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(Ethanolato-κO)[N'-(3-methoxy-2-oxidobenzylidene-κO^2)benzohydrazidato-$\kappa^2 N',O$]oxidovanadium(V)

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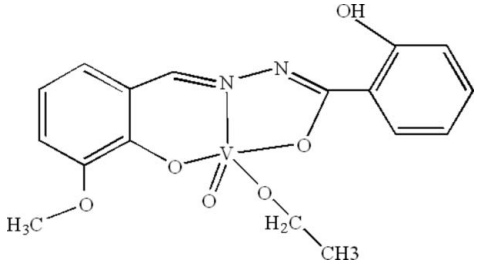
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.105; data-to-parameter ratio = 16.7.

In the title complex, $[V(C_{15}H_{12}N_2O_4)(C_2H_5O)O]$, the V^V ion is coordinated by an oxide O atom, an ethanolate anion and two O atoms and one N atom from the tridentate benzohydrazidate dianion in a distorted square-pyramidal geometry; the V atom is displaced by 0.4748 (8) Å from the basal plane towards the axial oxide O atom. An intramolecular O—H···N hydrogen bond occurs in the benzohydrazidate ligand. Weak intermolecular C—H···O hydrogen bonding is present in the crystal.

Related literature

For general background to the coordination chemistry and biochemisty of vanadium, see: Deng *et al.* (2007); Monfared *et al.* (2011); Sutradhar *et al.* (2006). For related structures, see: Chen *et al.* (2004); Liu *et al.* (2006); Ghosh *et al.* (2007); Seena *et al.* (2008). For the synthesis, see: Gao *et al.* (1998); Huang *et al.* (2010).



Experimental

Crystal data

$[V(C_{15}H_{12}N_2O_4)(C_2H_5O)O]$
M_r = 396.27
Monoclinic, $P2_1/c$
a = 15.808 (5) Å
b = 6.606 (2) Å
c = 16.693 (8) Å
β = 94.107 (16)°
V = 1738.6 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 293 K
0.37 × 0.25 × 0.13 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*TEXRAY*; Molecular Structure Corporation, 1999)
T_{min} = 0.834, T_{max} = 0.924
15371 measured reflections
3968 independent reflections
3243 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.08
3968 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected bond lengths (Å).

V1—N1	2.1029 (15)	V1—O5	1.5762 (15)
V1—O1	1.8325 (14)	V1—O6	1.7423 (13)
V1—O3	1.9453 (14)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4B···N2	0.82	1.86	2.581 (2)	147
C8—H8A···O4^{i}	0.93	2.31	3.236 (2)	177

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

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5587).

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supporting information

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(Ethanolato-κO)[N'-(3-methoxy-2-oxidobenzylidene-κO^2)benzohydrazidato-$\kappa^2 N',O$]oxidovanadium(V)

Xiao-Hua Chen, Qiong-Jie Wu, Li-Juan Chen and Ming-Xing Yang

S1. Comment

In the recent years, the coordination chemistry and biochemisty of vanadium has received considerable attention (Deng *et al.*, 2007; Monfared *et al.*, 2011; Sutradhar *et al.*, 2006). Generally, a tridentate hydrazone ligand is coordinated to the vanadium through O and N atoms, similar to those of the biological system. So, it is important to intensively study the relation ship of the syntheses and structural properties of vanadium hydrazone complexes.

In the title complex, $[VO(C_{15}H_{12}N_2O_4)(C_2H_5O)]$, the V^V ion exists in a distorted square-pyramidal coordination geometry. Three donor atoms (O1, O3 and N1) of the hydrozone ligand and O6 atom from the ethanol group define the coordination basal plane, with a maximum mean plane deviation of 0.030 (1) Å. The V atom is displaced towards the axial oxo O atom by 0.4748 (8) Å from the basal plane. Bond distances (Table 1) and bond angles around V1 atom are compared with those in reported oxovanadium complexes (Chen *et al.*, 2004; Seena *et al.*, 2008; Liu *et al.*,2006; Ghosh *et al.*, 2007). In the crystal structure there are the intramolecular O—H···N hydrogen bonding and intermolecular C—H···O hydrogen bonding (Table 2).

S2. Experimental

$VO(acac)_2$ (acac = acetylacetonate) was synthesized according to the reported method of Gao *et al.* (1998). The synthesis of the hydrazone ligand has already been reported in the literature (Huang *et al.*, 2010).

The title compound was prepared by reacting H_2L (0.1 mmol) with $VO(acac)_2$ (0.1 mmol) in ethanol solvent with stirring. The solution was filtered and allowed to stand at room temperature for one week, and dark-red crystals of complex (I) were obtained.

