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# Di-$\mu$-oxido-bis({2-[(*R*,*R*)-(−)-(2-aminocyclohexyl)iminomethyl]-4-nitrophenolato-$\kappa^3N,N',O$}oxidovanadium(V)) dimethyl sulfoxide disolvate

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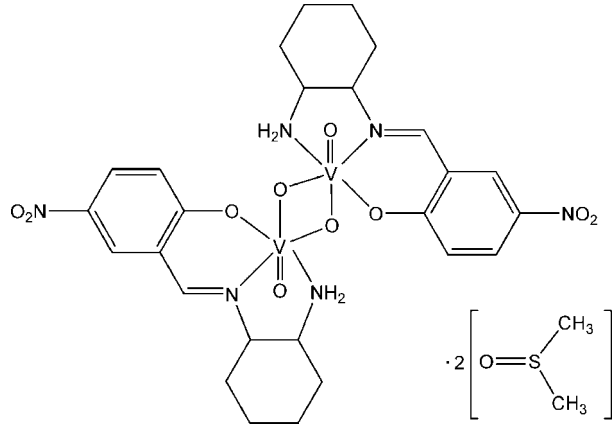
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.009 Å; disorder in main residue; $R$ factor = 0.079; $wR$ factor = 0.194; data-to-parameter ratio = 13.1.

The title compound, $[V_2(C_{13}H_{16}N_3O_3)_2O_4]\cdot 2C_2H_6OS$, is a centrosymmetric dimeric complex solvated by two dimethyl sulfoxide molecules. Each $V^V$ atom is six-coordinated by one oxide group, two N atoms and one O atom from the tridentate Schiff base ligand, and by two additional bridging O atoms in a distorted octahedral coordination geometry. Three atoms of the cyclohexane ring are each disordered over two sites, with occupancy factors of 0.501 (10) and 0.499 (10). C—H···O and N—H···O hydrogen bonds link the dimers and solvent molecules into a supramolecular network.

## Related literature

For general background, see: Carter-Franklin *et al.* (2003); Eady (2003); Evangelou (2002); Mendz (1991); Parekh *et al.* (2006); Rao *et al.* (1981); Rehder *et al.* (2002, 2003); Shahzadi *et al.* (2007). For related structures, see: Kwiatkowski *et al.* (2007); Mokry & Carrano (1993); Romanowski *et al.* (2008); Root *et al.* (1993). For the synthesis, see: Kwiatkowski *et al.* (2003).



## Experimental

### Crystal data

$[V_2(C_{13}H_{16}N_3O_3)_2O_4]\cdot 2C_2H_6OS$
$M_r$ = 846.71
Triclinic, $P\overline{1}$
$a$ = 7.249 (1) Å
$b$ = 11.747 (2) Å
$c$ = 11.809 (2) Å
$\alpha$ = 77.69 (3)°
$\beta$ = 88.62 (3)°
$\gamma$ = 76.13 (3)°
$V$ = 953.4 (3) Å$^3$
$Z$ = 1
Mo $K\alpha$ radiation
$\mu$ = 0.67 mm$^{-1}$
$T$ = 295 (2) K
0.60 × 0.25 × 0.10 mm

### Data collection

Oxford Diffraction Ruby CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min}$ = 0.720, $T_{max}$ = 0.936
6786 measured reflections
3355 independent reflections
3245 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.194
$S$ = 1.44
3355 reflections
256 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.44 e Å$^{-3}$

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| N11—V19 | 2.186 (5) | V19—O21$^{i}$ | 1.663 (4) |
| N18—V19 | 2.109 (5) | V19—O22 | 1.929 (4) |
| V19—O20 | 1.610 (4) | V19—O21 | 2.372 (4) |

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N18—H18$A$···O22$^{i}$ | 0.90 | 2.29 | 3.069 (6) | 145 |
| N18—H18$B$···O26$^{ii}$ | 0.90 | 2.16 | 2.913 (8) | 140 |
| C5—H5$A$···O26$^{iii}$ | 0.93 | 2.57 | 3.494 (9) | 171 |
| C10—H10$A$···O21$^{iv}$ | 0.93 | 2.49 | 3.167 (7) | 130 |
| C13—H13$B$···O21$^{iv}$ | 0.97 | 2.55 | 3.392 (7) | 145 |
| C14—H14$A$···O26$^{v}$ | 0.97 | 2.38 | 3.211 (11) | 144 |
| C15—H15$B$···O8$^{vi}$ | 0.97 | 2.32 | 3.109 (17) | 138 |

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2164).

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# supporting information

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# Di-$\mu$-oxido-bis({2-[(*R*,*R*)-(−)-(2-aminocyclohexyl)iminomethyl]-4-nitrophenolato-$\kappa^3N,N',O$}oxidovanadium(V)) dimethyl sulfoxide disolvate

**Grzegorz Romanowski, Michał Wera and Artur Sikorski**

## S1. Comment

Research in vanadium chemistry and biochemistry increased after discovery of insulin mimetic (Rehder *et al.*, 2002), anticancer (Evangelou, 2002), antifungal and antibacterial (Parekh *et al.*, 2006; Shahzadi *et al.*, 2007) activity of this element. Oxidovanadium(IV) and (V) compounds exert catalytic activity like some biological enzymes, *viz*. haloperoxidases (Carter-Franklin *et al.*, 2003; Rehder *et al.*, 2003), phosphomutases (Mendz, 1991) and nitrogenases (Eady, 2003). Moreover, investigation of vanadium(V) complexes with Schiff bases is prompted by the fact that these ligands are coordinated to the metal through O and N atoms, similar to the coordination environments of natural systems.

