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Di-μ-oxido-bis[(4-formyl-2-methoxyphenolato-κO^1)oxido(1,10-phenanthroline-$\kappa^2 N,N'$)vanadium(V)]

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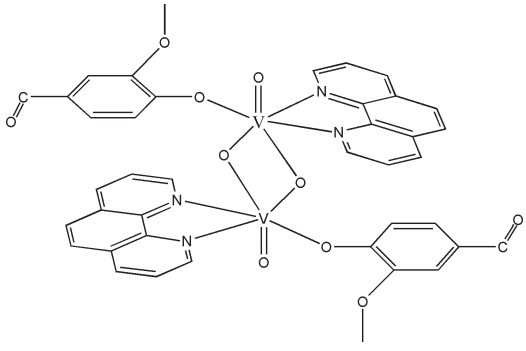
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.061; wR factor = 0.177; data-to-parameter ratio = 12.0.

The title complex, $[V_2(C_8H_7O_3)_2O_4(C_{12}H_8N_2)_2]$, is a centrosymmetric dimer formed by two V^V complex units bridged by two μ_2-oxido groups. The V^V atom is six-coordinated by three oxide O atoms, one O atom from a vanillinate ligand and two N atoms from a 1,10-phenanthroline ligand in a significantly distorted octahedral geometry. In the crystal structure, weak intermolecular C—H···O hydrogen bonds connect the molecules into a three-dimensional network.

Related literature

For general background to vanadium complexes, see: Dong *et al.* (2000); Thompson *et al.* (1999); Yuan *et al.* (2003). For related structures, see: Li *et al.* (2004); Mokry & Carrano (1993).



Experimental

Crystal data

$[V_2(C_8H_7O_3)_2O_4(C_{12}H_8N_2)_2]$
M_r = 828.56
Triclinic, $P\overline{1}$
a = 9.3453 (18) Å
b = 9.786 (2) Å
c = 11.090 (3) Å
α = 80.097 (2)°
β = 65.672 (1)°
γ = 71.535 (1)°
V = 875.6 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 298 K
0.21 × 0.18 × 0.17 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.884, T_{max} = 0.904
4641 measured reflections
3038 independent reflections
2372 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.177
S = 1.07
3038 reflections
254 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.82 e Å^{-3}

Table 1
Selected bond lengths (Å).

V1—O3	1.898 (3)	V1—O4^{i}	2.346 (3)
V1—O4	1.657 (3)	V1—N1	2.148 (3)
V1—O5	1.610 (3)	V1—N2	2.245 (3)

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10···O1^{ii}	0.93	2.48	3.393 (6)	168
C16—H16···O4^{iii}	0.93	2.44	3.192 (5)	138
C8—H8A···O5^{iv}	0.96	2.68	3.280 (6)	121
C11—H11···O5^{v}	0.93	2.67	3.312 (5)	127
C1—H1···O5^{vi}	0.93	2.62	3.441 (6)	148

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2219).

References

Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dong, Y. H., Narla, R. K., Sudbeck, E. & Uckun, F. M. (2000). *J. Inorg. Biochem.* **78**, 321–330.
Li, L. Z., Xu, T., Wang, D. Q. & Ji, H. W. (2004). *Chin. J. Inorg. Chem.* **20**, 236–240.
Mokry, L. M. & Carrano, C. J. (1993). *Inorg. Chem.* **32**, 6119–6121.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Thompson, K. H., McNeill, J. H. & Orvig, C. (1999). *Chem. Rev.* **99**, 2561–2571.
Yuan, M., Wang, E., Lu, Y., Wang, S., Li, Y., Wang, L. & Hu, C. (2003). *Inorg. Chim. Acta*, **344**, 257–261.

supplementary materials

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Di-μ-oxido-bis[(4-formyl-2-methoxyphenolato-κO^1)oxido(1,10-phenanthroline-$\kappa^2 N,N'$)vanadium(V)]

Z. Guo, L. Li, T. Xu and J. Li

Comment

There is an increased interest in vanadium complexes due to their possible uses in pharmaceuticals for the treatment of diabetes (Thompson *et al.*, 1999) and their practical applications in catalysis and material science (Yuan *et al.*, 2003). The vanadium complexes with 1,10-phenanthroline ligand have been reported to exhibit potent antitumor activity (Dong *et al.*, 2000). Vanillin is an useful organic compound with multifunctional groups including aldehyde, ether and phenol. In an effort to uncover the chemistry and biochemistry of vanadium with nitrogen- and oxygen-containing ligands, we report herein the synthesis and crystal structure of a new binuclear vanadium(V) complexes with mixed ligands of vanillin and 1,10-phenanthroline.

