# (4-Methylbenzohydrazidato-$\kappa^2 N'$,O)-[2-(4-methylbenzoylhydrazinylidene-$\kappa^2 N$,O)-3-phenylpropionato(2−)]oxido-vanadium(V) methanol monosolvate

## Hon Wee Wong, Kong Mun Lo and Seik Weng Ng*

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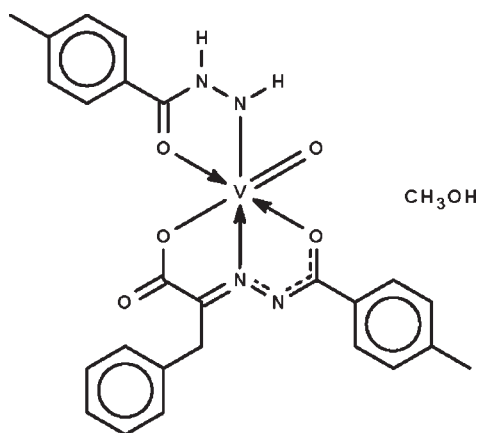Correspondence e-mail: seikweng@um.edu.my

The $V^V$ atom in the title compound, [V($C_8H_9N_2O$)($C_{17}H_{14}N_2O_3$)O]·$CH_3OH$, is *N,O*-chelated by the benzoylhydrazidate anion and *O,N,O'*-chelated by the (benzoylhydrazono)-propionate dianion. The octahedral *trans*-$N_2O_4$ coordination geometry is completed by the vanadyl O atom. Two mononuclear complexes and two solvent molecules are linked by O—H···O and O—H···N hydrogen bonds, generating a centrosymmetric aggregate.

## Related literature

For (benzohydrazidato)[2-(benzoylhydrazono)propionato]-oxidovanadium(V), see: Wong *et al.* (2009*a*) and for (4-chlorobenzohydrazidato)[2-(4-chlorobenzoylhydrazono)-propionato(2−)]oxidovanadium(V), see: Wong *et al.* (2009,*b*).



## Experimental

### Crystal data

[V($C_8H_9N_2O$)($C_{17}H_{14}N_2O_3$)O]·$CH_4O$
$M_r$ = 542.46
Triclinic, $P\bar{1}$
$a$ = 9.2770 (2) Å
$b$ = 11.2558 (2) Å
$c$ = 13.4691 (3) Å
$\alpha$ = 95.769 (2)°
$\beta$ = 96.708 (2)°
$\gamma$ = 109.675 (2)°
$V$ = 1300.48 (5) Å³
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.43 mm$^{-1}$
$T$ = 293 K
0.35 × 0.20 × 0.20 mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.864, $T_{max}$ = 0.919
12503 measured reflections
5956 independent reflections
3843 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.028

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.167
$S$ = 1.03
5956 reflections
337 parameters
13 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.48 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O6—H6···O4$^i$ | 0.84 | 2.18 | 2.821 (5) | 133 |
| N1—H1···O3$^i$ | 0.86 | 2.13 | 2.808 (3) | 135 |
| N2—H2···O6 | 0.86 | 1.99 | 2.800 (4) | 156 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

## References

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Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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# supporting information

# (4-Methylbenzohydrazidato-*κ²N′,O*)[2-(4-methylbenzoylhydrazinylidene-*κ²N,O*)-3-phenylpropionato(2−)]oxidovanadium(V) methanol monosolvate

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### S1. Comment

The reaction of vanadyl(IV) sulfate and the Schiff base that is synthesized by condensing a substituted benzhydrazine and a substituted pyruvic acid leads a vanadium(V) derivative of the Schiff base. However, another mole of the Schiff base is cleaved and the resulting benzhydrazine monoanion also chelates to the metal atom (Wong *et al.*, 2009*a*, 2009*b*). A similar product is isolated in the present study on the reaction of the Schiff base, 2-[*p*-methylbenzoylhydrazono]-3-phenylpropionic acid so that the metal atom is chelated by two different ligands. The mononuclear mixed-ligand compound crystallizes as a monosolvate (Scheme I, Fig. 1). The vanadium(V) atom is *N,O*-chelated by the benzoyl-hydrazidate anion and *O,N,O′*-chelated by the (benzoylhydrazono)propionate dianion; the terdentate chelate binds in a meridional mode. The octahedral *trans*-$N_2O_4$ coordination geometry is completed by the vanadyl O atom. Two mononuclear complexes and two solvent molecules are linked by hydrogen bonds to generate a centrosymmetric aggregate.

