

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

(Methanol- $\kappa$ O)(methanolato- $\kappa$ O)-  
oxido[N-(2-oxidobenzylidene)phenyl-  
alaninato- $\kappa^3$ O,N,O']vanadium(V)

Lin Bian and Lianzhi Li\*

School of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

Correspondence e-mail: lilianzhi1963@yahoo.com.cn

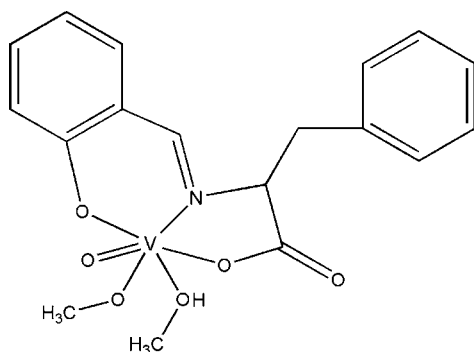
Received 17 January 2011; accepted 24 January 2011

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.008$  Å;  $R$  factor = 0.054;  $wR$  factor = 0.129; data-to-parameter ratio = 13.2.

In the title complex,  $[\text{V}(\text{C}_{16}\text{H}_{13}\text{NO}_3)(\text{CH}_3\text{O})\text{O}(\text{CH}_3\text{OH})]$ , the  $\text{V}^{\text{V}}$  atom is six-coordinated by a tridentate ligand derived from the condensation of salicylaldehyde and L-phenylalanine, a vanadyl O atom, a methanolate O atom and a methanol O atom, forming a distorted octahedral coordination geometry. In the crystal, intermolecular  $\text{O}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds result in a two-dimensional structure parallel to (001).

## Related literature

For general background to the coordination chemistry of vanadium, see: Diego *et al.* (2003); Kenji *et al.* (2000); Thompson *et al.* (1999); Thompson & Orvig (2006); Wikksky *et al.* (2001).



## Experimental

## Crystal data

$[\text{V}(\text{C}_{16}\text{H}_{13}\text{NO}_3)(\text{CH}_3\text{O})\text{O}(\text{CH}_3\text{O})]$   
 $M_r = 397.29$

Orthorhombic,  $P2_12_12$  $a = 14.3095$  (15) Å $b = 18.782$  (2) Å $c = 6.6986$  (7) Å $V = 1800.3$  (3) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 0.59$  mm<sup>-1</sup> $T = 298$  K $0.45 \times 0.42 \times 0.41$  mm

## Data collection

Bruker SMART 1000 CCD diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

 $T_{\text{min}} = 0.779$ ,  $T_{\text{max}} = 0.795$ 

8134 measured reflections

3177 independent reflections

2293 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.073$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$  $wR(F^2) = 0.129$  $S = 1.03$ 

3177 reflections

241 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.46$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.48$  e Å<sup>-3</sup>

Absolute structure: Flack (1983),

1328 Friedel pairs

Flack parameter: 0.04 (4)

Table 1

Selected bond lengths (Å).

V1—O1	1.570 (3)	V1—O5	2.366 (3)
V1—O2	1.934 (3)	V1—O6	1.762 (3)
V1—O4	1.831 (3)	V1—N1	2.086 (4)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O5}-\text{H19}\cdots\text{O3}^{\text{i}}$	1.00 (5)	1.73 (5)	2.687 (4)	161 (4)
$\text{C13}-\text{H13}\cdots\text{O3}^{\text{ii}}$	0.93	2.59	3.500 (7)	165

Symmetry codes: (i)  $-x + 2, -y + 2, z$ ; (ii)  $-x + \frac{3}{2}, y - \frac{1}{2}, -z$ .

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) and DIAMOND (Brandenburg, 1999); software used to prepare material for publication: SHELXTL.

The authors thank the Natural Science Foundation of Shandong Province (No. Y2004B02) for a research grant.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2401).

