019 (9)	-0.0014 (8)
O7	0.0099 (10)	0.0176 (11)	0.0160 (11)	-0.0025 (8)	0.0016 (9)	-0.0094 (9)
O8	0.0083 (10)	0.0172 (11)	0.0142 (11)	-0.0006 (8)	0.0006 (8)	0.0001 (9)
O9	0.0095 (11)	0.0224 (11)	0.0156 (11)	0.0013 (9)	-0.0015 (9)	-0.0114 (9)
O10	0.0217 (12)	0.0102 (10)	0.0103 (10)	0.0013 (9)	-0.0003 (9)	-0.0025 (8)
O11	0.0184 (12)	0.0234 (12)	0.0137 (11)	-0.0025 (9)	-0.0029 (9)	-0.0063 (9)
O12	0.0082 (10)	0.0146 (10)	0.0137 (11)	0.0003 (8)	-0.0015 (8)	0.0011 (8)
O13	0.0190 (12)	0.0105 (10)	0.0087 (10)	-0.0037 (8)	0.0014 (9)	-0.0022 (8)
O14	0.0149 (11)	0.0218 (12)	0.0135 (11)	-0.0046 (9)	0.0032 (9)	-0.0015 (9)
O15	0.0389 (15)	0.0112 (11)	0.0105 (11)	-0.0044 (10)	0.0079 (10)	-0.0040 (9)
O16	0.0095 (10)	0.0193 (11)	0.0122 (10)	-0.0033 (8)	0.0012 (8)	-0.0030 (9)
O17	0.0187 (12)	0.0112 (10)	0.0097 (10)	0.0000 (8)	0.0032 (9)	-0.0041 (8)
O18	0.0176 (11)	0.0128 (10)	0.0078 (10)	-0.0019 (8)	-0.0010 (8)	-0.0037 (8)
O19	0.0354 (14)	0.0116 (11)	0.0119 (11)	-0.0016 (10)	-0.0066 (10)	-0.0038 (9)
O20	0.0195 (12)	0.0199 (12)	0.0161 (12)	0.0051 (9)	-0.0034 (10)	-0.0021 (9)
O21	0.0183 (12)	0.0234 (12)	0.0150 (11)	0.0029 (9)	0.0039 (9)	-0.0085 (9)
O22	0.0214 (12)	0.0105 (10)	0.0113 (11)	0.0003 (9)	0.0010 (9)	-0.0041 (8)
O23	0.0105 (11)	0.0223 (11)	0.0117 (10)	-0.0019 (9)	-0.0019 (8)	-0.0076 (9)
O24	0.0197 (12)	0.0095 (10)	0.0090 (10)	0.0015 (8)	-0.0022 (9)	-0.0017 (8)
O25	0.0343 (14)	0.0103 (10)	0.0075 (10)	-0.0020 (9)	0.0023 (9)	-0.0028 (8)
O26	0.0294 (13)	0.0107 (10)	0.0086 (10)	0.0004 (9)	-0.0008 (9)	-0.0029 (8)
O27	0.0334 (14)	0.0109 (10)	0.0075 (10)	-0.0016 (9)	-0.0031 (10)	-0.0017 (8)
O28	0.0317 (13)	0.0099 (10)	0.0081 (10)	-0.0020 (9)	0.0023 (9)	-0.0027 (8)
O29	0.0166 (12)	0.0270 (13)	0.0201 (13)	0.0027 (10)	-0.0033 (10)	-0.0049 (10)
O30	0.0173 (13)	0.0282 (13)	0.0248 (13)	0.0003 (10)	-0.0031 (10)	-0.0082 (11)
O31	0.0165 (12)	0.0269 (13)	0.0213 (13)	-0.0015 (10)	0.0030 (10)	-0.0080 (10)
O32	0.0155 (12)	0.0304 (14)	0.0243 (13)	-0.0037 (10)	0.0018 (10)	-0.0098 (11)

Geometric parameters (Å, °)

P1—O23	1.521 (3)	V7—O7	1.695 (2)
P1—O4 ⁱ	1.529 (2)	V7—O23	1.917 (2)
P1—O17	1.536 (2)	V7—O4	1.955 (2)
P1—O15	1.539 (2)	V7—O1	2.010 (3)