### S2. Experimental

2-[*p*-Methylbenzoylhydrazono]-3-phenylpropionic acid prepared from the condensation reaction of *p*-methyl-benzhydrazide and 3-phenylpyruvic acid. The compound (0.85 g, 3 mmol) and vanadyl sulfate (1.25 g, 1.5 mmol) in 50 ml of 95% ethanol for 5 hours. Slow evaporation of the filtrate gave orange crystals. The presence of methanol in the crystal structure is attributed to the methanol present in the techical grade solvent.

### S3. Refinement

Carbon-, nitrogen- and oxygen-bound H-atoms were placed in calculated positions (C–H 0.95 to 0.96 Å, N–H 0.86 Å and O–H 0.84 Å) and were included in the refinement in the riding model approximation, with $U$(H) set to $1.2U$(C,N) or $1.5U$($C_{methyl}$,O).

The carbon-oxygen distance in the methanol molecule was tightly restrained to 1.500±0.005 Å; the anisotropic temperature factors of the two atoms were restrained to be nearly isotropic. Attempts to model this molecule as a molecule disordered over two positions did not lead to meaningful results.
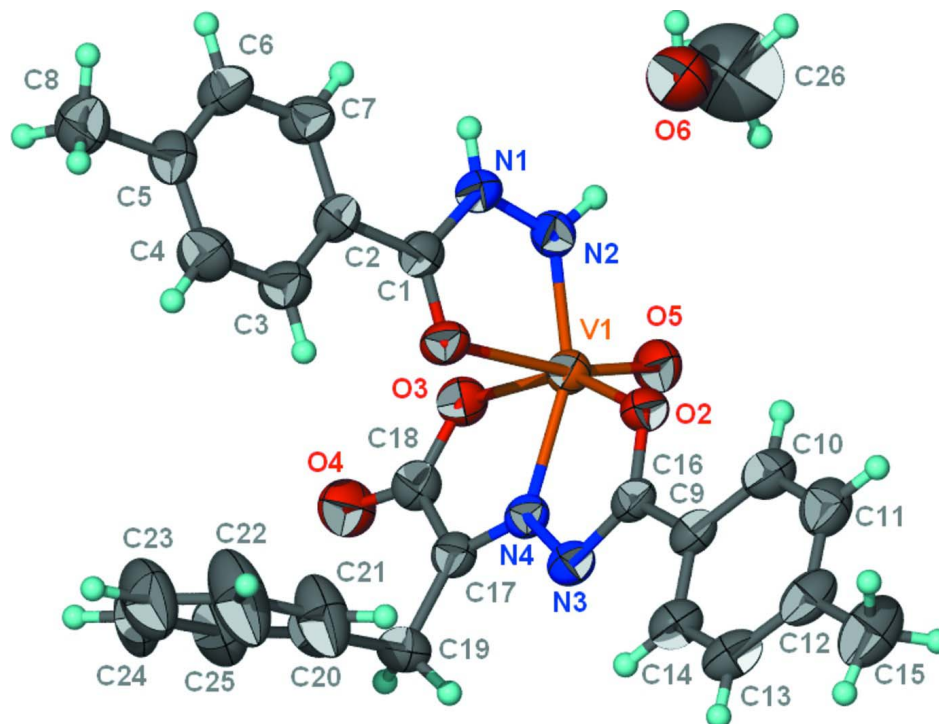
**Figure 1**

Anisotropic displacement ellipsoid plot (Barbour, 2001) of the title compound with ellipsoids at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

**(4-Methylbenzohydrazidato-κ²N′,O)[2-(4- methylbenzoylhydrazinylidene-κ²N,O)-3-phenylpropionato(2-)]oxidovanadium(V) methanol monosolvate**

*Crystal data*

| | |
|---|---|
| [V($C_8H_9N_2O$)($C_{17}H_{14}N_2O_3$)O]·$CH_4O$ | $Z = 2$ |
| $M_r = 542.46$ | $F(000) = 564$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.385$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.2770$ (2) Å | Cell parameters from 2688 reflections |
| $b = 11.2558$ (2) Å | $\theta = 2.5–22.6°$ |
| $c = 13.4691$ (3) Å | $\mu = 0.43$ mm$^{-1}$ |
| $\alpha = 95.769$ (2)° | $T = 293$ K |
| $\beta = 96.708$ (2)° | Block, orange |
| $\gamma = 109.675$ (2)° | $0.35 \times 0.20 \times 0.20$ mm |
| $V = 1300.48$ (5) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX diffractometer | 12503 measured reflections |
| Radiation source: fine-focus sealed tube | 5956 independent reflections |
| Graphite monochromator | 3843 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.028$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $\theta_{max} = 27.5°$, $\theta_{min} = 1.5°$ |
| $T_{min} = 0.864$, $T_{max} = 0.919$ | $h = -11 \rightarrow 12$ |
| | $k = -14 \rightarrow 14$ |
| | $l = -17 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.054$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.167$ | |
| $S = 1.03$ | H-atom parameters constrained |
| 5956 reflections | $w = 1/[\sigma^2(F_o^2) + (0.0856P)^2 + 0.2209P]$ |
| 337 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 13 restraints | $(\Delta/\sigma)_{max} = 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.55$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.48$ e Å$^{-3}$ |

