Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Di-μ-oxido-bis({2,2′-[propane-1,3-diylbis(nitrilomethylidyne)]diphenolato}-vanadium(IV)) dimethylformamide disolvate

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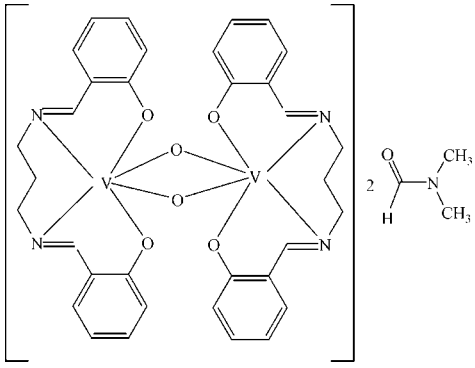
Received 5 October 2007; accepted 11 October 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.133; data-to-parameter ratio = 12.9.

In the title compound, $[V_2(C_{17}H_{17}N_2O_2)_2O_2]\cdot 2C_3H_7NO$, each V atom is chelated by a Schiff base ligand *via* two N and two O atoms. The two octahedrally coordinated V atoms are linked by two bridging oxide ligands to give a centrosymmetric dimer.

Related literature

For related literature, see: Garnovskii *et al.* (1993); Gresser & Tracey (1986); Huang *et al.* (2002); Posner *et al.* (1994).



Experimental

Crystal data

$[V_2(C_{17}H_{17}N_2O_2)_2O_2]\cdot 2C_3H_7NO$
M_r = 840.71
Monoclinic, $P2_1/c$
a = 10.9161 (5) Å
b = 9.9986 (5) Å
c = 17.7812 (10) Å
β = 101.291 (2)°
V = 1903.18 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 293 (2) K
0.43 × 0.28 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.797, T_{max} = 0.888
10745 measured reflections
3265 independent reflections
2841 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.134
S = 1.00
3265 reflections
253 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.95 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

The authors thank Liaocheng University for financial support and Professor Jianmin Dou for his help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2153).

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 doi:10.1107/S1600536807049987

supplementary materials

Acta Cryst. (2007). E**63**, m2731 [**doi:10.1107/S1600536807049987**]

Di-μ-oxido-bis({2,2'-[propane-1,3-diylbis(nitrilomethylidyne)]diphenolato}vanadium(IV)) dimethylformamide disolvate

Y. Liu, J. Dou, M. Niu and X. Zhang

Comment

Schiff base ligands have been studied for a long time due to their easy synthesis and versatile complexing properties. They play an important role in the development of coordination chemistry as well as inorganic biochemistry, catalysis, optical materials *etc*. (Garnovskii *et al.*, 1993; Huang *et al.*, 2002). The richness and importance of vanadate chemistry (Gresser & Tracey, 1986; Posner *et al.*, 1994) have aroused our particular interest in designing new vanadium complexes with ONNO-donor ligands. In this paper, we report the structure of the title compound, (I).

As shown in Fig. 1, each V atom is chelated by a Schiff base ligand *via* two N and two O atoms. The two octahedrally coordinated V atoms are linked by two bridging oxide ligands to give a centrosymmetric dimer. The V—N and V—O bond lengths are in the ranges 2.028 (2)–2.078 (2) and 1.8187 (18)–1.9364 (19) %A, respectively.

Experimental

A mixture of vanadium(IV) sulfate (1 mmol) and *N*,*N*'-bis(2-hydroxybenzyl)-1,3-propanediamine (1 mmol) in 20 ml me thanol was refluxed for two hours. The above solution was filtered and the filtrate was allowed to evaporate naturally at room temperature. Two days later, blue blocks of (I) were obtained with a yield of 16%. Anal. calc. for $C_{40}H_{46}V_2N_6O_8$: C 56.56, H 5.42, N 6.60%; found: C 56.52, H 5.44, N 6.51%.

Refinement

All H atoms were placed in calculated positions with C—H = 0.93–0.96Å and refined as riding with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

Figures

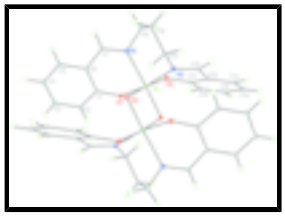


Fig. 1. The molecular structure of (I), drawn with 30% probability displacement ellipsoids for the non-hydrogen atoms. [Symmetry code for unlabelled atoms: $-x + 1, -y + 1, -z$.]
Fig. 2. Dimer of the title compound.