## References

- Brandenburg, K. (1999). DIAMOND. Crystal Impact GbR, Bonn, Germany.
- Bruker (2007). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Diego, D. R., Agustin, G., Ramon, V., Carlo, M., Andrea, I. & Dante, M. (2003). Dalton Trans. pp. 1813–1820.
- Flack, H. D. (1983). Acta Cryst. A39, 876–881.
- Kenji, K., Makoto, T., Ken, H., Naohisa, Y. & Yoshitane, K. (2000). Inorg. Chim. Acta, 305, 172–183.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
- Thompson, K. H., McNeill, J. H. & Orvig, C. (1999). Chem. Rev. 99, 2561–2571.
- Thompson, K. H. & Orvig, C. (2006). Dalton Trans. pp. 761–764.
- Wikksky, G. R., Goldfine, A. B., Kostyniak, P. J., McNeill, J. H., Yang, L. Q., Khan, H. R. & Crans, D. C. (2001). J. Inorg. Biochem. 85, 33–42.

## supporting information

*Acta Cryst.* (2011). E67, m274 [doi:10.1107/S1600536811003163]

**(Methanol- $\kappa$ O)(methanolato- $\kappa$ O)oxido[*N*-(2-oxidobenzylidene)phenylalaninato- $\kappa^3$ O,*N*,*O*']vanadium(V)**

**Lin Bian and Lianzhi Li**

### S1. Comment

The coordination chemistry of vanadium has been receiving increasing interest since it was found that vanadium compounds in various oxidation states have insulin-mimetic properties (Diego *et al.*, 2003; Kenji *et al.*, 2000; Thompson & Orvig, 2006). Compared with other transition metal complexes, less vanadium complexes have been synthesized and characterized (Thompson *et al.*, 1999; Wikksky *et al.*, 2001). We report here the synthesis and crystal structure of an oxovanadium(V) complex with a tetradentate Schiff-base ligand derived from the condensation of salicylaldehyde and *L*-phenylalanine.

As shown in Fig. 1, the V<sup>V</sup> ion is six-coordinated by the tridentate (O,N,O) donor ligand and three O atoms from an oxido group, a methanolate group and a methanol molecule (Table 1), forming a distorted octahedral geometry. In the complex, O2, N1 and O4 of the tridentate Schiff base ligand and O6 of the methanolate ligand define the equatorial plane and the oxido O1 and the methanol O5 occupy the axial positions. The Schiff base ligand coordinating to the V<sup>V</sup> atom forms two chelating rings, the five-membered V1—O2—C1—C2—N1 ring and the six-membered V1—N1—C10—C11—C16—O4 ring. The dihedral angle of the two rings is 15.69 (15)°, which increases the stability of the complex.

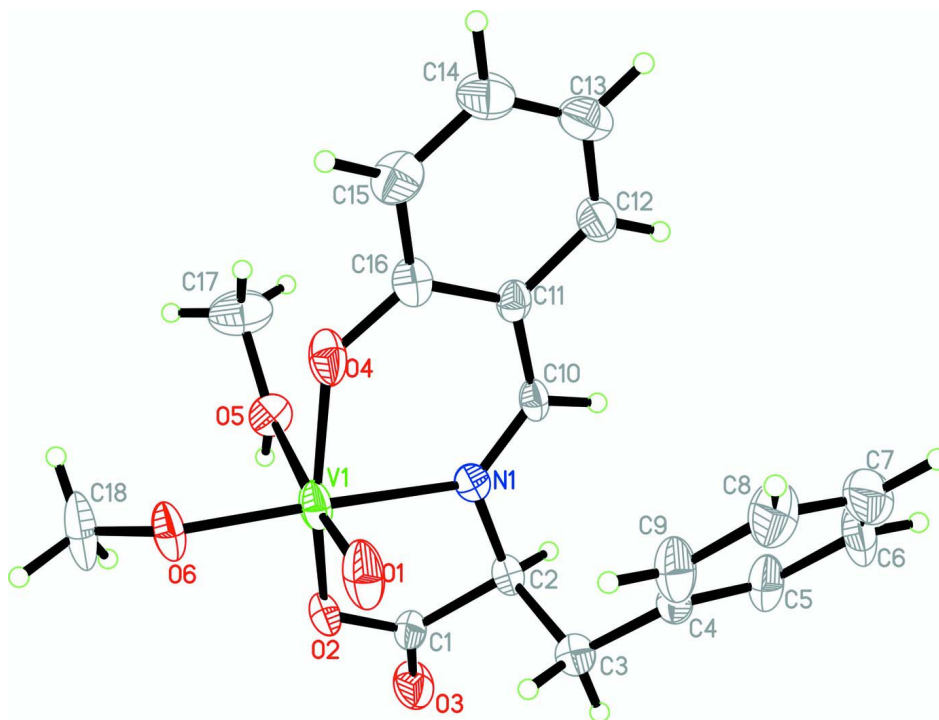
In the crystal, the intermolecular O—H...O and C—H...O hydrogen bonds (Table 2) result in a two-dimensional structure (Fig. 2).