The molecular structure of the title complex is shown in Fig.1. In the presence of atmosphere, V^{IV} is oxidized to V^{V}. The complex is centrosymmetric dimer formed by two V^{V} complex untis bridged by two μ_2-oxido groups. The V^{V} atom is six-coordinated by three oxido O atoms, one O atom from a vanillinate ligand and two N atoms from a 1,10-phenanthroline ligand in a significantly distorted octahedral geometry (Table 1). O3, O4, N1 and N2 are situated in the equatorial plane and O5 and $O4^{i}$ [symmetry code: (i) -x+2, -y+2, -z+1] are in the axial positions. The V^{V} atom deviates from the least-squares plane of O3, O4, N1 and N2 by 0.319 (1) Å. In the complex, V1—O4 is 1.657 (3) Å and V1—$O4^{i}$ is 2.346 (3) Å, which illustrates that it is a very asymmetric bridge. The asymmetric structure is similar to that previously reported (Li *et al.*, 2004; Mokry & Carrano, 1993).

There are extensive C—H···O hydrogen bonds in the crystal structure (Table 2). As shown in Fig. 2, the neighboring binuclear complex molecules are connected by the intermolecular hydrogen bonds into a three-dimensional network.

Experimental

Vanillin (0.152 g, 1 mmol) was dissolved in 5 ml absolute methanol and vanadyl sulfate hydrate (0.225 g, 1 mmol) was added to the solution, which was stirred and refluxed for 2 h at 323 K. Then, a methanol solution (5 ml) of 1,10-phenanthroline (0.198 g, 1 mmol) was added to the solution. The mixture was stirred and refluxed for 3 h at 323 K. The obtained brown solution was cooled to room temperature and filtered. The filtrate was kept at room temperature for 30 d. The crystals suitable for X-ray diffraction were obtained.

Refinement

H atoms were placed in geometrically calculated positions and allowed to ride on their parent atoms, with C—H = 0.93 (CH) and 0.96 (CH_3) Å and with U_{iso}(H) = 1.2(or 1.5 for methyl)U_{eq}(C).

Figures

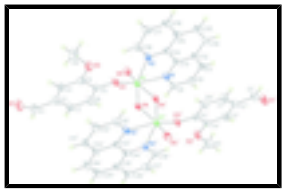


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids. [Symmetry code: (i) -x+2, -y+2, -z+1.]

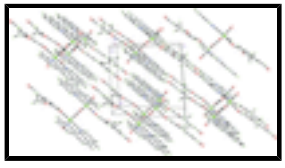


Fig. 2. The crystal packing of the title compound with hydrogen bonds (dashed lines).

Di-μ-oxido-bis[(4-formyl-2-methoxyphenolato-κO^1)oxido(1,10- phenanthroline-κ$^2N,N'$)vanadium(V)]

Crystal data

$[V_2(C_8H_7O_3)_2O_4(C_{12}H_8N_2)_2]$	$Z = 1$
$M_r = 828.56$	$F_{000} = 424$
Triclinic, $P\overline{1}$	$D_x = 1.571$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.3453$ (18) Å	Cell parameters from 943 reflections
$b = 9.786$ (2) Å	$\theta = 2.5$–25.8°
$c = 11.090$ (3) Å	$\mu = 0.60$ mm^{-1}
$\alpha = 80.097$ (2)°	$T = 298$ K
$\beta = 65.672$ (1)°	Needle, colorless
$\gamma = 71.535$ (1)°	0.21 × 0.18 × 0.17 mm
$V = 875.6$ (3) Å^3	

Data collection

Bruker SMART 1000 CCD diffractometer	3038 independent reflections
Radiation source: fine-focus sealed tube	2372 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.046$
$T = 298$ K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 11$
$T_{min} = 0.884$, $T_{max} = 0.904$	$k = -11 \rightarrow 8$
4641 measured reflections	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.061$

