Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

catena-Poly[[oxo(1,10-phenanthroline)-vanadium(IV)]-di-μ-hydrogenphosphato]

Yun-Long Fu,[a] Jia-Lin Ren[a] and Seik Weng Ng[b]*

[a]School of Chemistry and Materials Science, Shanxi Normal University, Linfen 041004, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.015 Å
R factor = 0.064
wR factor = 0.224
Data-to-parameter ratio = 11.6

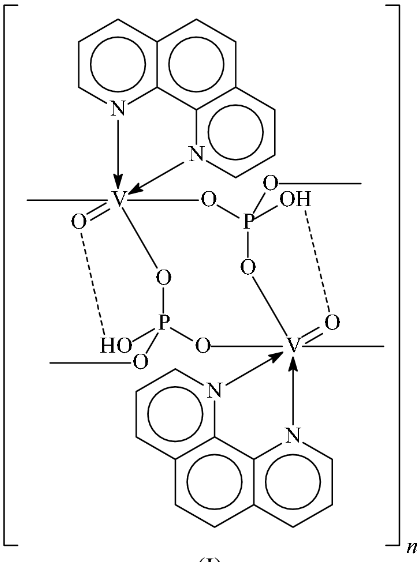
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of $[VO(HPO_4)(C_{12}H_8N_2)]_n$ consists of a bis(vanadyl hydrogen phosphate) skeleton that assumes a linear ribbon conformation; the nitrogen heterocycles that chelate to the V^{IV} atoms are connected to the sides of the flat ribbon. The compound is isostructural with the hydrogen arsenate, whose structure has been reported by Hou *et al.* [*Inorg. Chem. Commun.* (2004). **7**, 128–130]. The asymmetric unit, with the exception of one O atom, lies on a mirror plane

Received 15 September 2004
Accepted 23 September 2004
Online 30 September 2004

Comment

The 1:1 adduct of vanadyl(IV) hydrogen arsenate with 1,10-phenanthroline has a ladder-like $[(HAsO_4)(VO)]_n$ skeleton, at the sides of which are located the coordinated nitrogen heterocycles. The structure of this compound has been described in detail (Hou *et al.*, 2004). The title hydrogen phosphate, (I) (Fig. 1), is isostructural with the reported hydrogen arsenate. Its asymmetric unit, with the exception of atom O3, lies on a mirror plane. This feature is also found in the related 1:1 vanadium(V) pentoxide adduct with the same nitrogen heterocycle (Ng & Hu, 2004). In the phosphate unit, the bonds involving the hydrogen phoshate unit are similar to those found in vanadyl hydrogen phosphate dihydrate (Worzala *et al.*, 1998).



(I)

Experimental

The title compound was synthesized hydrothermally from vanadium(V) pentoxide (0.091 g, 0.5 mmol), concentrated hydrochloric acid (36%, 0.16 ml, 2 mmol), phosphoric acid (0.08 ml, 0.2 mmol), 1,10-phenanthroline (0.091 g, 0.5 mmol) and water (7.2 ml); the reagents were heated in a Teflon-lined stainless steel bomb at 453 K for 5 d along with zinc sulfate heptahydrate (0.28 g, 1.0 mmol). The pH of the mixture was < 1. Red crystals were obtained when the bomb was cooled slowly to room temperature. Analysis found: C 41.95, H 2.60, N 8.15%; calculated for $C_{12}H_9N_2O_5PV$: C 42.00, H 2.64, N 8.17%. Zinc was not incorporated into the product.

Crystal data

$[VO(HPO_4)(C_{12}H_8N_2)]$
$M_r = 343.12$
Monoclinic, $P2_1/m$
$a = 9.815$ (2) Å
$b = 6.372$ (1) Å
$c = 9.919$ (2) Å
$\beta = 102.753$ (4)°
$V = 605.0$ (2) Å^3
$Z = 2$
$D_x = 1.884$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 298 reflections
$\theta = 2.6$–19.6°
$\mu = 0.98$ mm^{-1}
$T = 295$ (2) K
Prism, red
0.10 × 0.10 × 0.03 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.368$, $T_{max} = 0.971$
3639 measured reflections
1434 independent reflections
852 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 27.1°$
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 6$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.224$
$S = 1.09$
1434 reflections
124 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.117P)^2 + 0.0738P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.08$ e Å^{-3}
$\Delta\rho_{min} = -0.70$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