### S2. Experimental

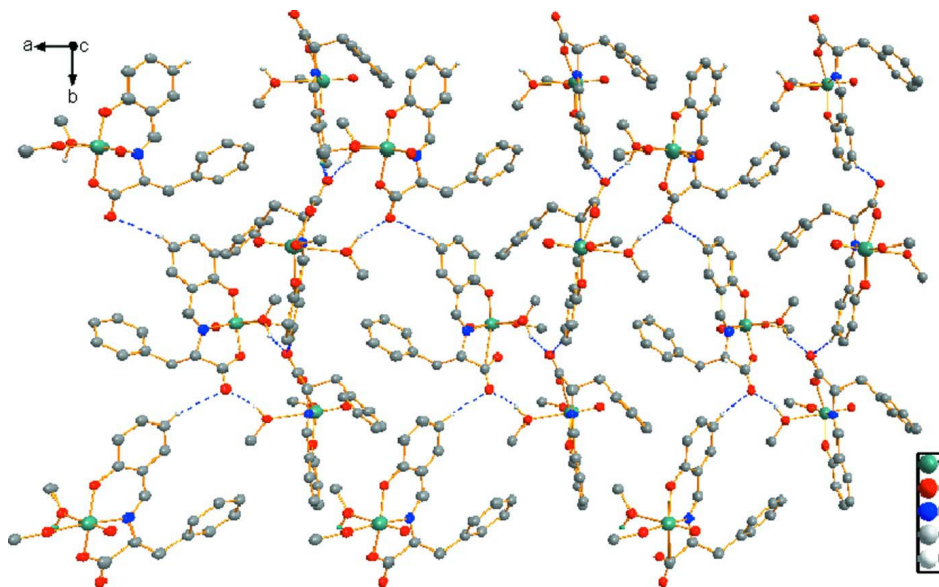
*L*-Phenylalanine (1 mmol, 0.165 g) and potassium hydroxide (1 mmol, 0.056 g) were dissolved in hot methanol (10 ml) with stirring. The mixture was added successively to a methanol solution (5 ml) of salicylaldehyde (1 mmol, 0.11 ml) with stirring at 323 K for 2 h. Subsequently, an aqueous solution (2 ml) of vanadyl sulfate hydrate (1 mmol, 0.225 g) was added dropwise and stirred for 3 h continuously. The resultant solution was filtered and the filtrate was held at room temperature for several days, whereupon red blocky crystals suitable for X-ray diffraction were obtained.

### S3. Refinement

H atoms on C atoms were placed in calculated positions and refined as riding atoms, with C—H = 0.93–0.98 Å and with  $U_{\text{iso}}(\text{H}) = 1.2(1.5 \text{ for methyl})U_{\text{eq}}(\text{C})$ . The hydroxy H atom (H19) of the methanol molecule was located from a difference Fourier map and refined isotropically.

**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

**Figure 2**

The two-dimensional structure of the title compound, linked by intermolecular hydrogen bonds (dashed lines).

(Methanol- $\kappa$ O)(methanolato- $\kappa$ O)oxido[N-(2-oxidobenzylidene)phenylalaninato- $\kappa^3$ O,N,O']vanadium(V)

Crystal data

[V(C<sub>16</sub>H<sub>13</sub>NO<sub>3</sub>)(CH<sub>3</sub>O)O(CH<sub>4</sub>O)]  
*M<sub>r</sub>* = 397.29  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2  
 Hall symbol: P 2 2ab  
*a* = 14.3095 (15) Å  
*b* = 18.782 (2) Å  
*c* = 6.6986 (7) Å  
*V* = 1800.3 (3) Å<sup>3</sup>  
*Z* = 4

*F*(000) = 824  
*D<sub>x</sub>* = 1.466 Mg m<sup>-3</sup>  
 Mo *K*α radiation, λ = 0.71073 Å  
 Cell parameters from 2512 reflections  
 θ = 2.6–23.6°  
 μ = 0.59 mm<sup>-1</sup>  
*T* = 298 K  
 Block, red  
 0.45 × 0.42 × 0.41 mm