$wR(F^2) = 0.177$

$S = 1.07$

3038 reflections

254 parameters

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1054P)^2 + 0.2207P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.84$ e Å^{-3}

$\Delta\rho_{min} = -0.81$ e Å^{-3}

Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.93548 (8)	0.89398 (7)	0.61983 (7)	0.0324 (3)
N1	1.0193 (4)	0.7386 (3)	0.4733 (3)	0.0337 (7)
N2	0.7109 (4)	0.8658 (3)	0.6109 (3)	0.0329 (7)
O1	0.6378 (5)	1.4243 (4)	1.1968 (4)	0.0683 (10)
O2	0.4949 (4)	1.1848 (4)	0.8894 (3)	0.0544 (9)
O3	0.7773 (3)	1.0430 (3)	0.7319 (3)	0.0385 (7)
O4	1.1051 (3)	0.9466 (3)	0.5544 (3)	0.0340 (6)
O5	0.9616 (4)	0.7689 (3)	0.7300 (3)	0.0439 (7)
C1	0.7557 (7)	1.3424 (6)	1.1248 (5)	0.0544 (12)
H1	0.8544	1.3312	1.1321	0.065*
C2	0.7590 (6)	1.2582 (5)	1.0265 (5)	0.0448 (11)
C3	0.6170 (5)	1.2648 (5)	1.0102 (4)	0.0434 (10)
H3	0.5168	1.3212	1.0643	0.052*
C4	0.6250 (5)	1.1877 (5)	0.9139 (4)	0.0366 (9)
C5	0.7772 (5)	1.1044 (4)	0.8284 (4)	0.0350 (9)
C6	0.9175 (5)	1.0962 (5)	0.8477 (5)	0.0435 (10)
H6	1.0182	1.0399	0.7942	0.052*
C7	0.9070 (6)	1.1723 (5)	0.9469 (5)	0.0465 (11)
H7	1.0011	1.1654	0.9601	0.056*
C8	0.3380 (6)	1.2439 (6)	0.9822 (6)	0.0684 (16)
H8A	0.3289	1.1997	1.0684	0.103*
H8B	0.2582	1.2264	0.9578	0.103*
H8C	0.3195	1.3459	0.9840	0.103*
C9	1.1750 (5)	0.6719 (4)	0.4093 (4)	0.0379 (9)
H9	1.2535	0.6951	0.4269	0.046*
C10	1.2269 (5)	0.5683 (4)	0.3167 (4)	0.0423 (10)
H10	1.3373	0.5232	0.2742	0.051*
C11	1.1117 (6)	0.5345 (4)	0.2897 (4)	0.0448 (11)
H11	1.1438	0.4656	0.2287	0.054*
C12	0.9468 (5)	0.6033 (4)	0.3537 (4)	0.0398 (10)
C13	0.9066 (5)	0.7047 (4)	0.4456 (4)	0.0301 (8)
C14	0.7401 (5)	0.7743 (4)	0.5200 (4)	0.0326 (9)
C15	0.6154 (5)	0.7467 (5)	0.4962 (5)	0.0419 (10)
C16	0.4555 (5)	0.8190 (5)	0.5727 (5)	0.0503 (12)
H16	0.3683	0.8040	0.5616	0.060*
C17	0.4273 (5)	0.9118 (5)	0.6636 (5)	0.0491 (11)