S3. Refinement

All H atoms were placed in idealized positions and treated as riding with O—H = 0.82 Å, C—H = 0.93–0.97 Å; U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C, O).

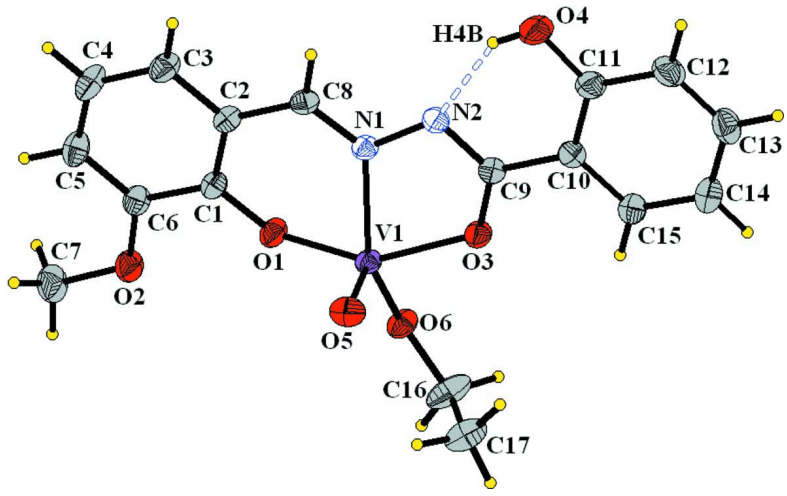

Figure 1

The molecular structure of the title compound, showing 30% probability displacement ellipsoids for non-H atoms. A dashed line indicates the intramoleculat hydrogen bonding.

(Ethanolato-κ*O*)[*N*′-(3-methoxy-2- oxidobenzylidene-κO^2)benzohydrazidato- κ^2*N*′,*O*]oxidovanadium(V)

Crystal data

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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
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c = 16.693 (8) Å
β = 94.107 (16)°
V = 1738.6 (12) Å^3
Z = 4
$F(000)$ = 816
D_x = 1.514 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3243 reflections
θ = 3.3–27.5°
μ = 0.61 mm^{-1}
T = 293 K
Prism, dark-red
0.37 × 0.25 × 0.13 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*TEXRAY*; Molecular Structure Corporation, 1999)
T_{min} = 0.834, T_{max} = 0.924
15371 measured reflections
3968 independent reflections
3243 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −19→20
k = −8→7
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.08
3968 reflections
237 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.3657P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.327165 (17)	0.03858 (5)	0.379657 (17)	0.03227 (11)
O1	0.30812 (8)	0.2237 (2)	0.45811 (8)	0.0442 (3)
O2	0.35894 (10)	0.5229 (2)	0.55498 (9)	0.0496 (4)
O3	0.28941 (8)	−0.2038 (2)	0.32138 (8)	0.0407 (3)
O4	0.04552 (9)	−0.2451 (3)	0.20545 (12)	0.0782 (6)
H4B	0.0647	−0.1583	0.2368	0.117*
O5	0.36775 (9)	0.1609 (2)	0.31098 (9)	0.0516 (4)
O6	0.40591 (8)	−0.1056 (2)	0.43088 (8)	0.0435 (3)
N1	0.19698 (9)	0.0812 (2)	0.34927 (8)	0.0328 (3)
N2	0.15908 (9)	−0.0609 (2)	0.29735 (9)	0.0372 (4)
C1	0.25579 (11)	0.3816 (3)	0.46463 (10)	0.0357 (4)
C2	0.17609 (11)	0.3877 (3)	0.42268 (10)	0.0358 (4)
C3	0.12022 (13)	0.5497 (3)	0.43503 (12)	0.0472 (5)
H3A	0.0668	0.5530	0.4077	0.057*
C4	0.14474 (15)	0.7014 (4)	0.48709 (13)	0.0546 (6)
H4A	0.1078	0.8079	0.4952	0.065*
C5	0.22441 (14)	0.6982 (3)	0.52816 (12)	0.0488 (5)
H5A	0.2402	0.8029	0.5633	0.059*
C6	0.28039 (13)	0.5414 (3)	0.51739 (11)	0.0394 (4)
C7	0.38639 (16)	0.6801 (4)	0.60921 (14)	0.0617 (6)
H7A	0.4451	0.6602	0.6263	0.092*
H7B	0.3531	0.6772	0.6551	0.092*
H7C	0.3795	0.8087	0.5828	0.092*
C8	0.14914 (11)	0.2298 (3)	0.36837 (11)	0.0366 (4)
H8A	0.0938	0.2341	0.3453	0.044*
C9	0.21338 (11)	−0.2052 (3)	0.28567 (10)	0.0342 (4)
C10	0.18783 (11)	−0.3739 (3)	0.23241 (10)	0.0353 (4)
C11	0.10557 (12)	−0.3869 (4)	0.19530 (13)	0.0471 (5)
C12	0.08370 (15)	−0.5527 (4)	0.14672 (14)	0.0570 (6)
H12A	0.0287	−0.5640	0.1234	0.068*