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| V1 | 0.51795 (6) | 0.51791 (5) | 0.29034 (4) | 0.04766 (18) |
| O1 | 0.6537 (2) | 0.40109 (19) | 0.34261 (14) | 0.0526 (5) |
| O2 | 0.6733 (2) | 0.57531 (19) | 0.20087 (15) | 0.0542 (5) |
| O3 | 0.3375 (2) | 0.3810 (2) | 0.32493 (15) | 0.0557 (5) |
| O4 | 0.1695 (3) | 0.1820 (2) | 0.28159 (19) | 0.0734 (7) |
| O5 | 0.4366 (3) | 0.6198 (2) | 0.27738 (17) | 0.0633 (6) |
| O6 | 0.6859 (6) | 0.8379 (3) | 0.5277 (3) | 0.1379 (14) |
| H6 | 0.7610 | 0.8725 | 0.5749 | 0.207* |
| N1 | 0.7041 (3) | 0.5407 (2) | 0.47916 (18) | 0.0493 (6) |
| H1 | 0.7457 | 0.5723 | 0.5409 | 0.059* |
| N2 | 0.6198 (3) | 0.5937 (2) | 0.42177 (18) | 0.0498 (6) |
| H2 | 0.6126 | 0.6646 | 0.4464 | 0.060* |
| N3 | 0.5391 (3) | 0.3913 (2) | 0.08943 (17) | 0.0475 (6) |
| N4 | 0.4523 (3) | 0.3744 (2) | 0.16640 (17) | 0.0450 (5) |
| C1 | 0.7176 (3) | 0.4347 (3) | 0.4322 (2) | 0.0466 (7) |
| C2 | 0.8042 (3) | 0.3663 (3) | 0.4855 (2) | 0.0480 (7) |
| C3 | 0.8251 (4) | 0.2638 (3) | 0.4316 (3) | 0.0645 (9) |
| H3A | 0.7844 | 0.2400 | 0.3632 | 0.077* |
| C4 | 0.9055 (5) | 0.1970 (4) | 0.4783 (3) | 0.0740 (10) |
| H4 | 0.9209 | 0.1299 | 0.4402 | 0.089* |
| C5 | 0.9646 (4) | 0.2264 (3) | 0.5806 (3) | 0.0578 (8) |
| C6 | 0.9450 (4) | 0.3295 (3) | 0.6338 (3) | 0.0631 (9) |
| H6A | 0.9854 | 0.3528 | 0.7023 | 0.076* |
| C7 | 0.8662 (4) | 0.3989 (3) | 0.5873 (2) | 0.0597 (8) |
| H7 | 0.8547 | 0.4683 | 0.6248 | 0.072* |
| C8 | 1.0483 (4) | 0.1494 (4) | 0.6323 (3) | 0.0776 (11) |
| H8A | 1.1188 | 0.2018 | 0.6908 | 0.116* |
| H8B | 1.1051 | 0.1202 | 0.5863 | 0.116* |
| H8C | 0.9740 | 0.0772 | 0.6528 | 0.116* |
| C9 | 0.7612 (3) | 0.5454 (3) | 0.0442 (2) | 0.0487 (7) |
| C10 | 0.8704 (4) | 0.6676 (3) | 0.0614 (3) | 0.0633 (8) |
| H10 | 0.8768 | 0.7232 | 0.1191 | 0.076* |
| C11 | 0.9708 (4) | 0.7072 (4) | −0.0079 (3) | 0.0696 (10) |
| H11 | 1.0430 | 0.7899 | 0.0039 | 0.083* |