H17	0.3208	0.9608	0.7142	0.059*
C18	0.5583 (5)	0.9333 (5)	0.6811 (4)	0.0407 (10)
H18	0.5375	0.9967	0.7437	0.049*
C19	0.8183 (6)	0.5772 (5)	0.3311 (5)	0.0510 (12)
H19	0.8438	0.5120	0.2686	0.061*
C20	0.6622 (6)	0.6447 (5)	0.3981 (5)	0.0519 (12)
H20	0.5813	0.6254	0.3809	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.0238 (4)	0.0321 (4)	0.0453 (4)	−0.0028 (3)	−0.0170 (3)	−0.0116 (3)
N1	0.0333 (18)	0.0263 (17)	0.0485 (19)	−0.0051 (14)	−0.0234 (15)	−0.0047 (14)
N2	0.0259 (17)	0.0315 (18)	0.0425 (19)	−0.0053 (14)	−0.0152 (14)	−0.0043 (14)
O1	0.067 (3)	0.074 (3)	0.067 (2)	−0.010 (2)	−0.023 (2)	−0.037 (2)
O2	0.0297 (16)	0.074 (2)	0.063 (2)	−0.0057 (15)	−0.0158 (15)	−0.0325 (17)
O3	0.0272 (14)	0.0411 (16)	0.0473 (17)	0.0006 (12)	−0.0148 (12)	−0.0200 (13)
O4	0.0232 (13)	0.0363 (15)	0.0449 (15)	−0.0048 (11)	−0.0131 (12)	−0.0143 (12)
O5	0.0441 (17)	0.0433 (17)	0.0491 (17)	−0.0068 (14)	−0.0247 (14)	−0.0059 (14)
C1	0.061 (3)	0.060 (3)	0.050 (3)	−0.020 (3)	−0.025 (3)	−0.009 (2)
C2	0.050 (3)	0.042 (3)	0.050 (3)	−0.012 (2)	−0.025 (2)	−0.007 (2)
C3	0.038 (2)	0.045 (3)	0.044 (2)	−0.0042 (19)	−0.0143 (19)	−0.013 (2)
C4	0.033 (2)	0.040 (2)	0.037 (2)	−0.0078 (18)	−0.0139 (17)	−0.0046 (18)
C5	0.036 (2)	0.035 (2)	0.033 (2)	−0.0100 (18)	−0.0114 (17)	−0.0038 (17)
C6	0.035 (2)	0.041 (2)	0.057 (3)	0.0017 (18)	−0.025 (2)	−0.014 (2)
C7	0.041 (3)	0.051 (3)	0.058 (3)	−0.010 (2)	−0.028 (2)	−0.007 (2)
C8	0.035 (3)	0.086 (4)	0.079 (4)	−0.006 (3)	−0.013 (3)	−0.033 (3)
C9	0.030 (2)	0.032 (2)	0.052 (2)	−0.0033 (17)	−0.0182 (19)	−0.0039 (18)
C10	0.038 (2)	0.031 (2)	0.049 (2)	0.0012 (18)	−0.013 (2)	−0.0095 (19)
C11	0.058 (3)	0.030 (2)	0.045 (2)	−0.007 (2)	−0.019 (2)	−0.0094 (19)
C12	0.048 (3)	0.030 (2)	0.048 (2)	−0.0098 (19)	−0.026 (2)	−0.0034 (18)
C13	0.030 (2)	0.030 (2)	0.033 (2)	−0.0093 (16)	−0.0142 (16)	−0.0009 (16)
C14	0.028 (2)	0.030 (2)	0.044 (2)	−0.0087 (16)	−0.0190 (17)	0.0004 (17)
C15	0.040 (2)	0.038 (2)	0.063 (3)	−0.0146 (19)	−0.034 (2)	0.006 (2)
C16	0.038 (3)	0.052 (3)	0.076 (3)	−0.018 (2)	−0.037 (2)	0.005 (2)
C17	0.027 (2)	0.052 (3)	0.068 (3)	−0.009 (2)	−0.020 (2)	−0.002 (2)
C18	0.027 (2)	0.040 (2)	0.053 (3)	−0.0036 (18)	−0.0163 (19)	−0.004 (2)
C19	0.060 (3)	0.049 (3)	0.064 (3)	−0.016 (2)	−0.037 (3)	−0.014 (2)
C20	0.057 (3)	0.051 (3)	0.071 (3)	−0.020 (2)	−0.043 (3)	−0.002 (2)

Geometric parameters (Å, °)

V1—O3	1.898 (3)	C7—H7	0.9300
V1—O4	1.657 (3)	C8—H8A	0.9600
V1—O5	1.610 (3)	C8—H8B	0.9600
V1—O4^{i}	2.346 (3)	C8—H8C	0.9600
V1—N1	2.148 (3)	C9—C10	1.398 (6)
V1—N2	2.245 (3)	C9—H9	0.9300