The title compound was earlier characterized by spectroscopic methods (IR, UV-Vis, $^1$H and $^{51}$V NMR) (Kwiatkowski *et al.*, 2007). The crystal structure consists of a centrosymmetric dimeric vanadium(V) complex and two dimethyl sulfoxide (DMSO) molecules (Fig. 1). The singly deprotonated Schiff base acts as a tridentate ligand, forming one five- and one six-membered chelate rings. Each $V^V$ atom is six-coordinated in a distorted octahedral environment. Two axial positions are occupied by one phenolate O atom (O22) and one amine N atom (N18), and the equatorial positions are occupied by one azomethine N atom (N11), two strongly [$O20^i$ and $O21^i$; symmetry code: (i) -*x*, -*y* + 1, -*z* + 2] and one weakly (O21) bonded oxide groups (Fig. 1). The V19—O20, V19—$O21^i$(bridging) and V19—O22(phenolate) bond distances (Table 1) agree well with the corresponding values reported for related compounds (Mokry & Carrano, 1993; Romanowski *et al.*, 2008; Root *et al.*, 1993). The V19—O21 bond is longer than O21—$V19^i$ bond due to the involvement of this O atom in bridging between the V atoms. The V19, O21, $V19^i$, $O21^i$ atoms are situated in the vertices of a parallelogram with the acute O21—V19—$O21^i$ angle of 77.9 (2)°. The V···V separation is 3.170 (1)Å and falls within the range of known V···V distances in double-bridged vanadium polynuclear systems (Mokry & Carrano, 1993; Romanowski *et al.*, 2008; Root *et al.*, 1993). Three C atoms of the cyclohexane ring exhibit twofold disorder. The C12, C15 and C17 atoms are each disordered over two sites, with occupancy factors of 0.501 (10) and 0.499 (10). The five-membered chelate ring defined by V19, N11, C12, C17, N18 adopts a twisted conformation on C12 and C17 atoms, with $P$ = 250.3 (5)° and $\tau_{(M)}$ = 56.7 (6)° for reference bond V19—N11 (Rao *et al.*, 1981) (Fig. 1).

In the crystal structure, C—H···O and N—H···O hydrogen bonds link the dimers and solvent molecules into a supramolecular network (Table 2; Fig. 2).

## S2. Experimental

The title compound was obtained in a template/complexation reaction analogous to that described for preparation of dioxidovanadium(V) complexes with Schiff base ligands (Kwiatkowski *et al.*, 2003). A solution of *R,R*-(–)-1,2-diaminocyclohexane (1 mmol) in absolute ethanol (10 ml) was added under stirring to a freshly filtered solution of vanadium(V) oxytriethoxide (1 mmol) in absolute ethanol (50 ml), producing a yellow suspension of the intermediate. 5-Nitrosalicylaldehyde (1 mmol) dissolved in absolute ethanol was added to the aforementioned suspension. After refluxing of the

resulting mixture (70 ml) for 2 h and its cooling to room temperature, the separated solids were filtered off, washed several times with ethanol, recrystallized from DMSO–EtOH mixture and dried over molecular sieves.

### S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 and N—H = 0.90 Å, and with $U_{iso}$(H) = 1.2(1.5 for methyl group)$U_{eq}$(C,*N*).