| | | | |
|---|---|---|---|
| C12 | 0.9656 (4) | 0.6271 (4) | −0.0932 (3) | 0.0643 (9) |
| C13 | 0.8574 (4) | 0.5052 (4) | −0.1092 (2) | 0.0631 (9) |
| H13 | 0.8527 | 0.4494 | −0.1663 | 0.076* |
| C14 | 0.7560 (4) | 0.4647 (3) | −0.0425 (2) | 0.0577 (8) |
| H14 | 0.6831 | 0.3823 | −0.0555 | 0.069* |
| C15 | 1.0760 (4) | 0.6718 (5) | −0.1666 (3) | 0.0884 (13) |
| H15A | 1.0625 | 0.6020 | −0.2183 | 0.133* |
| H15B | 1.1808 | 0.7028 | −0.1313 | 0.133* |
| H15C | 1.0553 | 0.7392 | −0.1969 | 0.133* |
| C16 | 0.6518 (3) | 0.5021 (3) | 0.1152 (2) | 0.0460 (6) |
| C17 | 0.3434 (3) | 0.2683 (3) | 0.1692 (2) | 0.0478 (7) |
| C18 | 0.2745 (4) | 0.2739 (3) | 0.2641 (2) | 0.0536 (7) |
| C19 | 0.2976 (4) | 0.1494 (3) | 0.0962 (2) | 0.0609 (8) |
| H19A | 0.3261 | 0.1701 | 0.0313 | 0.073* |
| H19B | 0.1860 | 0.1069 | 0.0868 | 0.073* |
| C20 | 0.3765 (4) | 0.0608 (3) | 0.1338 (3) | 0.0633 (9) |
| C21 | 0.5281 (5) | 0.0825 (4) | 0.1244 (4) | 0.1030 (16) |
| H21 | 0.5812 | 0.1509 | 0.0936 | 0.124* |
| C22 | 0.6049 (6) | 0.0033 (5) | 0.1603 (6) | 0.137 (2) |
| H22 | 0.7087 | 0.0204 | 0.1548 | 0.165* |
| C23 | 0.5271 (8) | −0.0973 (5) | 0.2027 (5) | 0.1208 (19) |
| H23 | 0.5769 | −0.1510 | 0.2255 | 0.145* |
| C24 | 0.3768 (8) | −0.1209 (4) | 0.2124 (4) | 0.1169 (19) |
| H24 | 0.3236 | −0.1910 | 0.2415 | 0.140* |
| C25 | 0.3016 (6) | −0.0412 (4) | 0.1791 (3) | 0.0907 (13) |
| H25 | 0.1990 | −0.0572 | 0.1876 | 0.109* |
| C26 | 0.5773 (8) | 0.9020 (8) | 0.5320 (7) | 0.192 (3) |
| H26A | 0.5100 | 0.8688 | 0.5794 | 0.287* |
| H26B | 0.5167 | 0.8886 | 0.4663 | 0.287* |
| H26C | 0.6320 | 0.9917 | 0.5531 | 0.287* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| V1 | 0.0597 (3) | 0.0463 (3) | 0.0420 (3) | 0.0246 (2) | 0.0100 (2) | 0.0063 (2) |
| O1 | 0.0668 (13) | 0.0564 (12) | 0.0399 (11) | 0.0298 (11) | 0.0082 (9) | 0.0026 (9) |
| O2 | 0.0625 (13) | 0.0522 (12) | 0.0424 (11) | 0.0141 (10) | 0.0074 (9) | 0.0052 (9) |
| O3 | 0.0648 (13) | 0.0579 (13) | 0.0488 (12) | 0.0243 (11) | 0.0197 (10) | 0.0067 (10) |
| O4 | 0.0693 (15) | 0.0715 (16) | 0.0716 (16) | 0.0101 (13) | 0.0239 (13) | 0.0126 (13) |
| O5 | 0.0803 (15) | 0.0571 (13) | 0.0625 (14) | 0.0378 (12) | 0.0081 (11) | 0.0098 (10) |
| O6 | 0.224 (4) | 0.093 (2) | 0.095 (2) | 0.063 (3) | 0.004 (3) | 0.0042 (19) |
| N1 | 0.0582 (15) | 0.0502 (14) | 0.0392 (12) | 0.0195 (12) | 0.0072 (11) | 0.0048 (11) |
| N2 | 0.0612 (15) | 0.0452 (13) | 0.0477 (13) | 0.0234 (12) | 0.0136 (11) | 0.0065 (10) |
| N3 | 0.0502 (13) | 0.0548 (15) | 0.0402 (12) | 0.0211 (12) | 0.0099 (10) | 0.0082 (11) |
| N4 | 0.0507 (13) | 0.0482 (14) | 0.0409 (12) | 0.0236 (12) | 0.0069 (10) | 0.0073 (10) |
| C1 | 0.0482 (16) | 0.0497 (16) | 0.0430 (16) | 0.0149 (13) | 0.0156 (13) | 0.0099 (13) |
| C2 | 0.0472 (16) | 0.0563 (17) | 0.0449 (16) | 0.0204 (14) | 0.0132 (12) | 0.0130 (13) |
| C3 | 0.087 (2) | 0.065 (2) | 0.0480 (18) | 0.0383 (19) | 0.0068 (17) | 0.0034 (15) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 0.097 (3) | 0.066 (2) | 0.068 (2) | 0.043 (2) | 0.010 (2) | 0.0006 (18) |