Di-μ-oxido-bis({2,2'-[propane-1,3- diylbis(nitrilomethylidyne)]diphenolato}vanadium(IV)) dimethylformamide disolvate

Crystal data

$[V_2(C_{17}H_{17}N_2O_2)_2O_2]\cdot 2C_3H_7NO$	$F_{000} = 876$
$M_r = 840.71$	$D_x = 1.467$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3265 reflections
$a = 10.9161$ (5) Å	$\theta = 3.1$–25.0º
$b = 9.9986$ (5) Å	$\mu = 0.55$ mm^{-1}
$c = 17.7812$ (10) Å	$T = 293$ (2) K
$\beta = 101.291$ (2)º	Block, blue
$V = 1903.18$ (17) Å^3	0.43 × 0.28 × 0.22 mm
$Z = 2$	

Data collection

Bruker APEXII CCD area-detector diffractometer	3265 independent reflections
Radiation source: fine-focus sealed tube	2841 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.032$
$T = 293$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 3.1$º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -12\rightarrow12$
$T_{min} = 0.797$, $T_{max} = 0.888$	$k = -11\rightarrow11$
10745 measured reflections	$l = -20\rightarrow20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters not refined
$wR(F^2) = 0.134$	$w = 1/[\sigma^2(F_o^2) + (0.089P)^2 + 1.6075P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
3265 reflections	$\Delta\rho_{max} = 0.95$ e Å^{-3}
253 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.50502 (3)	0.37734 (4)	0.03285 (2)	0.0161 (4)
O1	0.54652 (17)	0.43578 (18)	0.13786 (10)	0.0216 (4)
O2	0.63819 (17)	0.24749 (18)	0.05886 (11)	0.0231 (4)
N2	0.4575 (2)	0.2833 (2)	−0.06976 (13)	0.0201 (5)
C17	0.7134 (2)	0.2146 (2)	0.01051 (16)	0.0214 (6)
N1	0.3655 (2)	0.2627 (2)	0.06550 (13)	0.0211 (5)
C11	0.5372 (3)	0.2249 (2)	−0.10443 (15)	0.0214 (6)
H11A	0.5080	0.1932	−0.1539	0.026*
C16	0.8407 (3)	0.1822 (3)	0.04046 (16)	0.0242 (6)
H16A	0.8714	0.1874	0.0930	0.029*
C10	0.3227 (3)	0.2801 (3)	−0.10458 (16)	0.0245 (6)
H10A	0.3121	0.2555	−0.1582	0.029*
H10B	0.2871	0.3684	−0.1017	0.029*
C5	0.2591 (3)	0.4421 (3)	0.22177 (16)	0.0276 (6)
H5A	0.1824	0.3990	0.2169	0.033*
C7	0.3077 (2)	0.2952 (3)	0.12027 (16)	0.0233 (6)
H7A	0.2386	0.2444	0.1258	0.028*
C1	0.4629 (2)	0.4667 (3)	0.18088 (14)	0.0201 (6)
C8	0.3227 (3)	0.1436 (3)	0.01943 (17)	0.0268 (6)
H8A	0.2677	0.0922	0.0451	0.032*
H8B	0.3944	0.0881	0.0161	0.032*
C15	0.9199 (3)	0.1426 (3)	−0.00830 (18)	0.0276 (6)
H15A	1.0029	0.1229	0.0124	0.033*
C9	0.2529 (3)	0.1782 (3)	−0.06243 (17)	0.0311 (7)
H9A	0.1715	0.2144	−0.0596	0.037*
H9B	0.2397	0.0965	−0.0924	0.037*
C2	0.4954 (3)	0.5621 (3)	0.24014 (16)	0.0256 (6)
H2A	0.5749	0.5997	0.2493	0.031*
C6	0.3432 (3)	0.4046 (3)	0.17318 (16)	0.0224 (6)
C4	0.2908 (3)	0.5423 (3)	0.27629 (16)	0.0285 (6)
H4A	0.2345	0.5701	0.3062	0.034*
C12	0.6699 (3)	0.2055 (3)	−0.07122 (16)	0.0226 (6)
C14	0.8766 (3)	0.1315 (3)	−0.08890 (19)	0.0320 (7)