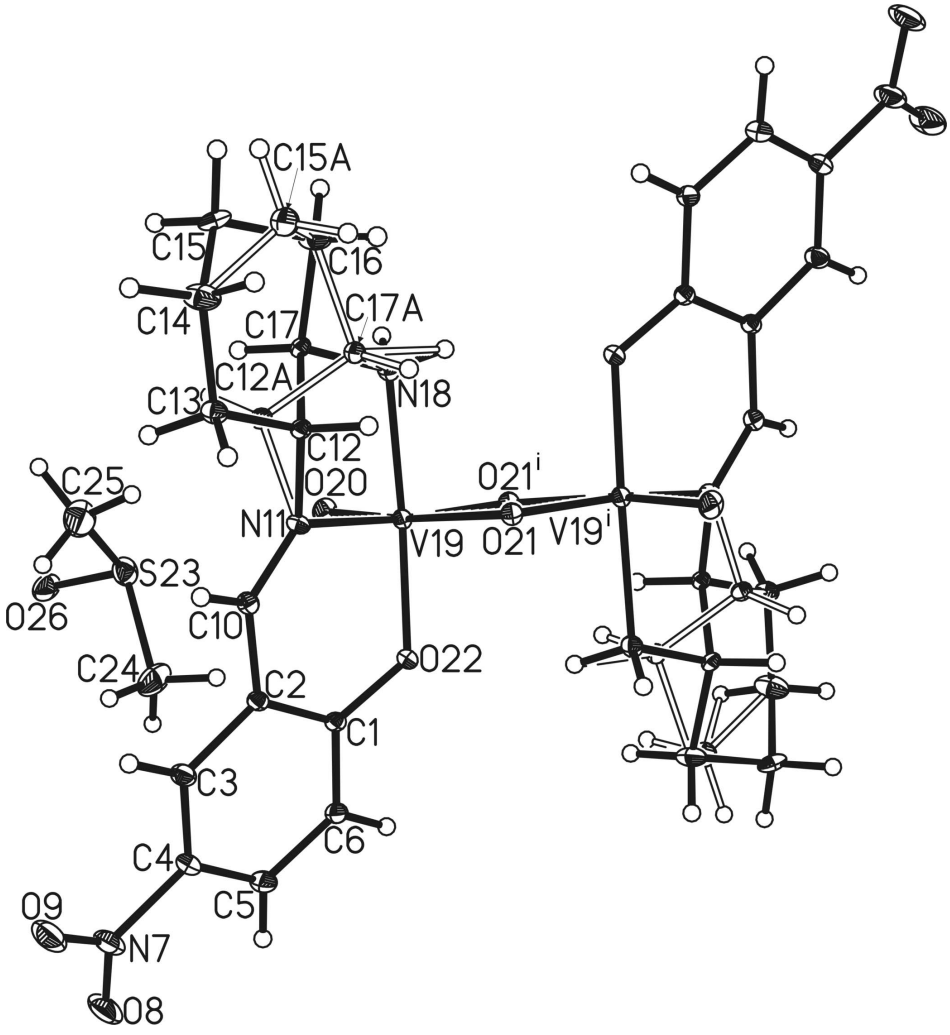


**Figure 1**

The molecular structure of the title compound, with displacement ellipsoids drawn at the 25% probability level. Disordered parts are shown by open bonds. [Symmetry code: (i) -*x*, -*y* + 1, -*z* + 2.]

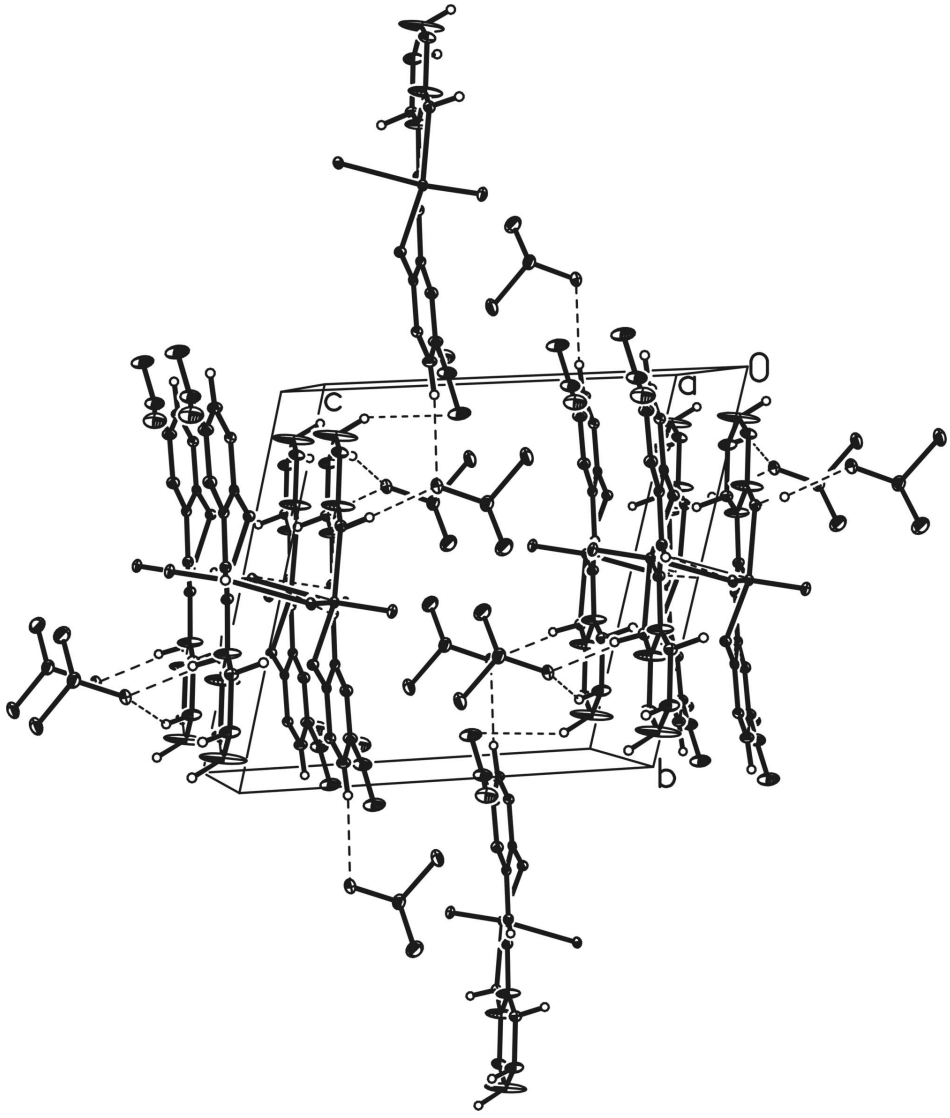


**Figure 2**

The arrangement of molecules in the title compound, viewed approximately along the *a* axis. Hydrogen bonds are represented by dashed lines.