C13	0.14231 (15)	−0.6997 (4)	0.13284 (13)	0.0541 (5)
H13A	0.1269	−0.8090	0.0998	0.065*
C14	0.22383 (15)	−0.6862 (3)	0.16759 (13)	0.0509 (5)
H14A	0.2636	−0.7851	0.1575	0.061*
C15	0.24613 (13)	−0.5261 (3)	0.21720 (11)	0.0409 (4)
H15A	0.3010	−0.5186	0.2411	0.049*
C16	0.47854 (16)	−0.2211 (5)	0.41625 (15)	0.0726 (8)
H16A	0.4651	−0.3632	0.4225	0.087*
H16B	0.5233	−0.1876	0.4569	0.087*
C17	0.51099 (16)	−0.1928 (5)	0.33759 (16)	0.0734 (8)
H17A	0.5644	−0.2613	0.3359	0.110*
H17B	0.5186	−0.0510	0.3279	0.110*
H17C	0.4713	−0.2476	0.2970	0.110*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.02768 (17)	0.03613 (19)	0.03235 (17)	0.00497 (12)	−0.00236 (11)	−0.00102 (12)
O1	0.0396 (7)	0.0454 (8)	0.0458 (7)	0.0120 (6)	−0.0079 (5)	−0.0127 (6)
O2	0.0487 (8)	0.0503 (8)	0.0481 (8)	0.0044 (7)	−0.0073 (6)	−0.0160 (7)
O3	0.0343 (7)	0.0413 (7)	0.0453 (7)	0.0066 (6)	−0.0066 (5)	−0.0083 (6)
O4	0.0364 (8)	0.0960 (14)	0.0991 (14)	0.0110 (9)	−0.0166 (8)	−0.0531 (11)
O5	0.0450 (8)	0.0587 (10)	0.0510 (8)	0.0002 (7)	0.0032 (6)	0.0117 (7)
O6	0.0391 (7)	0.0507 (8)	0.0391 (7)	0.0148 (6)	−0.0069 (5)	−0.0039 (6)
N1	0.0296 (7)	0.0363 (8)	0.0319 (7)	0.0023 (6)	−0.0013 (6)	−0.0012 (6)
N2	0.0315 (8)	0.0398 (9)	0.0393 (8)	0.0004 (7)	−0.0037 (6)	−0.0058 (7)
C1	0.0397 (9)	0.0352 (9)	0.0327 (9)	0.0047 (8)	0.0053 (7)	−0.0010 (7)
C2	0.0365 (9)	0.0382 (10)	0.0329 (9)	0.0065 (8)	0.0047 (7)	0.0011 (7)
C3	0.0441 (11)	0.0514 (13)	0.0454 (11)	0.0150 (9)	−0.0011 (9)	−0.0037 (9)
C4	0.0640 (14)	0.0497 (13)	0.0499 (12)	0.0238 (11)	0.0034 (10)	−0.0062 (10)
C5	0.0661 (13)	0.0419 (11)	0.0384 (10)	0.0080 (10)	0.0030 (9)	−0.0084 (9)
C6	0.0464 (11)	0.0398 (10)	0.0319 (9)	0.0032 (8)	0.0024 (7)	−0.0022 (8)
C7	0.0697 (15)	0.0599 (15)	0.0529 (13)	0.0004 (12)	−0.0131 (11)	−0.0198 (11)
C8	0.0298 (8)	0.0419 (10)	0.0376 (9)	0.0043 (8)	−0.0002 (7)	0.0021 (8)
C9	0.0347 (9)	0.0370 (10)	0.0306 (8)	0.0002 (8)	0.0003 (7)	0.0020 (7)
C10	0.0381 (9)	0.0386 (10)	0.0294 (8)	−0.0028 (8)	0.0037 (7)	0.0005 (7)
C11	0.0383 (10)	0.0558 (12)	0.0471 (11)	−0.0018 (9)	0.0030 (8)	−0.0124 (10)
C12	0.0467 (12)	0.0691 (16)	0.0548 (13)	−0.0144 (11)	0.0012 (10)	−0.0193 (11)
C13	0.0692 (15)	0.0495 (13)	0.0442 (11)	−0.0149 (11)	0.0076 (10)	−0.0117 (9)
C14	0.0667 (14)	0.0398 (11)	0.0464 (11)	0.0052 (10)	0.0061 (10)	−0.0048 (9)
C15	0.0467 (11)	0.0382 (10)	0.0376 (10)	0.0027 (8)	0.0008 (8)	0.0014 (8)
C16	0.0608 (15)	0.094 (2)	0.0636 (15)	0.0445 (15)	0.0118 (12)	0.0132 (14)
C17	0.0587 (15)	0.087 (2)	0.0769 (18)	0.0242 (14)	0.0189 (13)	0.0034 (15)