H14A	0.9301	0.1044	−0.1207	0.038*
C13	0.7522 (3)	0.1622 (3)	−0.11906 (17)	0.0266 (6)
H13A	0.7224	0.1542	−0.1716	0.032*
C3	0.4097 (3)	0.6008 (3)	0.28523 (17)	0.0285 (7)
H3A	0.4321	0.6670	0.3221	0.034*
N3	0.9330 (2)	0.5321 (3)	0.16965 (14)	0.0329 (6)
O4	0.9442 (2)	0.6587 (3)	0.27878 (13)	0.0459 (6)
C18	0.9720 (3)	0.6352 (3)	0.2145 (2)	0.0376 (8)
H18A	1.0246	0.6960	0.1970	0.045*
C19	0.8517 (3)	0.4318 (3)	0.1944 (2)	0.0375 (7)
H19A	0.8365	0.4553	0.2441	0.056*
H19B	0.8913	0.3458	0.1969	0.056*
H19C	0.7737	0.4286	0.1584	0.056*
C20	0.9680 (3)	0.5130 (4)	0.09462 (19)	0.0442 (8)
H20A	1.0212	0.5850	0.0852	0.066*
H20B	0.8941	0.5118	0.0552	0.066*
H20C	1.0117	0.4296	0.0944	0.066*
O3	0.39013 (16)	0.50834 (18)	0.00318 (10)	0.0204 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.0171 (9)	0.0190 (9)	0.0131 (9)	0.0006 (7)	0.0049 (7)	−0.0003 (7)
O1	0.0235 (10)	0.0223 (10)	0.0197 (10)	0.0004 (7)	0.0059 (8)	0.0003 (8)
O2	0.0258 (10)	0.0210 (9)	0.0236 (10)	0.0038 (7)	0.0076 (8)	0.0012 (8)
N2	0.0218 (11)	0.0163 (11)	0.0228 (12)	−0.0016 (8)	0.0059 (9)	0.0018 (9)
C17	0.0253 (14)	0.0131 (12)	0.0271 (15)	0.0006 (10)	0.0087 (11)	0.0021 (10)
N1	0.0228 (11)	0.0200 (11)	0.0208 (12)	0.0006 (9)	0.0052 (9)	0.0010 (9)
C11	0.0314 (15)	0.0155 (12)	0.0183 (14)	−0.0023 (10)	0.0074 (11)	−0.0001 (10)
C16	0.0273 (15)	0.0199 (14)	0.0264 (15)	−0.0009 (11)	0.0074 (12)	0.0025 (11)
C10	0.0254 (14)	0.0231 (14)	0.0245 (15)	−0.0036 (11)	0.0034 (11)	−0.0032 (11)
C5	0.0294 (15)	0.0291 (15)	0.0267 (15)	0.0021 (12)	0.0114 (12)	0.0026 (12)
C7	0.0209 (13)	0.0214 (14)	0.0286 (15)	0.0013 (10)	0.0069 (11)	0.0046 (11)
C1	0.0235 (13)	0.0206 (13)	0.0165 (13)	0.0042 (10)	0.0048 (10)	0.0038 (10)
C8	0.0273 (15)	0.0190 (13)	0.0362 (17)	−0.0053 (11)	0.0119 (13)	−0.0014 (12)
C15	0.0209 (14)	0.0243 (14)	0.0385 (18)	0.0026 (11)	0.0077 (12)	0.0030 (13)
C9	0.0320 (16)	0.0331 (16)	0.0292 (17)	−0.0111 (13)	0.0085 (13)	−0.0065 (13)
C2	0.0312 (15)	0.0225 (14)	0.0235 (15)	−0.0021 (11)	0.0068 (12)	−0.0002 (12)
C6	0.0242 (14)	0.0210 (13)	0.0224 (15)	0.0034 (11)	0.0059 (11)	0.0028 (11)
C4	0.0340 (16)	0.0300 (15)	0.0245 (16)	0.0068 (12)	0.0128 (12)	0.0022 (12)
C12	0.0288 (15)	0.0150 (13)	0.0257 (15)	0.0005 (10)	0.0092 (12)	0.0015 (11)
C14	0.0297 (16)	0.0296 (16)	0.0423 (19)	0.0030 (12)	0.0205 (14)	0.0019 (13)
C13	0.0324 (16)	0.0236 (14)	0.0254 (15)	0.0011 (12)	0.0097 (12)	0.0006 (12)
C3	0.0391 (17)	0.0245 (14)	0.0225 (16)	0.0021 (12)	0.0078 (13)	−0.0017 (11)
N3	0.0333 (14)	0.0319 (14)	0.0324 (15)	0.0003 (11)	0.0037 (11)	0.0008 (11)
O4	0.0515 (15)	0.0464 (14)	0.0384 (14)	−0.0017 (11)	0.0051 (11)	−0.0102 (11)
C18	0.0338 (17)	0.0344 (18)	0.043 (2)	−0.0042 (13)	0.0033 (15)	−0.0017 (14)
C19	0.0357 (17)	0.0321 (17)	0.044 (2)	−0.0005 (13)	0.0068 (14)	0.0001 (14)