N1—C9	1.324 (5)	C10—C11	1.372 (6)
N1—C13	1.355 (5)	C10—H10	0.9300
N2—C18	1.319 (5)	C11—C12	1.392 (6)
N2—C14	1.350 (5)	C11—H11	0.9300
O1—C1	1.196 (6)	C12—C13	1.400 (6)
O2—C4	1.359 (5)	C12—C19	1.426 (6)
O2—C8	1.403 (6)	C13—C14	1.425 (6)
O3—C5	1.314 (5)	C14—C15	1.407 (5)
O4—V1^{i}	2.346 (3)	C15—C16	1.395 (7)
C1—C2	1.459 (6)	C15—C20	1.440 (7)
C1—H1	0.9300	C16—C17	1.362 (7)
C2—C7	1.383 (7)	C16—H16	0.9300
C2—C3	1.393 (6)	C17—C18	1.398 (6)
C3—C4	1.375 (6)	C17—H17	0.9300
C3—H3	0.9300	C18—H18	0.9300
C4—C5	1.416 (6)	C19—C20	1.335 (7)
C5—C6	1.390 (6)	C19—H19	0.9300
C6—C7	1.385 (6)	C20—H20	0.9300
C6—H6	0.9300		
O5—V1—O4	105.63 (14)	C6—C7—H7	119.4
O5—V1—O3	99.73 (14)	O2—C8—H8A	109.5
O4—V1—O3	105.13 (13)	O2—C8—H8B	109.5
O5—V1—N1	91.67 (14)	H8A—C8—H8B	109.5
O4—V1—N1	93.98 (13)	O2—C8—H8C	109.5
O3—V1—N1	154.01 (13)	H8A—C8—H8C	109.5
O5—V1—N2	99.66 (13)	H8B—C8—H8C	109.5
O4—V1—N2	152.20 (13)	N1—C9—C10	123.1 (4)
O3—V1—N2	81.37 (12)	N1—C9—H9	118.5
N1—V1—N2	73.69 (12)	C10—C9—H9	118.5
O5—V1—O4^{i}	172.60 (13)	C11—C10—C9	118.7 (4)
O4—V1—O4^{i}	77.95 (12)	C11—C10—H10	120.6
O3—V1—O4^{i}	85.35 (11)	C9—C10—H10	120.6
N1—V1—O4^{i}	81.55 (11)	C10—C11—C12	120.0 (4)
N2—V1—O4^{i}	75.66 (10)	C10—C11—H11	120.0
C9—N1—C13	117.7 (3)	C12—C11—H11	120.0
C9—N1—V1	123.9 (3)	C11—C12—C13	117.1 (4)
C13—N1—V1	118.3 (3)	C11—C12—C19	124.3 (4)
C18—N2—C14	118.8 (3)	C13—C12—C19	118.6 (4)
C18—N2—V1	126.4 (3)	N1—C13—C12	123.4 (4)
C14—N2—V1	114.8 (2)	N1—C13—C14	116.2 (3)
C4—O2—C8	118.0 (4)	C12—C13—C14	120.4 (3)
C5—O3—V1	132.1 (3)	N2—C14—C15	123.2 (4)
V1—O4—V1^{i}	102.05 (12)	N2—C14—C13	117.0 (3)
O1—C1—C2	126.1 (5)	C15—C14—C13	119.9 (4)
O1—C1—H1	117.0	C16—C15—C14	116.6 (4)
C2—C1—H1	117.0	C16—C15—C20	125.5 (4)
C7—C2—C3	119.6 (4)	C14—C15—C20	117.9 (4)