Geometric parameters (Å, º)

V1—N1	2.1029 (15)	C5—C6	1.382 (3)
V1—O1	1.8325 (14)	C5—H5A	0.9300

V1—O3	1.9453 (14)	C7—H7A	0.9600
V1—O5	1.5762 (15)	C7—H7B	0.9600
V1—O6	1.7423 (13)	C7—H7C	0.9600
O1—C1	1.340 (2)	C8—H8A	0.9300
O2—C6	1.356 (2)	C9—C10	1.464 (3)
O2—C7	1.424 (3)	C10—C15	1.399 (3)
O3—C9	1.303 (2)	C10—C11	1.402 (3)
O4—C11	1.353 (3)	C11—C12	1.391 (3)
O4—H4B	0.8200	C12—C13	1.373 (3)
O6—C16	1.414 (2)	C12—H12A	0.9300
N1—C8	1.293 (2)	C13—C14	1.378 (3)
N1—N2	1.385 (2)	C13—H13A	0.9300
N2—C9	1.307 (2)	C14—C15	1.374 (3)
C1—C2	1.397 (3)	C14—H14A	0.9300
C1—C6	1.411 (3)	C15—H15A	0.9300
C2—C3	1.412 (3)	C16—C17	1.456 (3)
C2—C8	1.427 (3)	C16—H16A	0.9700
C3—C4	1.364 (3)	C16—H16B	0.9700
C3—H3A	0.9300	C17—H17A	0.9600
C4—C5	1.390 (3)	C17—H17B	0.9600
C4—H4A	0.9300	C17—H17C	0.9600

O5—V1—O6	108.88 (8)	O2—C7—H7C	109.5
O5—V1—O1	105.89 (9)	H7A—C7—H7C	109.5
O6—V1—O1	99.34 (7)	H7B—C7—H7C	109.5
O5—V1—O3	100.64 (8)	N1—C8—C2	123.93 (16)
O6—V1—O3	88.75 (6)	N1—C8—H8A	118.0
O1—V1—O3	147.85 (6)	C2—C8—H8A	118.0
O5—V1—N1	101.43 (7)	O3—C9—N2	121.40 (17)
O6—V1—N1	147.57 (7)	O3—C9—C10	119.24 (16)
O1—V1—N1	82.81 (6)	N2—C9—C10	119.35 (16)
O3—V1—N1	74.32 (6)	C15—C10—C11	118.54 (18)
C1—O1—V1	135.34 (12)	C15—C10—C9	120.02 (17)
C6—O2—C7	117.11 (17)	C11—C10—C9	121.44 (17)
C9—O3—V1	118.32 (12)	O4—C11—C12	118.07 (19)
C11—O4—H4B	109.5	O4—C11—C10	122.61 (19)
C16—O6—V1	140.40 (14)	C12—C11—C10	119.3 (2)
C8—N1—N2	115.75 (15)	C13—C12—C11	120.8 (2)
C8—N1—V1	128.45 (12)	C13—C12—H12A	119.6
N2—N1—V1	115.61 (11)	C11—C12—H12A	119.6
C9—N2—N1	109.18 (14)	C12—C13—C14	120.3 (2)
O1—C1—C2	121.40 (17)	C12—C13—H13A	119.8
O1—C1—C6	119.24 (17)	C14—C13—H13A	119.8
C2—C1—C6	119.32 (17)	C15—C14—C13	119.7 (2)
C1—C2—C3	119.96 (18)	C15—C14—H14A	120.1
C1—C2—C8	121.00 (17)	C13—C14—H14A	120.1
C3—C2—C8	119.03 (17)	C14—C15—C10	121.2 (2)
C4—C3—C2	119.83 (19)	C14—C15—H15A	119.4