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*Crystal data*

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Triclinic, $P\overline{1}$
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$\alpha$ = 77.69 (3)°
$\beta$ = 88.62 (3)°
$\gamma$ = 76.13 (3)°
$V$ = 953.4 (3) Å$^3$
$Z$ = 1
$F$(000) = 440
$D_x$ = 1.475 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3355 reflections
$\theta$ = 3.0–25.1°
$\mu$ = 0.67 mm$^{-1}$

$T$ = 295 K
Needle, yellow

0.60 × 0.25 × 0.10 mm

*Data collection*

Oxford Diffraction Ruby CCD diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.4002 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min}$ = 0.720, $T_{max}$ = 0.936

6786 measured reflections
3355 independent reflections
3245 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.021
$\theta_{max}$ = 25.1°, $\theta_{min}$ = 3.0°
$h$ = −8→8
$k$ = −13→11
$l$ = −13→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.194
$S$ = 1.44
3355 reflections
256 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
$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2 + 4.2049P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.44 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0061 (16)

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.1768 (8) | 0.7547 (5) | 0.8238 (5) | 0.0318 (12) | |
| C2 | 0.3625 (8) | 0.7030 (5) | 0.7934 (5) | 0.0322 (12) | |
| C3 | 0.4761 (10) | 0.7777 (6) | 0.7378 (6) | 0.0448 (15) | |
| H3A | 0.5999 | 0.7450 | 0.7188 | 0.054* | |
| C4 | 0.4032 (11) | 0.8999 (6) | 0.7112 (6) | 0.0505 (17) | |
| C5 | 0.2207 (11) | 0.9522 (6) | 0.7375 (6) | 0.0525 (18) | |
| H5A | 0.1727 | 1.0348 | 0.7155 | 0.063* | |
| C6 | 0.1108 (10) | 0.8802 (5) | 0.7969 (5) | 0.0403 (14) | |
| H6A | −0.0095 | 0.9151 | 0.8197 | 0.048* | |
| N7 | 0.5242 (12) | 0.9771 (6) | 0.6526 (7) | 0.077 (2) | |
| O8 | 0.4474 (11) | 1.0817 (5) | 0.6099 (7) | 0.103 (3) | |
| O9 | 0.6933 (10) | 0.9331 (6) | 0.6482 (8) | 0.101 (3) | |
| C10 | 0.4411 (8) | 0.5748 (5) | 0.8148 (5) | 0.0335 (13) | |
| H10A | 0.5713 | 0.5473 | 0.8071 | 0.040* | |
| N11 | 0.3421 (7) | 0.4965 (4) | 0.8437 (4) | 0.0329 (11) | |
| C12 | 0.436 (2) | 0.3677 (13) | 0.8877 (13) | 0.030 (3) | 0.501 (10) |
| H12A | 0.4263 | 0.3496 | 0.9722 | 0.036* | 0.501 (10) |
| C12A | 0.428 (2) | 0.3682 (13) | 0.8308 (15) | 0.032 (3) | 0.499 (10) |
| H12B | 0.4040 | 0.3614 | 0.7515 | 0.038* | 0.499 (10) |
| C13 | 0.6421 (8) | 0.3255 (5) | 0.8594 (6) | 0.0405 (14) | |
| H13A | 0.6607 | 0.3629 | 0.7798 | 0.049* | |