Data collection

Bruker SMART 1000 CCD  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 φ and ω scans  
 Absorption correction: multi-scan  
 (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.779, *T<sub>max</sub>* = 0.795

8134 measured reflections  
 3177 independent reflections  
 2293 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.073  
 θ<sub>max</sub> = 25.0°, θ<sub>min</sub> = 1.8°  
*h* = -10→17  
*k* = -22→12  
*l* = -7→7

Refinement

Refinement on *F*<sup>2</sup>  
 Least-squares matrix: full  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.054  
*wR* (*F*<sup>2</sup>) = 0.129  
*S* = 1.03  
 3177 reflections  
 241 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 atoms treated by a mixture of independent  
 and constrained refinement  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0594*P*)<sup>2</sup>]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> < 0.001  
 Δρ<sub>max</sub> = 0.46 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.48 e Å<sup>-3</sup>  
 Absolute structure: Flack (1983), 1328 Friedel  
 pairs  
 Absolute structure parameter: 0.04 (4)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>iso</sub></i> */ <i>U<sub>eq</sub></i>
V1	0.84142 (5)	0.86795 (4)	0.57437 (11)	0.0419 (3)
O1	0.7400 (2)	0.8810 (2)	0.6561 (5)	0.0676 (12)
O2	0.8790 (2)	0.96385 (16)	0.5061 (5)	0.0482 (9)
O3	0.8863 (2)	1.05118 (18)	0.2846 (6)	0.0613 (10)
O4	0.8383 (2)	0.77184 (17)	0.5287 (5)	0.0535 (9)
O5	0.9878 (2)	0.85346 (17)	0.4151 (5)	0.0465 (8)
O6	0.9045 (2)	0.8721 (2)	0.7999 (4)	0.0549 (9)
N1	0.7976 (2)	0.87816 (19)	0.2787 (5)	0.0323 (9)
C1	0.8574 (3)	0.9935 (3)	0.3387 (8)	0.0425 (12)
C2	0.7896 (3)	0.9517 (2)	0.2140 (7)	0.0369 (11)
H2	0.8048	0.9560	0.0719	0.044*
C3	0.6910 (3)	0.9796 (2)	0.2553 (8)	0.0480 (13)

H3A	0.6811	0.9811	0.3985	0.058*
H3B	0.6864	1.0280	0.2057	0.058*
C4	0.6158 (3)	0.9364 (2)	0.1631 (7)	0.0405 (12)
C5	0.5940 (4)	0.9426 (3)	-0.0351 (8)	0.0579 (15)
H5	0.6261	0.9753	-0.1138	0.069*
C6	0.5261 (4)	0.9017 (4)	-0.1182 (10)	0.081 (2)
H6	0.5114	0.9076	-0.2524	0.097*
C7	0.4796 (4)	0.8527 (4)	-0.0100 (13)	0.082 (2)
H7	0.4332	0.8248	-0.0679	0.099*
C8	0.5020 (4)	0.8451 (3)	0.1849 (12)	0.0731 (19)
H8	0.4712	0.8109	0.2609	0.088*
C9	0.5687 (4)	0.8864 (3)	0.2722 (9)	0.0613 (16)
H9	0.5823	0.8805	0.4069	0.074*
C10	0.7750 (3)	0.8287 (2)	0.1591 (7)	0.0338 (10)
H10	0.7585	0.8417	0.0298	0.041*
C11	0.7727 (3)	0.7552 (2)	0.2063 (7)	0.0363 (11)
C12	0.7375 (3)	0.7080 (3)	0.0650 (9)	0.0502 (12)
H12	0.7199	0.7248	-0.0600	0.060*
C13	0.7286 (3)	0.6378 (3)	0.1077 (9)	0.0607 (15)
H13	0.7044	0.6068	0.0124	0.073*
C14	0.7550 (4)	0.6124 (3)	0.2902 (10)	0.0632 (16)
H14	0.7480	0.5643	0.3190	0.076*
C15	0.7917 (4)	0.6572 (3)	0.4311 (10)	0.0575 (14)
H15	0.8104	0.6391	0.5540	0.069*
C16	0.8010 (3)	0.7291 (3)	0.3918 (7)	0.0436 (12)
C17	1.0399 (4)	0.7920 (3)	0.3821 (11)	0.078 (2)
H17A	1.0050	0.7513	0.4264	0.117*
H17B	1.0975	0.7948	0.4551	0.117*
H17C	1.0530	0.7874	0.2422	0.117*
C18	0.9966 (5)	0.8865 (4)	0.8526 (10)	0.096 (2)
H18A	1.0209	0.9233	0.7677	0.143*
H18B	1.0334	0.8441	0.8372	0.143*
H18C	0.9989	0.9019	0.9891	0.143*
H19	1.024 (4)	0.896 (3)	0.376 (8)	0.065 (17)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
V1	0.0372 (4)	0.0665 (5)	0.0222 (4)	-0.0044 (4)	-0.0019 (4)	-0.0005 (4)
O1	0.042 (2)	0.132 (4)	0.0290 (18)	-0.001 (2)	0.0042 (16)	-0.004 (2)
O2	0.050 (2)	0.057 (2)	0.038 (2)	0.0011 (17)	-0.0172 (16)	-0.0117 (15)
O3	0.068 (2)	0.048 (2)	0.068 (3)	-0.0181 (19)	-0.021 (2)	0.0047 (19)
O4	0.060 (2)	0.062 (2)	0.039 (2)	-0.0178 (18)	-0.0140 (19)	0.0158 (16)
O5	0.0393 (18)	0.051 (2)	0.050 (2)	-0.0013 (16)	0.0070 (18)	0.0033 (19)
O6	0.050 (2)	0.089 (3)	0.0254 (17)	-0.008 (2)	-0.0088 (16)	-0.0017 (19)
N1	0.0256 (19)	0.041 (2)	0.030 (2)	-0.0024 (17)	0.0054 (15)	-0.0029 (18)
C1	0.035 (3)	0.050 (3)	0.043 (3)	0.005 (2)	-0.011 (2)	-0.004 (2)
C2	0.036 (3)	0.044 (3)	0.031 (3)	-0.006 (2)	-0.005 (2)	-0.001 (2)