C4—C3—H3A	120.1	C10—C15—H15A	119.4
C2—C3—H3A	120.1	O6—C16—C17	115.4 (2)
C3—C4—C5	120.60 (19)	O6—C16—H16A	108.4
C3—C4—H4A	119.7	C17—C16—H16A	108.4
C5—C4—H4A	119.7	O6—C16—H16B	108.4
C6—C5—C4	120.9 (2)	C17—C16—H16B	108.4
C6—C5—H5A	119.6	H16A—C16—H16B	107.5
C4—C5—H5A	119.6	C16—C17—H17A	109.5
O2—C6—C5	125.51 (18)	C16—C17—H17B	109.5
O2—C6—C1	115.07 (17)	H17A—C17—H17B	109.5
C5—C6—C1	119.41 (18)	C16—C17—H17C	109.5
O2—C7—H7A	109.5	H17A—C17—H17C	109.5
O2—C7—H7B	109.5	H17B—C17—H17C	109.5
H7A—C7—H7B	109.5		

O5—V1—O1—C1	68.39 (19)	C7—O2—C6—C5	−1.0 (3)
O6—V1—O1—C1	−178.79 (18)	C7—O2—C6—C1	−179.65 (19)
O3—V1—O1—C1	−76.1 (2)	C4—C5—C6—O2	−179.3 (2)
N1—V1—O1—C1	−31.50 (18)	C4—C5—C6—C1	−0.7 (3)
O5—V1—O3—C9	−89.56 (14)	O1—C1—C6—O2	2.8 (3)
O6—V1—O3—C9	161.46 (13)	C2—C1—C6—O2	−179.60 (17)
O1—V1—O3—C9	55.77 (18)	O1—C1—C6—C5	−175.88 (18)
N1—V1—O3—C9	9.46 (12)	C2—C1—C6—C5	1.7 (3)
O5—V1—O6—C16	−37.9 (3)	N2—N1—C8—C2	178.23 (17)
O1—V1—O6—C16	−148.3 (3)	V1—N1—C8—C2	−6.9 (3)
O3—V1—O6—C16	63.0 (3)	C1—C2—C8—N1	−5.5 (3)
N1—V1—O6—C16	120.4 (3)	C3—C2—C8—N1	175.82 (19)
O5—V1—N1—C8	−85.71 (17)	V1—O3—C9—N2	−9.5 (2)
O6—V1—N1—C8	115.25 (17)	V1—O3—C9—C10	171.59 (12)
O1—V1—N1—C8	19.13 (16)	N1—N2—C9—O3	1.2 (2)
O3—V1—N1—C8	176.30 (17)	N1—N2—C9—C10	−179.87 (15)
O5—V1—N1—N2	89.16 (14)	O3—C9—C10—C15	−3.2 (3)
O6—V1—N1—N2	−69.88 (17)	N2—C9—C10—C15	177.87 (17)
O1—V1—N1—N2	−166.00 (13)	O3—C9—C10—C11	177.21 (17)
O3—V1—N1—N2	−8.83 (11)	N2—C9—C10—C11	−1.8 (3)
C8—N1—N2—C9	−177.68 (16)	C15—C10—C11—O4	−178.9 (2)
V1—N1—N2—C9	6.78 (18)	C9—C10—C11—O4	0.7 (3)
V1—O1—C1—C2	29.7 (3)	C15—C10—C11—C12	1.9 (3)
V1—O1—C1—C6	−152.82 (15)	C9—C10—C11—C12	−178.46 (19)
O1—C1—C2—C3	175.72 (18)	O4—C11—C12—C13	178.7 (2)
C6—C1—C2—C3	−1.8 (3)	C10—C11—C12—C13	−2.0 (4)
O1—C1—C2—C8	−3.0 (3)	C11—C12—C13—C14	0.6 (4)
C6—C1—C2—C8	179.54 (17)	C12—C13—C14—C15	0.9 (3)
C1—C2—C3—C4	0.9 (3)	C13—C14—C15—C10	−0.9 (3)
C8—C2—C3—C4	179.6 (2)	C11—C10—C15—C14	−0.5 (3)
C2—C3—C4—C5	0.1 (3)	C9—C10—C15—C14	179.89 (18)
C3—C4—C5—C6	−0.2 (4)	V1—O6—C16—C17	14.1 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*B*···N2	0.82	1.86	2.581 (2)	147
C8—H8*A*···O4^{i}	0.93	2.31	3.236 (2)	177

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