| | | | | | |
|---|---|---|---|---|---|
| H13B | 0.7202 | 0.3522 | 0.9090 | 0.049* | |
| C14 | 0.7089 (11) | 0.1916 (6) | 0.8742 (9) | 0.070 (2) | |
| H14A | 0.8251 | 0.1749 | 0.8318 | 0.083* | |
| H14B | 0.7415 | 0.1579 | 0.9557 | 0.083* | |
| C15 | 0.5840 (19) | 0.1297 (11) | 0.8390 (14) | 0.048 (4) | 0.501 (10) |
| H15A | 0.6353 | 0.0441 | 0.8656 | 0.058* | 0.501 (10) |
| H15B | 0.5754 | 0.1470 | 0.7550 | 0.058* | 0.501 (10) |
| C15A | 0.595 (2) | 0.1146 (13) | 0.9356 (14) | 0.054 (4)* | 0.499 (10) |
| H15C | 0.6012 | 0.1126 | 1.0180 | 0.064* | 0.499 (10) |
| H15D | 0.6433 | 0.0335 | 0.9240 | 0.064* | 0.499 (10) |
| C16 | 0.3804 (10) | 0.1654 (6) | 0.8875 (8) | 0.057 (2) | |
| H16A | 0.3852 | 0.1485 | 0.9715 | 0.069* | |
| H16B | 0.2972 | 0.1216 | 0.8621 | 0.069* | |
| C17 | 0.3085 (16) | 0.3038 (10) | 0.8368 (11) | 0.032 (3) | 0.501 (10) |
| H17A | 0.3097 | 0.3219 | 0.7519 | 0.038* | 0.501 (10) |
| C17A | 0.3189 (16) | 0.2942 (10) | 0.9138 (13) | 0.033 (3)* | 0.499 (10) |
| H17B | 0.3453 | 0.2941 | 0.9948 | 0.040* | 0.499 (10) |
| N18 | 0.1126 (7) | 0.3474 (4) | 0.8793 (4) | 0.0361 (11) | |
| H18A | 0.1072 | 0.3108 | 0.9541 | 0.043* | |
| H18B | 0.0280 | 0.3264 | 0.8382 | 0.043* | |
| V19 | 0.03456 (13) | 0.53484 (9) | 0.86534 (8) | 0.0289 (3) | |
| O20 | −0.0303 (7) | 0.5634 (4) | 0.7308 (4) | 0.0490 (12) | |
| O21 | 0.1601 (5) | 0.4698 (4) | 1.0582 (3) | 0.0325 (9) | |
| O22 | 0.0653 (6) | 0.6894 (3) | 0.8812 (3) | 0.0364 (9) | |
| S23 | −0.0005 (3) | 0.6916 (2) | 0.44455 (18) | 0.0657 (6) | |
| C24 | 0.0490 (17) | 0.8021 (9) | 0.5086 (8) | 0.094 (3) | |
| H24A | −0.0272 | 0.8791 | 0.4711 | 0.140* | |
| H24B | 0.1811 | 0.8019 | 0.5006 | 0.140* | |
| H24C | 0.0202 | 0.7860 | 0.5894 | 0.140* | |
| C25 | 0.2180 (17) | 0.5849 (10) | 0.4774 (10) | 0.104 (4) | |
| H25A | 0.2351 | 0.5309 | 0.4253 | 0.156* | |
| H25B | 0.2182 | 0.5404 | 0.5557 | 0.156* | |
| H25C | 0.3197 | 0.6252 | 0.4693 | 0.156* | |
| O26 | −0.0092 (10) | 0.7404 (5) | 0.3172 (5) | 0.0747 (17) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.038 (3) | 0.034 (3) | 0.028 (3) | −0.015 (2) | −0.004 (2) | −0.008 (2) |
| C2 | 0.040 (3) | 0.033 (3) | 0.027 (3) | −0.016 (2) | −0.003 (2) | −0.006 (2) |
| C3 | 0.045 (4) | 0.040 (4) | 0.053 (4) | −0.019 (3) | 0.003 (3) | −0.009 (3) |
| C4 | 0.065 (5) | 0.036 (3) | 0.055 (4) | −0.024 (3) | 0.014 (3) | −0.006 (3) |
| C5 | 0.066 (5) | 0.033 (3) | 0.057 (4) | −0.013 (3) | 0.004 (4) | −0.006 (3) |
| C6 | 0.051 (4) | 0.033 (3) | 0.038 (3) | −0.011 (3) | 0.002 (3) | −0.008 (3) |
| N7 | 0.081 (5) | 0.046 (4) | 0.109 (6) | −0.033 (4) | 0.030 (5) | −0.011 (4) |
| O8 | 0.115 (6) | 0.048 (4) | 0.140 (7) | −0.034 (4) | 0.046 (5) | 0.007 (4) |
| O9 | 0.081 (5) | 0.072 (4) | 0.156 (7) | −0.041 (4) | 0.047 (5) | −0.016 (4) |
| C10 | 0.029 (3) | 0.038 (3) | 0.034 (3) | −0.012 (2) | −0.002 (2) | −0.007 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N11 | 0.031 (2) | 0.028 (2) | 0.040 (3) | −0.011 (2) | −0.003 (2) | −0.003 (2) |
| C12 | 0.035 (7) | 0.030 (7) | 0.026 (8) | −0.010 (5) | −0.002 (7) | −0.008 (7) |
| C12A | 0.031 (7) | 0.033 (7) | 0.034 (9) | −0.008 (5) | −0.003 (7) | −0.011 (7) |
| C13 | 0.034 (3) | 0.038 (3) | 0.050 (4) | −0.011 (3) | −0.003 (3) | −0.008 (3) |
| C14 | 0.040 (4) | 0.040 (4) | 0.121 (8) | −0.002 (3) | 0.001 (4) | −0.008 (4) |
| C15 | 0.053 (8) | 0.028 (6) | 0.064 (10) | 0.003 (6) | −0.013 (7) | −0.023 (6) |
| C16 | 0.050 (4) | 0.036 (4) | 0.093 (6) | −0.017 (3) | 0.004 (4) | −0.021 (4) |
| C17 | 0.039 (6) | 0.032 (6) | 0.027 (7) | −0.010 (5) | 0.001 (5) | −0.013 (5) |
| N18 | 0.032 (3) | 0.042 (3) | 0.039 (3) | −0.015 (2) | 0.001 (2) | −0.009 (2) |
| V19 | 0.0284 (5) | 0.0349 (6) | 0.0256 (5) | −0.0116 (4) | −0.0012 (4) | −0.0062 (4) |
| O20 | 0.061 (3) | 0.053 (3) | 0.028 (2) | −0.007 (2) | −0.009 (2) | −0.005 (2) |
| O21 | 0.029 (2) | 0.041 (2) | 0.031 (2) | −0.0135 (17) | −0.0036 (16) | −0.0099 (17) |
| O22 | 0.042 (2) | 0.033 (2) | 0.037 (2) | −0.0149 (18) | 0.0058 (18) | −0.0062 (17) |
| S23 | 0.0761 (14) | 0.0722 (14) | 0.0477 (11) | −0.0224 (11) | 0.0021 (10) | −0.0052 (10) |
| C24 | 0.125 (9) | 0.093 (7) | 0.062 (6) | −0.005 (6) | −0.013 (6) | −0.038 (5) |
| C25 | 0.121 (10) | 0.086 (7) | 0.080 (7) | 0.006 (7) | 0.012 (7) | 0.003 (6) |
| O26 | 0.114 (5) | 0.066 (4) | 0.043 (3) | −0.014 (3) | 0.001 (3) | −0.020 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—O22 | 1.323 (7) | C15—C16 | 1.564 (16) |
| C1—C6 | 1.405 (8) | C15—H15A | 0.9700 |
| C1—C2 | 1.410 (8) | C15—H15B | 0.9700 |
| C2—C3 | 1.397 (8) | C15A—C16 | 1.599 (16) |
| C2—C10 | 1.446 (8) | C15A—H15C | 0.9700 |
| C3—C4 | 1.375 (9) | C15A—H15D | 0.9700 |
| C3—H3A | 0.9300 | C16—C17A | 1.567 (13) |
| C4—C5 | 1.377 (10) | C16—C17 | 1.570 (13) |
| C4—N7 | 1.468 (9) | C16—H16A | 0.9700 |
| C5—C6 | 1.373 (9) | C16—H16B | 0.9700 |
| C5—H5A | 0.9300 | C17—N18 | 1.503 (12) |
| C6—H6A | 0.9300 | C17—H17A | 0.9800 |
| N7—O9 | 1.215 (10) | C17A—N18 | 1.505 (12) |
| N7—O8 | 1.222 (9) | C17A—H17B | 0.9800 |
| C10—N11 | 1.284 (7) | N18—V19 | 2.109 (5) |
| C10—H10A | 0.9300 | N18—H18A | 0.9000 |
| N11—C12 | 1.487 (15) | N18—H18B | 0.9000 |
| N11—C12A | 1.526 (15) | V19—O20 | 1.610 (4) |
| N11—V19 | 2.186 (5) | V19—O21$^{i}$ | 1.663 (4) |
| C12—C13 | 1.507 (16) | V19—O22 | 1.929 (4) |
| C12—C17 | 1.529 (18) | V19—O21 | 2.372 (4) |
| C12—H12A | 0.9800 | O21—V19$^{i}$ | 1.663 (4) |
| C12A—C17A | 1.51 (2) | S23—O26 | 1.489 (6) |
| C12A—C13 | 1.534 (16) | S23—C24 | 1.744 (10) |
| C12A—H12B | 0.9800 | S23—C25 | 1.761 (11) |
| C13—C14 | 1.503 (9) | C24—H24A | 0.9600 |
| C13—H13A | 0.9700 | C24—H24B | 0.9600 |
| C13—H13B | 0.9700 | C24—H24C | 0.9600 |