| C5 | 0.0551 (18) | 0.0573 (19) | 0.064 (2) | 0.0216 (15) | 0.0102 (15) | 0.0146 (15) |
| C6 | 0.069 (2) | 0.077 (2) | 0.0493 (18) | 0.0343 (19) | 0.0046 (15) | 0.0096 (16) |
| C7 | 0.067 (2) | 0.068 (2) | 0.0499 (18) | 0.0332 (17) | 0.0084 (15) | 0.0012 (15) |
| C8 | 0.078 (2) | 0.076 (3) | 0.086 (3) | 0.039 (2) | 0.001 (2) | 0.018 (2) |
| C9 | 0.0483 (16) | 0.0611 (19) | 0.0412 (15) | 0.0244 (15) | 0.0032 (12) | 0.0143 (13) |
| C10 | 0.064 (2) | 0.072 (2) | 0.0525 (19) | 0.0227 (18) | 0.0051 (15) | 0.0120 (16) |
| C11 | 0.0538 (19) | 0.075 (2) | 0.074 (2) | 0.0112 (18) | 0.0077 (17) | 0.0268 (19) |
| C12 | 0.0523 (18) | 0.097 (3) | 0.0536 (19) | 0.035 (2) | 0.0096 (15) | 0.0272 (19) |
| C13 | 0.0589 (19) | 0.093 (3) | 0.0459 (17) | 0.036 (2) | 0.0107 (15) | 0.0135 (17) |
| C14 | 0.0541 (18) | 0.073 (2) | 0.0489 (18) | 0.0246 (16) | 0.0087 (14) | 0.0125 (15) |
| C15 | 0.066 (2) | 0.131 (4) | 0.084 (3) | 0.039 (2) | 0.032 (2) | 0.047 (3) |
| C16 | 0.0522 (16) | 0.0533 (17) | 0.0379 (15) | 0.0251 (14) | 0.0050 (12) | 0.0105 (12) |
| C17 | 0.0507 (16) | 0.0483 (17) | 0.0465 (16) | 0.0205 (14) | 0.0051 (13) | 0.0083 (13) |
| C18 | 0.0531 (17) | 0.0575 (19) | 0.0533 (18) | 0.0218 (16) | 0.0118 (14) | 0.0098 (15) |
| C19 | 0.064 (2) | 0.058 (2) | 0.0531 (18) | 0.0173 (16) | 0.0009 (15) | −0.0016 (15) |
| C20 | 0.075 (2) | 0.0433 (17) | 0.066 (2) | 0.0184 (16) | 0.0049 (17) | −0.0017 (15) |
| C21 | 0.081 (3) | 0.064 (3) | 0.170 (5) | 0.029 (2) | 0.020 (3) | 0.032 (3) |
| C22 | 0.096 (4) | 0.073 (3) | 0.249 (8) | 0.037 (3) | 0.012 (4) | 0.040 (4) |
| C23 | 0.139 (5) | 0.073 (3) | 0.163 (6) | 0.055 (3) | 0.014 (4) | 0.024 (3) |
| C24 | 0.176 (6) | 0.066 (3) | 0.131 (4) | 0.054 (4) | 0.051 (4) | 0.042 (3) |
| C25 | 0.105 (3) | 0.064 (2) | 0.108 (3) | 0.028 (2) | 0.036 (3) | 0.018 (2) |
| C26 | 0.147 (5) | 0.208 (7) | 0.229 (7) | 0.077 (5) | 0.040 (5) | 0.015 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| V1—O5 | 1.583 (2) | C9—C10 | 1.384 (4) |
| V1—N2 | 1.875 (2) | C9—C14 | 1.391 (4) |
| V1—O2 | 1.970 (2) | C9—C16 | 1.473 (4) |
| V1—O3 | 1.996 (2) | C10—C11 | 1.395 (5) |
| V1—N4 | 2.080 (2) | C10—H10 | 0.9300 |
| V1—O1 | 2.215 (2) | C11—C12 | 1.373 (5) |
| O1—C1 | 1.241 (3) | C11—H11 | 0.9300 |
| O2—C16 | 1.301 (3) | C12—C13 | 1.378 (5) |
| O3—C18 | 1.297 (4) | C12—C15 | 1.507 (5) |
| O4—C18 | 1.224 (4) | C13—C14 | 1.375 (4) |
| O6—C26 | 1.426 (4) | C13—H13 | 0.9300 |
| O6—H6 | 0.8400 | C14—H14 | 0.9300 |
| N1—C1 | 1.344 (4) | C15—H15A | 0.9600 |
| N1—N2 | 1.353 (3) | C15—H15B | 0.9600 |
| N1—H1 | 0.8600 | C15—H15C | 0.9600 |
| N2—H2 | 0.8600 | C17—C19 | 1.481 (4) |
| N3—C16 | 1.312 (4) | C17—C18 | 1.500 (4) |
| N3—N4 | 1.375 (3) | C19—C20 | 1.513 (5) |
| N4—C17 | 1.286 (4) | C19—H19A | 0.9700 |
| C1—C2 | 1.465 (4) | C19—H19B | 0.9700 |
| C2—C3 | 1.383 (4) | C20—C21 | 1.368 (5) |
| C2—C7 | 1.384 (4) | C20—C25 | 1.370 (5) |