P2—O5	1.524 (2)	V8—O21 ^{viii}	1.588 (2)
P2—O9	1.532 (2)	V8—O6	1.694 (2)
P2—O18	1.539 (2)	V8—O16	1.918 (3)
P2—O10	1.546 (2)	V8—O8	1.950 (3)

P3—O3	1.523 (2)	V8—O2 ^{vii}	2.005 (3)
P3—O12	1.531 (2)	K1—O18 ^{viii}	2.771 (3)
P3—O13 ⁱⁱ	1.537 (2)	K1—O14	2.852 (3)
P3—O22	1.544 (2)	K1—O7	2.856 (3)
P4—O16	1.525 (3)	K1—O28 ⁱⁱⁱ	2.885 (3)
P4—O8 ⁱⁱⁱ	1.529 (2)	K1—O9 ^{iv}	2.895 (3)
P4—O19	1.537 (2)	K1—O14 ⁱⁱⁱ	2.996 (3)
P4—O24	1.539 (2)	K1—O32 ^{iv}	3.008 (4)
V1—O29	1.596 (2)	K1—O18 ^{iv}	3.097 (3)
V1—O28	1.690 (2)	K1—O29	3.287 (3)
V1—O15	1.899 (3)	K2—O13 ⁱ	2.763 (3)
V1—O24 ^{iv}	1.934 (2)	K2—O6	2.866 (3)
V1—O26	2.041 (2)	K2—O11	2.873 (3)
V2—O11 ^v	1.593 (2)	K2—O27 ⁱ	2.901 (4)
V2—O2	1.694 (2)	K2—O12 ⁱⁱ	2.910 (3)
V2—O3 ⁱⁱⁱ	1.927 (3)	K2—O11 ⁱ	3.037 (3)
V2—O12	1.952 (3)	K2—O30	3.038 (3)
V2—O6 ^{vi}	2.008 (3)	K2—O13	3.124 (3)
V3—O31	1.598 (2)	K2—O32	3.256 (4)
V3—O26	1.692 (2)	K3—O21 ⁱⁱⁱ	2.704 (3)
V3—O22	1.918 (2)	K3—O2	2.802 (3)
V3—O17	1.941 (2)	K3—O24 ^{iv}	2.848 (3)
V3—O25 ⁱⁱ	2.048 (2)	K3—O8 ^{iv}	2.853 (3)
V4—O32	1.592 (2)	K3—O29	2.903 (3)
V4—O27	1.689 (2)	K3—O24 ^{viii}	2.988 (3)
V4—O19	1.912 (2)	K3—O26 ⁱⁱⁱ	3.040 (4)
V4—O18	1.942 (2)	K3—O31	3.181 (4)
V4—O28 ^{iv}	2.014 (2)	K3—O26	3.193 (4)
V5—O14	1.595 (3)	K3—O21	3.240 (3)
V5—O1	1.694 (2)	K4—O20 ⁱ	2.744 (3)
V5—O5 ⁱ	1.926 (2)	K4—O1	2.806 (3)
V5—O9	1.952 (2)	K4—O4 ⁱⁱ	2.822 (3)
V5—O7 ⁱ	2.009 (3)	K4—O17 ⁱⁱ	2.889 (3)
V6—O30	1.593 (3)	K4—O31 ⁱⁱ	2.933 (3)
V6—O25	1.688 (2)	K4—O17 ^{ix}	2.977 (3)
V6—O10	1.924 (3)	K4—O25 ⁱ	3.041 (3)
V6—O13	1.948 (2)	K4—O30	3.147 (3)
V6—O27	2.021 (2)	K4—O25	3.171 (3)
V7—O20	1.590 (3)	K4—O20	3.264 (4)
O23—P1—O4 ⁱ	109.01 (14)	O22—V3—O25 ⁱⁱ	83.48 (11)
O23—P1—O17	109.42 (14)	O17—V3—O25 ⁱⁱ	76.99 (11)
O4 ⁱ —P1—O17	106.61 (14)	O32—V4—O27	105.91 (15)
O23—P1—O15	110.17 (15)	O32—V4—O19	103.89 (15)
O4 ⁱ —P1—O15	109.90 (17)	O27—V4—O19	95.54 (12)
O17—P1—O15	111.63 (13)	O32—V4—O18	100.53 (14)
O5—P2—O9	109.44 (14)	O27—V4—O18	90.26 (12)
O5—P2—O18	108.38 (13)	O19—V4—O18	152.23 (11)

O9—P2—O18	106.84 (14)	O32—V4—O28 ^{iv}	105.13 (15)
O5—P2—O10	109.41 (14)	O27—V4—O28 ^{iv}	148.24 (12)
O9—P2—O10	111.43 (16)	O19—V4—O28 ^{iv}	83.39 (11)
O18—P2—O10	111.26 (13)	O18—V4—O28 ^{iv}	77.61 (11)