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|---|---|---|---|
| C14—C15 | 1.409 (15) | C25—H25A | 0.9600 |
| C14—C15A | 1.439 (16) | C25—H25B | 0.9600 |
| C14—H14A | 0.9700 | C25—H25C | 0.9600 |
| C14—H14B | 0.9700 | | |

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|---|---|---|---|
| O22—C1—C6 | 118.8 (5) | C14—C15A—H15D | 110.0 |
| O22—C1—C2 | 122.3 (5) | C16—C15A—H15D | 110.0 |
| C6—C1—C2 | 118.9 (5) | H15C—C15A—H15D | 108.3 |
| C3—C2—C1 | 119.3 (5) | C15—C16—C17A | 116.3 (7) |
| C3—C2—C10 | 117.8 (6) | C15—C16—C17 | 105.1 (8) |
| C1—C2—C10 | 122.9 (5) | C17A—C16—C15A | 104.9 (9) |
| C4—C3—C2 | 119.5 (6) | C17—C16—C15A | 118.5 (8) |
| C4—C3—H3A | 120.3 | C15—C16—H16A | 110.7 |
| C2—C3—H3A | 120.3 | C17A—C16—H16A | 77.7 |
| C3—C4—C5 | 122.2 (6) | C17—C16—H16A | 110.7 |
| C3—C4—N7 | 118.8 (7) | C15A—C16—H16A | 69.4 |
| C5—C4—N7 | 119.0 (6) | C15—C16—H16B | 110.7 |
| C6—C5—C4 | 118.8 (6) | C17A—C16—H16B | 126.2 |
| C6—C5—H5A | 120.6 | C17—C16—H16B | 110.7 |
| C4—C5—H5A | 120.6 | C15A—C16—H16B | 127.8 |
| C5—C6—C1 | 121.1 (6) | H16A—C16—H16B | 108.8 |
| C5—C6—H6A | 119.4 | N18—C17—C12 | 106.1 (9) |
| C1—C6—H6A | 119.4 | N18—C17—C16 | 109.3 (8) |
| O9—N7—O8 | 124.1 (7) | C12—C17—C16 | 107.9 (9) |
| O9—N7—C4 | 118.3 (7) | N18—C17—H17A | 111.1 |
| O8—N7—C4 | 117.6 (8) | C12—C17—H17A | 111.1 |
| N11—C10—C2 | 124.0 (5) | C16—C17—H17A | 111.1 |
| N11—C10—H10A | 118.0 | N18—C17A—C12A | 105.7 (10) |
| C2—C10—H10A | 118.0 | N18—C17A—C16 | 109.3 (8) |
| C10—N11—C12 | 120.6 (7) | C12A—C17A—C16 | 104.9 (10) |
| C10—N11—C12A | 118.6 (7) | N18—C17A—H17B | 112.2 |
| C10—N11—V19 | 125.9 (4) | C12A—C17A—H17B | 112.2 |
| C12—N11—V19 | 112.6 (6) | C16—C17A—H17B | 112.2 |
| C12A—N11—V19 | 113.3 (6) | C17—N18—V19 | 113.2 (5) |
| N11—C12—C13 | 117.7 (10) | C17A—N18—V19 | 112.5 (5) |
| N11—C12—C17 | 102.5 (10) | C17—N18—H18A | 108.9 |
| C13—C12—C17 | 112.1 (11) | C17A—N18—H18A | 77.3 |
| N11—C12—H12A | 108.1 | V19—N18—H18A | 108.9 |
| C13—C12—H12A | 108.1 | C17—N18—H18B | 108.9 |
| C17—C12—H12A | 108.1 | C17A—N18—H18B | 133.8 |
| C17A—C12A—N11 | 103.9 (11) | V19—N18—H18B | 108.9 |
| C17A—C12A—C13 | 110.9 (11) | H18A—N18—H18B | 107.8 |
| N11—C12A—C13 | 113.7 (10) | O20—V19—O21$^{i}$ | 106.7 (2) |
| C17A—C12A—H12B | 109.4 | O20—V19—O22 | 101.0 (2) |
| N11—C12A—H12B | 109.4 | O21$^{i}$—V19—O22 | 99.26 (19) |
| C13—C12A—H12B | 109.4 | O20—V19—N18 | 94.0 (2) |
| C14—C13—C12 | 113.9 (7) | O21$^{i}$—V19—N18 | 93.45 (19) |
| C14—C13—C12A | 111.2 (7) | O22—V19—N18 | 156.55 (19) |