C3	0.045 (3)	0.049 (3)	0.049 (3)	0.001 (2)	-0.005 (3)	-0.005 (2)
C4	0.034 (3)	0.046 (3)	0.042 (3)	0.002 (2)	-0.005 (2)	-0.007 (2)
C5	0.036 (3)	0.088 (4)	0.049 (4)	-0.001 (3)	-0.010 (3)	0.003 (3)
C6	0.053 (4)	0.135 (6)	0.055 (4)	0.002 (4)	-0.024 (3)	-0.025 (4)
C7	0.038 (3)	0.088 (5)	0.121 (7)	-0.007 (3)	-0.009 (4)	-0.038 (4)
C8	0.035 (3)	0.093 (5)	0.091 (5)	-0.011 (3)	-0.001 (4)	0.008 (4)
C9	0.041 (3)	0.097 (5)	0.047 (3)	-0.013 (3)	-0.004 (3)	0.007 (3)
C10	0.027 (2)	0.051 (3)	0.023 (2)	0.000 (2)	-0.0017 (19)	-0.003 (2)
C11	0.028 (3)	0.048 (3)	0.032 (3)	0.002 (2)	0.000 (2)	-0.001 (2)
C12	0.045 (3)	0.056 (3)	0.049 (3)	0.002 (2)	-0.006 (3)	-0.012 (3)
C13	0.050 (3)	0.051 (3)	0.081 (5)	-0.005 (3)	0.006 (3)	-0.017 (4)
C14	0.070 (4)	0.042 (3)	0.077 (5)	-0.014 (3)	0.004 (4)	0.004 (3)
C15	0.058 (3)	0.054 (3)	0.061 (4)	-0.008 (3)	0.005 (3)	0.019 (3)
C16	0.037 (3)	0.056 (3)	0.039 (3)	-0.007 (2)	0.002 (2)	0.003 (2)
C17	0.067 (4)	0.063 (4)	0.105 (6)	0.005 (3)	0.032 (4)	-0.003 (4)
C18	0.078 (5)	0.165 (7)	0.044 (4)	-0.014 (4)	-0.032 (4)	0.001 (4)