C20	0.048 (2)	0.043 (2)	0.042 (2)	−0.0009 (16)	0.0111 (16)	−0.0048 (16)
O3	0.0210 (10)	0.0190 (9)	0.0217 (10)	0.0001 (7)	0.0053 (7)	0.0007 (8)

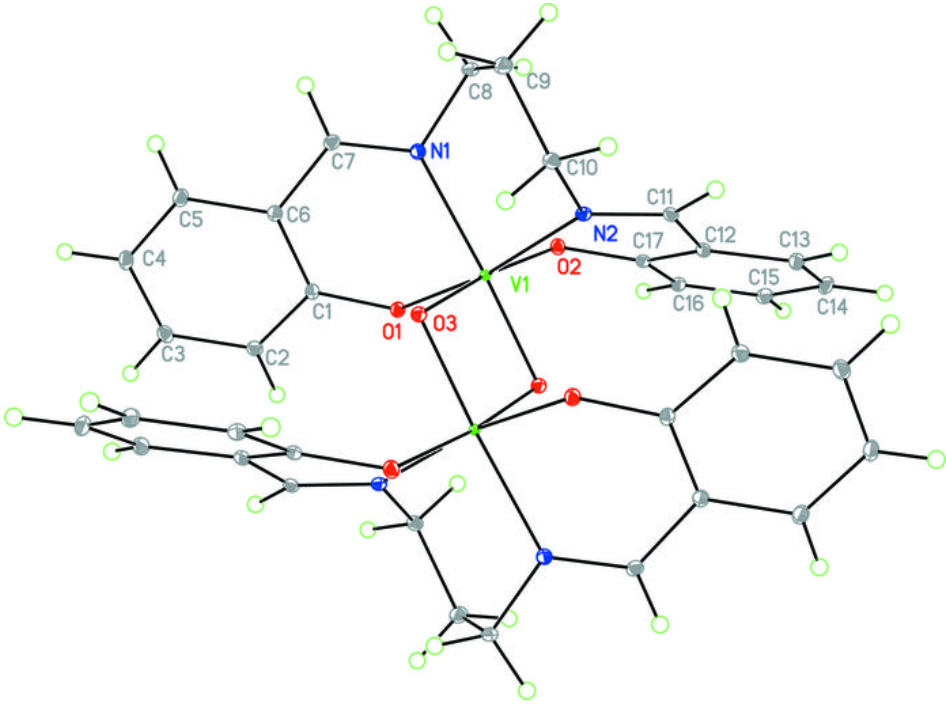
Geometric parameters (Å, °)

V1—O3	1.8187 (18)	C8—C9	1.544 (4)
V1—O3^{i}	1.8204 (17)	C8—H8A	0.970
V1—O1	1.9240 (18)	C8—H8B	0.970
V1—O2	1.9364 (19)	C15—C14	1.423 (5)
V1—N2	2.028 (2)	C15—H15A	0.930
V1—N1	2.078 (2)	C9—H9A	0.970
V1—V1^{i}	2.7098 (7)	C9—H9B	0.970
O1—C1	1.337 (3)	C2—C3	1.401 (4)
O2—C17	1.340 (3)	C2—H2A	0.930
N2—C11	1.299 (3)	C4—C3	1.403 (4)
N2—C10	1.481 (3)	C4—H4A	0.930
C17—C16	1.425 (4)	C12—C13	1.420 (4)
C17—C12	1.441 (4)	C14—C13	1.393 (4)
N1—C7	1.301 (3)	C14—H14A	0.930
N1—C8	1.468 (3)	C13—H13A	0.930
C11—C12	1.467 (4)	C3—H3A	0.930
C11—H11A	0.930	N3—C18	1.322 (4)
C16—C15	1.396 (4)	N3—C19	1.463 (4)
C16—H16A	0.930	N3—C20	1.470 (4)
C10—C9	1.550 (4)	O4—C18	1.261 (4)
C10—H10A	0.970	C18—H18A	0.930
C10—H10B	0.970	C19—H19A	0.960
C5—C4	1.391 (4)	C19—H19B	0.960
C5—C6	1.428 (4)	C19—H19C	0.960
C5—H5A	0.930	C20—H20A	0.960
C7—C6	1.446 (4)	C20—H20B	0.960
C7—H7A	0.930	C20—H20C	0.960
C1—C2	1.414 (4)	O3—V1^{i}	1.8204 (17)
C1—C6	1.430 (4)		
O3—V1—O3^{i}	83.74 (8)	N1—C8—H8A	109.0
O3—V1—O1	94.64 (8)	C9—C8—H8A	109.0
O3^{i}—V1—O1	96.08 (8)	N1—C8—H8B	109.0
O3—V1—O2	174.25 (8)	C9—C8—H8B	109.0
O3^{i}—V1—O2	90.61 (8)	H8A—C8—H8B	107.8
O1—V1—O2	87.06 (8)	C16—C15—C14	121.6 (3)
O3—V1—N2	91.86 (8)	C16—C15—H15A	119.2
O3^{i}—V1—N2	92.43 (8)	C14—C15—H15A	119.2
O1—V1—N2	169.80 (8)	C8—C9—C10	113.9 (2)
O2—V1—N2	87.24 (8)	C8—C9—H9A	108.8
O3—V1—N1	88.68 (8)	C10—C9—H9A	108.8
O3^{i}—V1—N1	172.10 (8)	C8—C9—H9B	108.8
O1—V1—N1	86.71 (8)	C10—C9—H9B	108.8