C7—C2—C1	119.1 (4)	C17—C16—C15	119.8 (4)
C3—C2—C1	121.3 (4)	C17—C16—H16	120.1
C4—C3—C2	119.9 (4)	C15—C16—H16	120.1
C4—C3—H3	120.1	C16—C17—C18	120.0 (4)
C2—C3—H3	120.1	C16—C17—H17	120.0
O2—C4—C3	125.1 (4)	C18—C17—H17	120.0
O2—C4—C5	114.2 (4)	N2—C18—C17	121.6 (4)
C3—C4—C5	120.7 (4)	N2—C18—H18	119.2
O3—C5—C6	123.8 (4)	C17—C18—H18	119.2
O3—C5—C4	117.4 (4)	C20—C19—C12	121.2 (4)
C6—C5—C4	118.8 (4)	C20—C19—H19	119.4
C7—C6—C5	119.8 (4)	C12—C19—H19	119.4
C7—C6—H6	120.1	C19—C20—C15	121.9 (4)
C5—C6—H6	120.1	C19—C20—H20	119.1
C2—C7—C6	121.2 (4)	C15—C20—H20	119.1
C2—C7—H7	119.4		
O5—V1—N1—C9	−77.6 (3)	C3—C4—C5—C6	3.1 (6)
O4—V1—N1—C9	28.2 (3)	O3—C5—C6—C7	175.7 (4)
O3—V1—N1—C9	166.0 (3)	C4—C5—C6—C7	−1.8 (6)
N2—V1—N1—C9	−177.1 (3)	C3—C2—C7—C6	2.3 (7)
$O4^{i}$—V1—N1—C9	105.4 (3)	C1—C2—C7—C6	−176.7 (4)
O5—V1—N1—C13	101.4 (3)	C5—C6—C7—C2	−0.9 (7)
O4—V1—N1—C13	−152.8 (3)	C13—N1—C9—C10	−0.7 (6)
O3—V1—N1—C13	−15.1 (5)	V1—N1—C9—C10	178.3 (3)
N2—V1—N1—C13	1.8 (3)	N1—C9—C10—C11	0.5 (6)
$O4^{i}$—V1—N1—C13	−75.6 (3)	C9—C10—C11—C12	0.2 (6)
O5—V1—N2—C18	91.8 (3)	C10—C11—C12—C13	−0.7 (6)
O4—V1—N2—C18	−112.8 (4)	C10—C11—C12—C19	179.1 (4)
O3—V1—N2—C18	−6.6 (3)	C9—N1—C13—C12	0.1 (6)
N1—V1—N2—C18	−179.2 (4)	V1—N1—C13—C12	−178.9 (3)
$O4^{i}$—V1—N2—C18	−94.0 (3)	C9—N1—C13—C14	177.6 (3)
O5—V1—N2—C14	−90.9 (3)	V1—N1—C13—C14	−1.4 (4)
O4—V1—N2—C14	64.4 (4)	C11—C12—C13—N1	0.5 (6)
O3—V1—N2—C14	170.6 (3)	C19—C12—C13—N1	−179.2 (4)
N1—V1—N2—C14	−2.0 (3)	C11—C12—C13—C14	−176.8 (4)
$O4^{i}$—V1—N2—C14	83.2 (3)	C19—C12—C13—C14	3.4 (6)
O5—V1—O3—C5	55.9 (3)	C18—N2—C14—C15	−0.1 (6)
O4—V1—O3—C5	−53.3 (3)	V1—N2—C14—C15	−177.6 (3)
N1—V1—O3—C5	170.7 (3)	C18—N2—C14—C13	179.5 (3)
N2—V1—O3—C5	154.3 (3)	V1—N2—C14—C13	2.0 (4)
$O4^{i}$—V1—O3—C5	−129.5 (3)	N1—C13—C14—N2	−0.5 (5)
O5—V1—O4—$V1^{i}$	173.33 (13)	C12—C13—C14—N2	177.1 (3)
O3—V1—O4—$V1^{i}$	−81.74 (13)	N1—C13—C14—C15	179.1 (3)
N1—V1—O4—$V1^{i}$	80.47 (12)	C12—C13—C14—C15	−3.3 (6)
N2—V1—O4—$V1^{i}$	18.6 (3)	N2—C14—C15—C16	−0.3 (6)
$O4^{i}$—V1—O4—$V1^{i}$	0.0	C13—C14—C15—C16	−179.9 (4)
O1—C1—C2—C7	177.4 (5)	N2—C14—C15—C20	−178.9 (4)