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| C14—C13—H13A | 108.8 | O20—V19—N11 | 98.4 (2) |
| C12—C13—H13A | 108.8 | O21$^{i}$—V19—N11 | 153.60 (19) |
| C12A—C13—H13A | 86.8 | O22—V19—N11 | 83.61 (18) |
| C14—C13—H13B | 108.8 | N18—V19—N11 | 76.42 (18) |
| C12—C13—H13B | 108.8 | O20—V19—O21 | 172.2 (2) |
| C12A—C13—H13B | 129.9 | O21$^{i}$—V19—O21 | 77.86 (17) |
| H13A—C13—H13B | 107.7 | O22—V19—O21 | 84.18 (16) |
| C15—C14—C15A | 46.2 (9) | N18—V19—O21 | 79.31 (17) |
| C15—C14—C13 | 118.0 (8) | N11—V19—O21 | 76.33 (16) |
| C15A—C14—C13 | 120.1 (9) | V19$^{i}$—O21—V19 | 102.14 (17) |
| C15—C14—H14A | 107.8 | C1—O22—V19 | 129.1 (4) |
| C15A—C14—H14A | 131.9 | O26—S23—C24 | 106.2 (4) |
| C13—C14—H14A | 107.8 | O26—S23—C25 | 107.2 (5) |
| C15—C14—H14B | 107.8 | C24—S23—C25 | 98.3 (6) |
| C15A—C14—H14B | 63.4 | S23—C24—H24A | 109.5 |
| C13—C14—H14B | 107.8 | S23—C24—H24B | 109.5 |
| H14A—C14—H14B | 107.2 | H24A—C24—H24B | 109.5 |
| C14—C15—C16 | 112.3 (9) | S23—C24—H24C | 109.5 |
| C14—C15—H15A | 109.1 | H24A—C24—H24C | 109.5 |
| C16—C15—H15A | 109.1 | H24B—C24—H24C | 109.5 |
| C14—C15—H15B | 109.1 | S23—C25—H25A | 109.5 |
| C16—C15—H15B | 109.1 | S23—C25—H25B | 109.5 |
| H15A—C15—H15B | 107.9 | H25A—C25—H25B | 109.5 |
| C14—C15A—C16 | 108.7 (10) | S23—C25—H25C | 109.5 |
| C14—C15A—H15C | 110.0 | H25A—C25—H25C | 109.5 |
| C16—C15A—H15C | 110.0 | H25B—C25—H25C | 109.5 |

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|---|---|---|---|
| O22—C1—C2—C3 | −176.9 (5) | C15—C16—C17—N18 | −179.6 (8) |
| C6—C1—C2—C3 | 0.2 (8) | C17A—C16—C17—N18 | −64.4 (11) |
| O22—C1—C2—C10 | 4.5 (8) | C15A—C16—C17—N18 | −137.2 (10) |
| C6—C1—C2—C10 | −178.4 (5) | C15—C16—C17—C12 | −64.7 (12) |
| C1—C2—C3—C4 | −1.6 (9) | C17A—C16—C17—C12 | 50.5 (11) |
| C10—C2—C3—C4 | 177.1 (6) | C15A—C16—C17—C12 | −22.3 (14) |
| C2—C3—C4—C5 | −0.1 (11) | N11—C12A—C17A—N18 | 55.5 (12) |
| C2—C3—C4—N7 | −179.9 (7) | C13—C12A—C17A—N18 | 178.1 (9) |
| C3—C4—C5—C6 | 3.0 (12) | N11—C12A—C17A—C16 | 170.9 (9) |
| N7—C4—C5—C6 | −177.2 (7) | C13—C12A—C17A—C16 | −66.5 (13) |
| C4—C5—C6—C1 | −4.4 (11) | C15—C16—C17A—N18 | 141.3 (9) |
| O22—C1—C6—C5 | −180.0 (6) | C17—C16—C17A—N18 | 64.3 (12) |
| C2—C1—C6—C5 | 2.8 (9) | C15A—C16—C17A—N18 | −176.0 (9) |
| C3—C4—N7—O9 | −13.0 (13) | C15—C16—C17A—C12A | 28.3 (14) |
| C5—C4—N7—O9 | 167.2 (9) | C17—C16—C17A—C12A | −48.7 (12) |
| C3—C4—N7—O8 | 166.1 (8) | C15A—C16—C17A—C12A | 71.0 (12) |
| C5—C4—N7—O8 | −13.7 (13) | C12—C17—N18—C17A | −51.4 (11) |
| C3—C2—C10—N11 | −165.8 (6) | C16—C17—N18—C17A | 64.7 (11) |
| C1—C2—C10—N11 | 12.8 (9) | C12—C17—N18—V19 | 45.1 (10) |
| C2—C10—N11—C12 | −167.3 (8) | C16—C17—N18—V19 | 161.1 (6) |
| C2—C10—N11—C12A | 163.0 (8) | C12A—C17A—N18—C17 | 47.5 (11) |