O3—P3—O12	109.42 (13)	O14—V5—O1	106.78 (13)
O3—P3—O13 ⁱⁱ	108.56 (14)	O14—V5—O5 ⁱ	110.53 (13)
O12—P3—O13 ⁱⁱ	106.81 (15)	O1—V5—O5 ⁱ	142.54 (11)
O3—P3—O22	109.33 (14)	O14—V5—O9	103.24 (12)
O12—P3—O22	111.71 (15)	O1—V5—O9	93.29 (12)
O13 ⁱⁱ —P3—O22	110.93 (13)	O5 ⁱ —V5—O9	81.56 (10)
O16—P4—O8 ⁱⁱⁱ	109.06 (13)	O14—V5—O7 ⁱ	96.23 (12)
O16—P4—O19	109.92 (14)	O1—V5—O7 ⁱ	93.05 (11)
O8 ⁱⁱⁱ —P4—O19	110.26 (15)	O5 ⁱ —V5—O7 ⁱ	79.85 (10)
O16—P4—O24	108.94 (14)	O9—V5—O7 ⁱ	156.79 (10)
O8 ⁱⁱⁱ —P4—O24	106.65 (15)	O30—V6—O25	105.48 (15)
O19—P4—O24	111.92 (13)	O30—V6—O10	103.21 (15)
O29—V1—O28	105.95 (15)	O25—V6—O10	96.97 (11)
O29—V1—O15	104.16 (15)	O30—V6—O13	101.01 (15)
O28—V1—O15	95.75 (11)	O25—V6—O13	90.43 (12)
O29—V1—O24 ^{iv}	100.43 (15)	O10—V6—O13	151.70 (10)
O28—V1—O24 ^{iv}	90.45 (11)	O30—V6—O27	103.95 (14)
O15—V1—O24 ^{iv}	151.84 (11)	O25—V6—O27	149.67 (12)
O29—V1—O26	102.98 (14)	O10—V6—O27	83.24 (10)
O28—V1—O26	150.24 (12)	O13—V6—O27	76.87 (10)
O15—V1—O26	83.72 (10)	O20—V7—O7	107.46 (13)
O24 ^{iv} —V1—O26	77.53 (10)	O20—V7—O23	111.76 (13)
O11 ^v —V2—O2	106.92 (13)	O7—V7—O23	140.64 (12)
O11 ^v —V2—O3 ⁱⁱⁱ	110.30 (13)	O20—V7—O4	101.96 (12)
O2—V2—O3 ⁱⁱⁱ	142.66 (11)	O7—V7—O4	94.01 (12)
O11 ^v —V2—O12	103.25 (12)	O23—V7—O4	81.21 (10)
O2—V2—O12	93.04 (11)	O20—V7—O1	95.37 (13)
O3 ⁱⁱⁱ —V2—O12	81.45 (11)	O7—V7—O1	93.37 (11)
O11 ^v —V2—O6 ^{vi}	96.50 (12)	O23—V7—O1	80.05 (10)
O2—V2—O6 ^{vi}	92.90 (11)	O4—V7—O1	158.15 (10)
O3 ⁱⁱⁱ —V2—O6 ^{vi}	80.19 (10)	O21 ^{vii} —V8—O6	107.21 (13)
O12—V2—O6 ^{vi}	156.70 (10)	O21 ^{vii} —V8—O16	110.63 (12)
O31—V3—O26	105.43 (15)	O6—V8—O16	142.02 (11)
O31—V3—O22	102.93 (14)	O21 ^{vii} —V8—O8	102.02 (12)
O26—V3—O22	97.24 (12)	O6—V8—O8	93.61 (11)
O31—V3—O17	101.01 (14)	O16—V8—O8	81.75 (11)
O26—V3—O17	90.49 (12)	O21 ^{vii} —V8—O2 ^{vii}	95.70 (12)
O22—V3—O17	151.81 (10)	O6—V8—O2 ^{vii}	93.36 (10)
O31—V3—O25 ⁱⁱ	102.47 (15)	O16—V8—O2 ^{vii}	79.98 (10)
O26—V3—O25 ⁱⁱ	151.14 (12)	O8—V8—O2 ^{vii}	158.08 (10)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+2, -z+1$; (iii) $x-1, y, z$; (iv) $-x+1, -y+1, -z+1$; (v) $x, y, z+1$; (vi) $x-1, y, z+1$; (vii) $x, y, z-1$; (viii) $-x, -y+1, -z+1$; (ix) $-x+2, -y+2, -z+1$.