O1—C1—C2—C3	−1.6 (8)	C13—C14—C15—C20	1.5 (6)
C7—C2—C3—C4	−1.0 (7)	C14—C15—C16—C17	0.6 (6)
C1—C2—C3—C4	178.0 (4)	C20—C15—C16—C17	179.2 (4)
C8—O2—C4—C3	−11.6 (7)	C15—C16—C17—C18	−0.6 (7)
C8—O2—C4—C5	169.6 (4)	C14—N2—C18—C17	0.2 (6)
C2—C3—C4—O2	179.5 (4)	V1—N2—C18—C17	177.3 (3)
C2—C3—C4—C5	−1.7 (6)	C16—C17—C18—N2	0.2 (7)
V1—O3—C5—C6	17.7 (6)	C11—C12—C19—C20	178.5 (4)
V1—O3—C5—C4	−164.7 (3)	C13—C12—C19—C20	−1.7 (7)
O2—C4—C5—O3	4.3 (5)	C12—C19—C20—C15	−0.1 (8)
C3—C4—C5—O3	−174.6 (3)	C16—C15—C20—C19	−178.3 (5)
O2—C4—C5—C6	−178.0 (4)	C14—C15—C20—C19	0.2 (7)

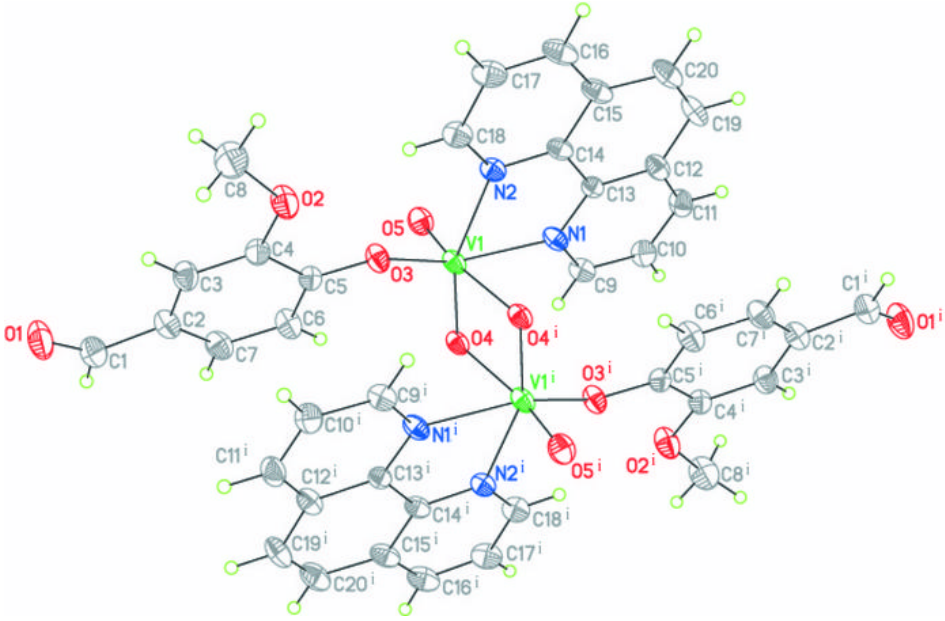
Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10···O1^{ii}	0.93	2.48	3.393 (6)	168
C16—H16···O4^{iii}	0.93	2.44	3.192 (5)	138
C8—H8A···O5^{iv}	0.96	2.68	3.280 (6)	121
C11—H11···O5^{v}	0.93	2.67	3.312 (5)	127
C1—H1···O5^{vi}	0.93	2.62	3.441 (6)	148

Symmetry codes: (ii) *x*+1, *y*−1, *z*−1; (iii) *x*−1, *y*, *z*; (iv) −*x*+1, −*y*+2, −*z*+2; (v) −*x*+2, −*y*+1, −*z*+1; (vi) −*x*+2, −*y*+2, −*z*+2.

Fig. 1


C16
C17
C20
C15
C19
C18
C14
C12
N2
C13
C11
O5
V1
N1
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C8
C4
C3
C5
O3
C9
C1i
C6i
O1i
C7i
C2i
O1
C2
C6
C1
C7
O4
O4i
V1i
O3i
C5i
C3i
C9i
C4i
C10i
N1i
C11i
C12i
C13i
N2i
O5i
O2i
C8i
C14i
C18i
C19i
C15i
C17i
C20i
C16i

Fig. 2