| C3—C4 | 1.372 (5) | C21—C22 | 1.403 (6) |
|---|---|---|---|
| C3—H3A | 0.9300 | C21—H21 | 0.9300 |
| C4—C5 | 1.385 (5) | C22—C23 | 1.344 (7) |
| C4—H4 | 0.9300 | C22—H22 | 0.9300 |
| C5—C6 | 1.377 (5) | C23—C24 | 1.354 (7) |
| C5—C8 | 1.513 (4) | C23—H23 | 0.9300 |
| C6—C7 | 1.384 (4) | C24—C25 | 1.387 (7) |
| C6—H6A | 0.9300 | C24—H24 | 0.9300 |
| C7—H7 | 0.9300 | C25—H25 | 0.9300 |
| C8—H8A | 0.9600 | C26—H26A | 0.9600 |
| C8—H8B | 0.9600 | C26—H26B | 0.9600 |
| C8—H8C | 0.9600 | C26—H26C | 0.9600 |
| | | | |
| O5—V1—N2 | 93.97 (11) | C9—C10—H10 | 120.1 |
| O5—V1—O2 | 98.37 (11) | C11—C10—H10 | 120.1 |
| N2—V1—O2 | 105.98 (10) | C12—C11—C10 | 121.5 (3) |
| O5—V1—O3 | 97.34 (11) | C12—C11—H11 | 119.2 |
| N2—V1—O3 | 98.78 (9) | C10—C11—H11 | 119.2 |
| O2—V1—O3 | 149.52 (9) | C11—C12—C13 | 118.2 (3) |
| O5—V1—N4 | 112.73 (10) | C11—C12—C15 | 120.5 (4) |
| N2—V1—N4 | 153.13 (10) | C13—C12—C15 | 121.3 (4) |
| O2—V1—N4 | 74.21 (9) | C14—C13—C12 | 121.3 (3) |
| O3—V1—N4 | 75.66 (9) | C14—C13—H13 | 119.4 |
| O5—V1—O1 | 167.08 (10) | C12—C13—H13 | 119.4 |
| N2—V1—O1 | 73.11 (9) | C13—C14—C9 | 120.8 (3) |
| O2—V1—O1 | 85.61 (8) | C13—C14—H14 | 119.6 |
| O3—V1—O1 | 84.85 (8) | C9—C14—H14 | 119.6 |
| N4—V1—O1 | 80.17 (8) | C12—C15—H15A | 109.5 |
| C1—O1—V1 | 113.77 (18) | C12—C15—H15B | 109.5 |
| C16—O2—V1 | 116.79 (18) | H15A—C15—H15B | 109.5 |
| C18—O3—V1 | 119.46 (18) | C12—C15—H15C | 109.5 |
| C26—O6—H6 | 109.5 | H15A—C15—H15C | 109.5 |
| C1—N1—N2 | 114.8 (2) | H15B—C15—H15C | 109.5 |
| C1—N1—H1 | 122.6 | O2—C16—N3 | 123.4 (3) |
| N2—N1—H1 | 122.6 | O2—C16—C9 | 118.2 (3) |
| N1—N2—V1 | 122.31 (18) | N3—C16—C9 | 118.3 (2) |
| N1—N2—H2 | 118.8 | N4—C17—C19 | 126.7 (3) |
| V1—N2—H2 | 118.8 | N4—C17—C18 | 110.9 (3) |
| C16—N3—N4 | 107.1 (2) | C19—C17—C18 | 122.1 (3) |
| C17—N4—N3 | 122.2 (2) | O4—C18—O3 | 124.2 (3) |
| C17—N4—V1 | 119.05 (19) | O4—C18—C17 | 121.2 (3) |
| N3—N4—V1 | 118.32 (17) | O3—C18—C17 | 114.6 (3) |
| O1—C1—N1 | 115.7 (3) | C17—C19—C20 | 110.7 (3) |
| O1—C1—C2 | 123.6 (3) | C17—C19—H19A | 109.5 |
| N1—C1—C2 | 120.7 (3) | C20—C19—H19A | 109.5 |
| C3—C2—C7 | 118.2 (3) | C17—C19—H19B | 109.5 |
| C3—C2—C1 | 118.4 (3) | C20—C19—H19B | 109.5 |
| C7—C2—C1 | 123.4 (3) | H19A—C19—H19B | 108.1 |