*Geometric parameters (Å, °)*

V1—O1	1.570 (3)	C6—H6	0.9300
V1—O2	1.934 (3)	C7—C8	1.352 (9)
V1—O4	1.831 (3)	C7—H7	0.9300
V1—O5	2.366 (3)	C8—C9	1.361 (8)
V1—O6	1.762 (3)	C8—H8	0.9300
V1—N1	2.086 (4)	C9—H9	0.9300
O2—C1	1.289 (5)	C10—C11	1.417 (6)
O3—C1	1.215 (6)	C10—H10	0.9300
O4—C16	1.331 (5)	C11—C12	1.390 (6)
O5—C17	1.392 (6)	C11—C16	1.395 (6)
O5—H19	1.00 (5)	C12—C13	1.356 (6)
O6—C18	1.390 (6)	C12—H12	0.9300
N1—C10	1.269 (5)	C13—C14	1.365 (8)
N1—C2	1.451 (5)	C13—H13	0.9300
C1—C2	1.503 (6)	C14—C15	1.369 (8)
C2—C3	1.531 (6)	C14—H14	0.9300
C2—H2	0.9800	C15—C16	1.382 (6)
C3—C4	1.483 (6)	C15—H15	0.9300
C3—H3A	0.9700	C17—H17A	0.9600
C3—H3B	0.9700	C17—H17B	0.9600
C4—C9	1.368 (7)	C17—H17C	0.9600
C4—C5	1.369 (7)	C18—H18A	0.9600
C5—C6	1.359 (8)	C18—H18B	0.9600
C5—H5	0.9300	C18—H18C	0.9600
C6—C7	1.347 (9)		
O1—V1—O6	99.65 (17)	C7—C6—C5	121.3 (7)
O1—V1—O4	100.91 (19)	C7—C6—H6	119.4
O6—V1—O4	101.54 (16)	C5—C6—H6	119.4

O1—V1—O2	101.17 (18)	C6—C7—C8	118.3 (6)
O6—V1—O2	91.07 (15)	C6—C7—H7	120.8
O4—V1—O2	152.27 (15)	C8—C7—H7	120.8
O1—V1—N1	92.22 (16)	C7—C8—C9	121.4 (6)
O6—V1—N1	164.59 (15)	C7—C8—H8	119.3
O4—V1—N1	85.67 (14)	C9—C8—H8	119.3
O2—V1—N1	76.92 (14)	C8—C9—C4	120.5 (6)
O1—V1—O5	173.31 (16)	C8—C9—H9	119.8
O6—V1—O5	86.45 (14)	C4—C9—H9	119.8
O4—V1—O5	80.40 (14)	N1—C10—C11	125.3 (4)
O2—V1—O5	75.80 (13)	N1—C10—H10	117.3
N1—V1—O5	81.31 (12)	C11—C10—H10	117.3
C1—O2—V1	122.7 (3)	C12—C11—C16	119.2 (5)
C16—O4—V1	136.1 (3)	C12—C11—C10	118.5 (4)
C17—O5—V1	129.8 (3)	C16—C11—C10	122.3 (4)
C17—O5—H19	110 (3)	C13—C12—C11	120.7 (5)
V1—O5—H19	119 (3)	C13—C12—H12	119.7
C18—O6—V1	135.4 (3)	C11—C12—H12	119.7
C10—N1—C2	119.2 (4)	C12—C13—C14	120.1 (5)
C10—N1—V1	127.5 (3)	C12—C13—H13	119.9
C2—N1—V1	113.2 (3)	C14—C13—H13	119.9
O3—C1—O2	124.3 (5)	C13—C14—C15	120.7 (5)
O3—C1—C2	121.3 (4)	C13—C14—H14	119.7
O2—C1—C2	114.4 (4)	C15—C14—H14	119.7
N1—C2—C1	106.3 (4)	C14—C15—C16	120.3 (6)
N1—C2—C3	110.2 (4)	C14—C15—H15	119.8
C1—C2—C3	108.4 (4)	C16—C15—H15	119.8
N1—C2—H2	110.6	O4—C16—C15	119.8 (5)
C1—C2—H2	110.6	O4—C16—C11	121.2 (4)
C3—C2—H2	110.6	C15—C16—C11	119.0 (5)
C4—C3—C2	113.9 (4)	O5—C17—H17A	109.5
C4—C3—H3A	108.8	O5—C17—H17B	109.5
C2—C3—H3A	108.8	H17A—C17—H17B	109.5
C4—C3—H3B	108.8	O5—C17—H17C	109.5
C2—C3—H3B	108.8	H17A—C17—H17C	109.5
H3A—C3—H3B	107.7	H17B—C17—H17C	109.5
C9—C4—C5	117.7 (5)	O6—C18—H18A	109.5
C9—C4—C3	120.8 (5)	O6—C18—H18B	109.5
C5—C4—C3	121.5 (5)	H18A—C18—H18B	109.5
C6—C5—C4	120.8 (6)	O6—C18—H18C	109.5
C6—C5—H5	119.6	H18A—C18—H18C	109.5
C4—C5—H5	119.6	H18B—C18—H18C	109.5
O1—V1—O2—C1	-83.7 (4)	V1—N1—C2—C3	-90.1 (4)
O6—V1—O2—C1	176.2 (4)	O3—C1—C2—N1	158.9 (4)
O4—V1—O2—C1	58.5 (5)	O2—C1—C2—N1	-23.0 (5)
N1—V1—O2—C1	6.0 (3)	O3—C1—C2—C3	-82.7 (6)
O5—V1—O2—C1	90.1 (3)	O2—C1—C2—C3	95.4 (5)