O2—V1—N1	96.92 (8)	H9A—C9—H9B	107.7
N2—V1—N1	85.58 (9)	C3—C2—C1	120.9 (3)
O3—V1—V1^{i}	41.90 (5)	C3—C2—H2A	119.5
O3^{i}—V1—V1^{i}	41.85 (6)	C1—C2—H2A	119.5
O1—V1—V1^{i}	97.21 (6)	C5—C6—C1	120.8 (3)
O2—V1—V1^{i}	132.45 (6)	C5—C6—C7	118.1 (2)
N2—V1—V1^{i}	92.88 (6)	C1—C6—C7	121.0 (2)
N1—V1—V1^{i}	130.54 (7)	C5—C4—C3	118.6 (3)
C1—O1—V1	124.62 (16)	C5—C4—H4A	120.7
C17—O2—V1	122.43 (17)	C3—C4—H4A	120.7
C11—N2—C10	119.7 (2)	C13—C12—C17	120.1 (3)
C11—N2—V1	123.91 (19)	C13—C12—C11	119.3 (3)
C10—N2—V1	116.40 (16)	C17—C12—C11	120.1 (2)
O2—C17—C16	119.4 (2)	C13—C14—C15	118.5 (3)
O2—C17—C12	122.7 (2)	C13—C14—H14A	120.8
C16—C17—C12	117.8 (2)	C15—C14—H14A	120.8
C7—N1—C8	118.2 (2)	C14—C13—C12	121.3 (3)
C7—N1—V1	124.11 (19)	C14—C13—H13A	119.3
C8—N1—V1	117.49 (16)	C12—C13—H13A	119.3
N2—C11—C12	124.7 (2)	C2—C3—C4	121.8 (3)
N2—C11—H11A	117.7	C2—C3—H3A	119.1
C12—C11—H11A	117.7	C4—C3—H3A	119.1
C15—C16—C17	120.6 (3)	C18—N3—C19	119.7 (3)
C15—C16—H16A	119.7	C18—N3—C20	122.6 (3)
C17—C16—H16A	119.7	C19—N3—C20	117.7 (3)
N2—C10—C9	110.7 (2)	O4—C18—N3	125.9 (3)
N2—C10—H10A	109.5	O4—C18—H18A	117.0
C9—C10—H10A	109.5	N3—C18—H18A	117.0
N2—C10—H10B	109.5	N3—C19—H19A	109.5
C9—C10—H10B	109.5	N3—C19—H19B	109.5
H10A—C10—H10B	108.1	H19A—C19—H19B	109.5
C4—C5—C6	120.6 (3)	N3—C19—H19C	109.5
C4—C5—H5A	119.7	H19A—C19—H19C	109.5
C6—C5—H5A	119.7	H19B—C19—H19C	109.5
N1—C7—C6	124.7 (2)	N3—C20—H20A	109.5
N1—C7—H7A	117.7	N3—C20—H20B	109.5
C6—C7—H7A	117.7	H20A—C20—H20B	109.5
O1—C1—C2	118.8 (2)	N3—C20—H20C	109.5
O1—C1—C6	123.9 (2)	H20A—C20—H20C	109.5
C2—C1—C6	117.1 (2)	H20B—C20—H20C	109.5
N1—C8—C9	112.9 (2)	V1—O3—V1^{i}	96.26 (8)

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

Fig. 1


C8
C9
N1
C7
C10
C11
C5
C6
N2
C17
C12
C13
C4
O2
V1
C15
C16
C14
C1
O1
O3
C3
C2