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| C4—C3—C2 | 120.5 (3) | C21—C20—C25 | 117.8 (4) |
| C4—C3—H3A | 119.8 | C21—C20—C19 | 120.1 (3) |
| C2—C3—H3A | 119.8 | C25—C20—C19 | 122.2 (4) |
| C3—C4—C5 | 121.9 (3) | C20—C21—C22 | 121.3 (4) |
| C3—C4—H4 | 119.0 | C20—C21—H21 | 119.4 |
| C5—C4—H4 | 119.0 | C22—C21—H21 | 119.4 |
| C6—C5—C4 | 117.4 (3) | C23—C22—C21 | 119.4 (5) |
| C6—C5—C8 | 121.0 (3) | C23—C22—H22 | 120.3 |
| C4—C5—C8 | 121.6 (3) | C21—C22—H22 | 120.3 |
| C5—C6—C7 | 121.3 (3) | C22—C23—C24 | 120.4 (5) |
| C5—C6—H6A | 119.4 | C22—C23—H23 | 119.8 |
| C7—C6—H6A | 119.4 | C24—C23—H23 | 119.8 |
| C2—C7—C6 | 120.7 (3) | C23—C24—C25 | 120.3 (5) |
| C2—C7—H7 | 119.6 | C23—C24—H24 | 119.8 |
| C6—C7—H7 | 119.6 | C25—C24—H24 | 119.8 |
| C5—C8—H8A | 109.5 | C20—C25—C24 | 120.8 (4) |
| C5—C8—H8B | 109.5 | C20—C25—H25 | 119.6 |
| H8A—C8—H8B | 109.5 | C24—C25—H25 | 119.6 |
| C5—C8—H8C | 109.5 | O6—C26—H26A | 109.5 |
| H8A—C8—H8C | 109.5 | O6—C26—H26B | 109.5 |
| H8B—C8—H8C | 109.5 | H26A—C26—H26B | 109.5 |
| C10—C9—C14 | 118.4 (3) | O6—C26—H26C | 109.5 |
| C10—C9—C16 | 120.9 (3) | H26A—C26—H26C | 109.5 |
| C14—C9—C16 | 120.7 (3) | H26B—C26—H26C | 109.5 |
| C9—C10—C11 | 119.9 (3) | | |
| | | | |
| O5—V1—O1—C1 | 5.1 (5) | C4—C5—C6—C7 | 1.5 (5) |
| N2—V1—O1—C1 | 5.47 (19) | C8—C5—C6—C7 | −178.9 (3) |
| O2—V1—O1—C1 | 113.6 (2) | C3—C2—C7—C6 | −0.9 (5) |
| O3—V1—O1—C1 | −95.3 (2) | C1—C2—C7—C6 | 179.1 (3) |
| N4—V1—O1—C1 | −171.6 (2) | C5—C6—C7—C2 | 0.2 (5) |
| O5—V1—O2—C16 | −108.5 (2) | C14—C9—C10—C11 | 0.5 (5) |
| N2—V1—O2—C16 | 154.9 (2) | C16—C9—C10—C11 | −178.8 (3) |
| O3—V1—O2—C16 | 11.8 (3) | C9—C10—C11—C12 | −0.7 (5) |
| N4—V1—O2—C16 | 2.85 (19) | C10—C11—C12—C13 | 0.2 (5) |
| O1—V1—O2—C16 | 83.9 (2) | C10—C11—C12—C15 | −179.6 (3) |
| O5—V1—O3—C18 | 115.4 (2) | C11—C12—C13—C14 | 0.5 (5) |
| N2—V1—O3—C18 | −149.4 (2) | C15—C12—C13—C14 | −179.6 (3) |
| O2—V1—O3—C18 | −5.1 (3) | C12—C13—C14—C9 | −0.8 (5) |
| N4—V1—O3—C18 | 3.8 (2) | C10—C9—C14—C13 | 0.3 (5) |
| O1—V1—O3—C18 | −77.4 (2) | C16—C9—C14—C13 | 179.5 (3) |
| C1—N1—N2—V1 | 4.3 (3) | V1—O2—C16—N3 | −4.0 (4) |
| O5—V1—N2—N1 | 174.8 (2) | V1—O2—C16—C9 | 176.91 (18) |
| O2—V1—N2—N1 | −85.3 (2) | N4—N3—C16—O2 | 2.2 (4) |
| O3—V1—N2—N1 | 76.8 (2) | N4—N3—C16—C9 | −178.7 (2) |
| N4—V1—N2—N1 | 1.2 (4) | C10—C9—C16—O2 | −8.9 (4) |
| O1—V1—N2—N1 | −5.07 (19) | C14—C9—C16—O2 | 171.9 (3) |
| C16—N3—N4—C17 | −171.9 (2) | C10—C9—C16—N3 | 172.0 (3) |

