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Redetermination of poly[hexa-μ_2-oxo-tetraoxo-bis(1,10-phenanthroline)tetravanadium(V)]

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.030
wR factor = 0.081
Data-to-parameter ratio = 12.5

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

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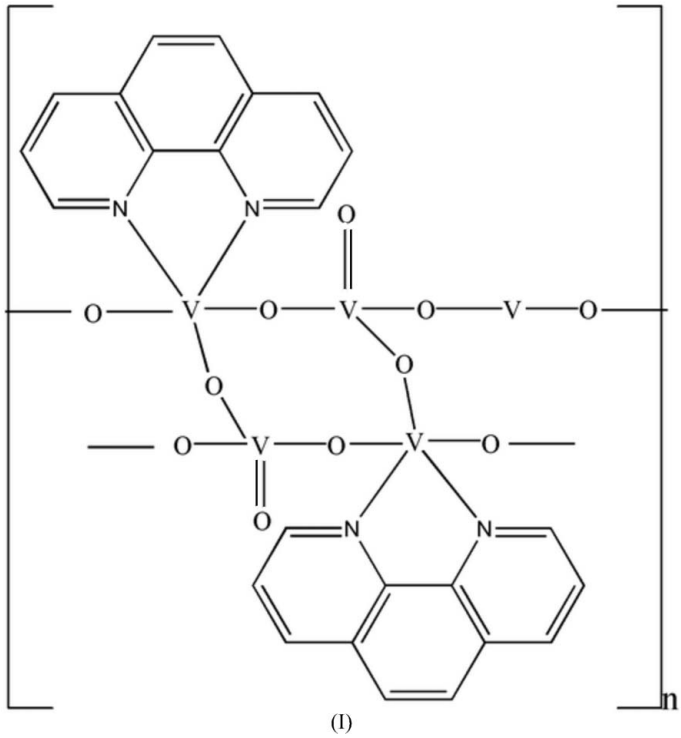
The low-temperature study of the title compound, $[V_4O_{10}(C_{12}H_8N_2)_2]_n$, confirms the $P2_1/m$ space group revision made by Ng & Hu (2004) [*J. Solid State Chem.* **177**, 1780]. Almost all atoms lie on mirror planes.

Comment

The crystal structure of (I) has been described in detail previously, although in an incorrect space group (Li, Wang, Zhang *et al.*, 2002; Li, Wang, Wang *et al.*, 2002). The space group was revised to $P2_1/m$ (Ng & Hu, 2004), and this is confirmed by the present low-temperature study. The compound exists as a linear chain, in which all atoms except the O atoms linking V atoms along the chain, lie on mirror planes (Fig. 1).



(I)

Experimental

The title compound, (I), was the unexpected product from a hydrothermal synthesis. The reactants $K_2(H_4V_{10}O_{28})\cdot 7H_2O$ (0.583 g, 0.5 mmol), $Co(NO_3)_3\cdot 6H_2O$ (0.115 g, 0.5 mmol), 1,10-phen (0.198 g, 1 mmol), and H_2O (9.0 g, 0.5 mmol), in a 1:1:2:100 molar ratio, were first mixed to give a suspension. Sufficient HNO_3 was added to give a pH of 4.5. The mixture was sealed in a 20 ml Teflon-lined reactor and

heated at 433 K for 4 d. The reactor was cooled to room temperature at a rate of 6 K h^{-1}. Red prism-shaped **[prism below?]** crystals were obtained in 35% yield (based on vanadium).

Crystal data

$[V_4O_{10}(C_{12}H_8N_2)_2]$
M_r = 724.16
Monoclinic, $P2_1/m$
a = 9.8024 (7) Å
b = 6.5184 (4) Å
c = 19.8144 (13) Å
β = 100.650 (2)°
V = 1244.25 (14) Å^3
Z = 2
D_x = 1.933 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.53 mm^{-1}
T = 173 (2) K
Prism, red
0.22 × 0.20 × 0.15 mm

Data collection

Rigaku Mercury70 diffractometer
φ and ω scans
Absorption correction: multi-scan (*XEMP* in *SHELXTL*; Siemens, 1994)
T_{min} = 0.720, T_{max} = 0.798
9785 measured reflections
3094 independent reflections
2772 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.081
S = 1.05
3094 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.7806P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 1
Selected bond lengths (Å).

V1—O1	1.596 (2)	V1—O3^{i}	1.8851 (13)
V1—O2	1.7987 (19)	V1—N1	2.186 (2)
V1—O3	1.8851 (13)	V1—N2	2.307 (2)

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

The positions of all H atoms were positioned geometrically (C—H = 0.95 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

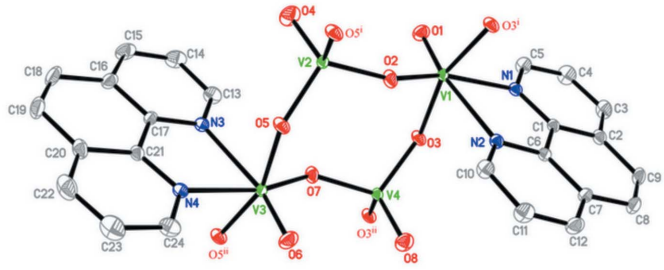


Figure 1
The cordination environment of (I), showing the atom labeling and displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted. [Symmetry codes: (i) $x, \frac{1}{2}-y, z$; (ii) $x, \frac{3}{2}-y, z$.]

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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References

Li, Y. G., Wang, E. B., Wang, S. T., Lu, Y., Hu, C. W., Hu, N. H. & Jia, H. Q. (2002). *J. Mol. Struct.* **202**, 175–180.
Li, Y. G., Wang, E. B., Zhang, H., Luan, G. Y., Hu, C. W., Hu, N. H. & Jia, H. Q. (2002). *J. Solid State Chem.* **163**, 10–16.
Ng, S. W. & Hu, S. Z. (2004). *J. Solid State Chem.* **177**, 1780.
Rigaku/MSC (2001). *CrystalClear*. Version 1.3. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Siemens (1994). *SHELXTL*. Version 5. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.