V1—O1	1.607 (7)	V1—N1	2.133 (8)
V1—O3^i	2.000 (4)	V1—N2	2.288 (8)
V1—O4	1.964 (7)		
O1—V1—O3^i	98.3 (1)	O3^i—V1—N1	85.0 (1)
O1—V1—O4	104.5 (3)	O3^i—V1—N2	81.0 (1)
O1—V1—N1	93.8 (4)	O3^{ii}—V1—O4	92.2 (2)
O1—V1—N2	167.9 (4)	O4—V1—N1	161.7 (3)
O3^i—V1—O3^{ii}	161.2 (3)	O4—V1—N2	87.6 (3)
O3^i—V1—O4	92.2 (1)	N1—V1—N2	74.1 (3)

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

H atoms were placed at calculated positions (C—H = 0.93 Å and O—H = 0.85 Å) and were included in the refinements in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$. The final difference Fourier map had a large peak about 1 Å from atom C2, but was otherwise featureless.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; method used to solve structure: atomic coordinates taken from the published As analog (Hou *et al.*, 2004); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

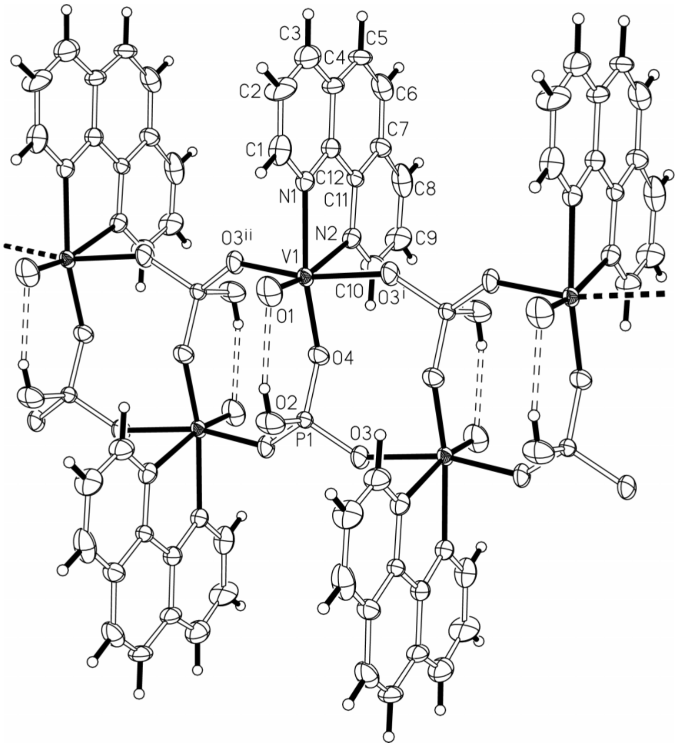


Figure 1
*ORTEP*II (Johnson, 1976) plot of a portion of $[(C_{12}H_8N)_2(HPO_4)(VO)]_n$. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are drawn as spheres of arbitrary radii. Dashed lines indicate O—H···O hydrogen bonds. [Symmetry codes: (i) $2 - x, y - \frac{1}{2}, 2 - z$; (ii) $2 - x, 1 - y, 2 - z$.]

We thank the Natural Scientific Foundation Committee of Shanxi Province (No. 20041031) and the University of Malaya for generously supporting this study.

References

Bruker (2002). *SADABS*, *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

Hou, Y., Wei, Y.-G., Shen, E.-H., Xiao, D.-R., Wang, E.-B., Wang, S.-T., Lia, Y.-G., Xu, L. & Hu, C.-W. (2004). *Inorg. Chem. Commun.* **7**, 128–130.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Ng, S. W. & Hu, S.-Z. (2004). *J. Solid State Chem.* **177**, 1780.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Worzala, H., Goetze, T., Fratzky, D. & Miesel, M. (1998). *Acta Cryst.* C**54**, 283–285.