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| C16—N3—N4—V1 | 0.5 (3) | C14—C9—C16—N3 | −7.3 (4) |
| O5—V1—N4—C17 | −96.6 (2) | N3—N4—C17—C19 | 2.4 (4) |
| N2—V1—N4—C17 | 76.5 (3) | V1—N4—C17—C19 | −169.9 (2) |
| O2—V1—N4—C17 | 170.8 (2) | N3—N4—C17—C18 | 176.7 (2) |
| O3—V1—N4—C17 | −4.5 (2) | V1—N4—C17—C18 | 4.3 (3) |
| O1—V1—N4—C17 | 82.6 (2) | V1—O3—C18—O4 | 175.6 (2) |
| O5—V1—N4—N3 | 90.8 (2) | V1—O3—C18—C17 | −2.7 (3) |
| N2—V1—N4—N3 | −96.1 (3) | N4—C17—C18—O4 | −179.5 (3) |
| O2—V1—N4—N3 | −1.82 (17) | C19—C17—C18—O4 | −4.9 (5) |
| O3—V1—N4—N3 | −177.2 (2) | N4—C17—C18—O3 | −1.1 (4) |
| O1—V1—N4—N3 | −90.01 (18) | C19—C17—C18—O3 | 173.5 (3) |
| V1—O1—C1—N1 | −5.0 (3) | N4—C17—C19—C20 | 95.4 (4) |
| V1—O1—C1—C2 | 175.4 (2) | C18—C17—C19—C20 | −78.3 (4) |
| N2—N1—C1—O1 | 1.1 (4) | C17—C19—C20—C21 | −78.5 (4) |
| N2—N1—C1—C2 | −179.3 (2) | C17—C19—C20—C25 | 100.5 (4) |
| O1—C1—C2—C3 | 4.6 (4) | C25—C20—C21—C22 | −0.3 (7) |
| N1—C1—C2—C3 | −175.0 (3) | C19—C20—C21—C22 | 178.8 (5) |
| O1—C1—C2—C7 | −175.4 (3) | C20—C21—C22—C23 | 1.5 (9) |
| N1—C1—C2—C7 | 5.0 (4) | C21—C22—C23—C24 | −1.2 (10) |
| C7—C2—C3—C4 | −0.1 (5) | C22—C23—C24—C25 | −0.3 (10) |
| C1—C2—C3—C4 | 179.9 (3) | C21—C20—C25—C24 | −1.2 (7) |
| C2—C3—C4—C5 | 1.9 (6) | C19—C20—C25—C24 | 179.8 (4) |
| C3—C4—C5—C6 | −2.5 (6) | C23—C24—C25—C20 | 1.5 (8) |
| C3—C4—C5—C8 | 177.8 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| O6—H6···O4[i] | 0.84 | 2.18 | 2.821 (5) | 133 |
| N1—H1···O3[i] | 0.86 | 2.13 | 2.808 (3) | 135 |
| N2—H2···O6 | 0.86 | 1.99 | 2.800 (4) | 156 |

Symmetry code: (i) −x+1, −y+1, −z+1.