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| C2—C10—N11—V19 | 1.0 (8) | C16—C17A—N18—C17 | −64.9 (12) |
| C10—N11—C12—C13 | −20.5 (15) | C12A—C17A—N18—V19 | −51.1 (11) |
| C12A—N11—C12—C13 | 73 (2) | C16—C17A—N18—V19 | −163.6 (6) |
| V19—N11—C12—C13 | 169.7 (8) | C17—N18—V19—O20 | 82.6 (6) |
| C10—N11—C12—C17 | −144.0 (8) | C17A—N18—V19—O20 | 120.2 (7) |
| C12A—N11—C12—C17 | −50.9 (19) | C17—N18—V19—O21$^{i}$ | −170.4 (6) |
| V19—N11—C12—C17 | 46.2 (11) | C17A—N18—V19—O21$^{i}$ | −132.7 (7) |
| C10—N11—C12A—C17A | 157.6 (8) | C17—N18—V19—O22 | −47.4 (8) |
| C12—N11—C12A—C17A | 56 (2) | C17A—N18—V19—O22 | −9.8 (9) |
| V19—N11—C12A—C17A | −38.3 (12) | C17—N18—V19—N11 | −15.1 (6) |
| C10—N11—C12A—C13 | 36.8 (14) | C17A—N18—V19—N11 | 22.6 (6) |
| C12—N11—C12A—C13 | −65 (2) | C17—N18—V19—O21 | −93.4 (6) |
| V19—N11—C12A—C13 | −159.0 (8) | C17A—N18—V19—O21 | −55.8 (7) |
| N11—C12—C13—C14 | −162.4 (9) | C10—N11—V19—O20 | 80.1 (5) |
| C17—C12—C13—C14 | −43.9 (13) | C12—N11—V19—O20 | −110.8 (7) |
| N11—C12—C13—C12A | −73 (2) | C12A—N11—V19—O20 | −82.7 (8) |
| C17—C12—C13—C12A | 45.4 (18) | C10—N11—V19—O21$^{i}$ | −118.1 (6) |
| C17A—C12A—C13—C14 | 49.1 (14) | C12—N11—V19—O21$^{i}$ | 51.0 (9) |
| N11—C12A—C13—C14 | 165.8 (9) | C12A—N11—V19—O21$^{i}$ | 79.1 (9) |
| C17A—C12A—C13—C12 | −52 (2) | C10—N11—V19—O22 | −20.1 (5) |
| N11—C12A—C13—C12 | 64.4 (19) | C12—N11—V19—O22 | 149.0 (7) |
| C12—C13—C14—C15 | 39.7 (14) | C12A—N11—V19—O22 | 177.1 (7) |
| C12A—C13—C14—C15 | 12.1 (14) | C10—N11—V19—N18 | 172.2 (5) |
| C12—C13—C14—C15A | −13.6 (14) | C12—N11—V19—N18 | −18.6 (7) |
| C12A—C13—C14—C15A | −41.2 (14) | C12A—N11—V19—N18 | 9.5 (7) |
| C15A—C14—C15—C16 | 57.2 (12) | C10—N11—V19—O21 | −105.7 (5) |
| C13—C14—C15—C16 | −48.9 (15) | C12—N11—V19—O21 | 63.5 (7) |
| C15—C14—C15A—C16 | −53.4 (10) | C12A—N11—V19—O21 | 91.6 (7) |
| C13—C14—C15A—C16 | 47.8 (15) | O21$^{i}$—V19—O21—V19$^{i}$ | 0.000 (2) |
| C14—C15—C16—C17A | 27.0 (15) | O22—V19—O21—V19$^{i}$ | 100.8 (2) |
| C14—C15—C16—C17 | 60.4 (13) | N18—V19—O21—V19$^{i}$ | −95.9 (2) |
| C14—C15—C16—C15A | −55.7 (12) | N11—V19—O21—V19$^{i}$ | −174.4 (2) |
| C14—C15A—C16—C15 | 52.1 (11) | C6—C1—O22—V19 | 144.0 (4) |
| C14—C15A—C16—C17A | −60.9 (13) | C2—C1—O22—V19 | −38.9 (7) |
| C14—C15A—C16—C17 | −28.2 (15) | O20—V19—O22—C1 | −58.2 (5) |
| N11—C12—C17—N18 | −57.2 (11) | O21$^{i}$—V19—O22—C1 | −167.4 (5) |
| C13—C12—C17—N18 | 175.7 (9) | N18—V19—O22—C1 | 70.7 (7) |
| N11—C12—C17—C16 | −174.2 (8) | N11—V19—O22—C1 | 39.1 (5) |
| C13—C12—C17—C16 | 58.7 (13) | O21—V19—O22—C1 | 116.0 (5) |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N18—H18*A*···O22$^{i}$ | 0.90 | 2.29 | 3.069 (6) | 145 |
| N18—H18*B*···O26$^{ii}$ | 0.90 | 2.16 | 2.913 (8) | 140 |
| C5—H5*A*···O26$^{iii}$ | 0.93 | 2.57 | 3.494 (9) | 171 |

| | | | | |
|---|---|---|---|---|
| C10—H10*A*···O21$^{iv}$ | 0.93 | 2.49 | 3.167 (7) | 130 |
| C13—H13*B*···O21$^{iv}$ | 0.97 | 2.55 | 3.392 (7) | 145 |
| C14—H14*A*···O26$^{v}$ | 0.97 | 2.38 | 3.211 (11) | 144 |
| C15—H15*B*···O8$^{vi}$ | 0.97 | 2.32 | 3.109 (17) | 138 |

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