O1—V1—O4—C16	75.3 (5)	N1—C2—C3—C4	-56.0 (6)
O6—V1—O4—C16	177.7 (4)	C1—C2—C3—C4	-171.9 (4)
O2—V1—O4—C16	-66.9 (6)	C2—C3—C4—C9	97.9 (6)
N1—V1—O4—C16	-16.1 (4)	C2—C3—C4—C5	-79.1 (6)
O5—V1—O4—C16	-98.0 (5)	C9—C4—C5—C6	1.8 (8)
O6—V1—O5—C17	85.4 (5)	C3—C4—C5—C6	178.9 (5)
O4—V1—O5—C17	-16.9 (5)	C4—C5—C6—C7	-1.5 (9)
O2—V1—O5—C17	177.4 (5)	C5—C6—C7—C8	0.1 (10)
N1—V1—O5—C17	-104.0 (5)	C6—C7—C8—C9	1.1 (10)
O1—V1—O6—C18	-157.1 (6)	C7—C8—C9—C4	-0.8 (9)
O4—V1—O6—C18	99.6 (6)	C5—C4—C9—C8	-0.6 (8)
O2—V1—O6—C18	-55.6 (6)	C3—C4—C9—C8	-177.7 (5)
N1—V1—O6—C18	-17.2 (10)	C2—N1—C10—C11	-175.2 (4)
O5—V1—O6—C18	20.1 (6)	V1—N1—C10—C11	1.8 (6)
O1—V1—N1—C10	-95.5 (4)	N1—C10—C11—C12	173.7 (4)
O6—V1—N1—C10	124.0 (6)	N1—C10—C11—C16	-3.7 (7)
O4—V1—N1—C10	5.3 (3)	C16—C11—C12—C13	1.5 (7)
O2—V1—N1—C10	163.6 (4)	C10—C11—C12—C13	-175.9 (4)
O5—V1—N1—C10	86.2 (4)	C11—C12—C13—C14	-0.6 (8)
O1—V1—N1—C2	81.7 (3)	C12—C13—C14—C15	-0.7 (8)
O6—V1—N1—C2	-58.8 (7)	C13—C14—C15—C16	1.1 (8)
O4—V1—N1—C2	-177.5 (3)	V1—O4—C16—C15	-162.9 (4)
O2—V1—N1—C2	-19.3 (3)	V1—O4—C16—C11	18.6 (7)
O5—V1—N1—C2	-96.6 (3)	C14—C15—C16—O4	-178.8 (5)
V1—O2—C1—O3	-173.7 (4)	C14—C15—C16—C11	-0.2 (8)
V1—O2—C1—C2	8.3 (5)	C12—C11—C16—O4	177.5 (4)
C10—N1—C2—C1	-155.4 (4)	C10—C11—C16—O4	-5.2 (7)
V1—N1—C2—C1	27.2 (4)	C12—C11—C16—C15	-1.1 (7)
C10—N1—C2—C3	87.4 (5)	C10—C11—C16—C15	176.2 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O5—H19...O3 <sup>i</sup>	1.00 (5)	1.73 (5)	2.687 (4)	161 (4)
C13—H13...O3 <sup>ii</sup>	0.93	2.59	3.500 (7)	165

Symmetry codes: (i)  $-x+2, -y+2, z$ ; (ii)  $-x+3/2, y-1/2, -z$ .