Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(4-Chlorobenzohydrazidato-κ^2N',O)-[2-(4-chlorobenzoylhydrazono-κ^2N,O)-propionato(2−)-κO]oxidovanadium(V)

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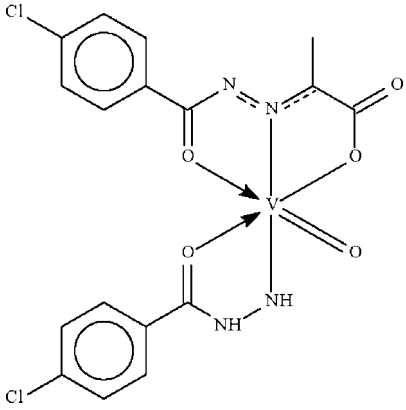
Received 21 May 2009; accepted 26 May 2009

Key indicators: single-crystal X-ray study; T = 119 K; mean σ(C–C) = 0.007 Å; R factor = 0.059; wR factor = 0.175; data-to-parameter ratio = 17.5.

In the crystal structure of the title compound, [VO($C_7H_6ClN_2O$)($C_{10}H_7ClN_2O_3$)], the V^V atom is N,O-chelated by the chlorobenzoylhydrazidate anion and O,N,O'-chelated by the (chlorobenzoylhydrazono)propionate dianion. The distorted octahedral *trans*-N_2O_4 coordination geometry is completed by the vanadyl O atom. In the crystal, molecules are linked by N—H···O hydrogen bonds into a linear chain parallel to [010].

Related literature

For the analogous vanadyl complex without the chlorine substituent in the two ligands, see: Wong *et al.* (2009).



Experimental

Crystal data

[V($C_7H_6ClN_2O$)($C_{10}H_7ClN_2O_3$)O] M_r = 475.15
Monoclinic, $C2/c$
a = 26.628 (2) Å
b = 5.7109 (3) Å
c = 24.772 (1) Å
β = 100.396 (3)°
V = 3705.2 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.86 mm^{-1}
T = 119 K
0.40 × 0.04 × 0.04 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.724, T_{max} = 0.966
11303 measured reflections
4189 independent reflections
2154 reflections with $I > 2\sigma(I)$
R_{int} = 0.174

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.175
S = 0.97
4189 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.40 e Å^{-3}
$\Delta\rho_{min}$ = −1.20 e Å^{-3}

Table 1
Selected bond lengths (Å).

V1—N1	2.064 (4)	V1—O3	1.989 (3)
V1—N4	1.888 (4)	V1—O4	2.207 (4)
V1—O1	1.988 (4)	V1—O5	1.593 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2^{i}	0.88	1.92	2.744 (5)	156
N4—H4···O1^{i}	0.88	2.14	2.840 (5)	136

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); 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, 2009).

We thank the University of Malaya (RG020/09AFR) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2529).

References

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supporting information

Acta Cryst. (2009). E**65**, m718 [doi:10.1107/S1600536809019928]

(4-Chlorobenzohydrazidato-κ^2N',O)[2-(4-chlorobenzoylhydrazono-κ^2N,O)propionato(2−)-κO]oxidovanadium(V)

Hon Wee Wong, Kong Mun Lo and Seik Weng Ng

S1. Experimental

2-[*p*-Chlorobenzoylhydrazono]propionic acid was prepared from the condensation reaction of *p*-chlorobenzhydrazide and pyruvic acid. The compound (0.70 g, 3 mmol) and vanadyl sulfate (0.25 g, 1.5 mmol) in 20 ml of distilled water were heated for 5 h. Slow evaporation of the filtrate gave orange crystals.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C). The nitrogen-bound H-atoms were similarly treated (N–H 0.88 Å).

All phenylene rings were refined was rigid hexagons of 1.39 Å sides.

The final difference Fourier map had a large peak/deep hole in the vicnity of the V1 atom.

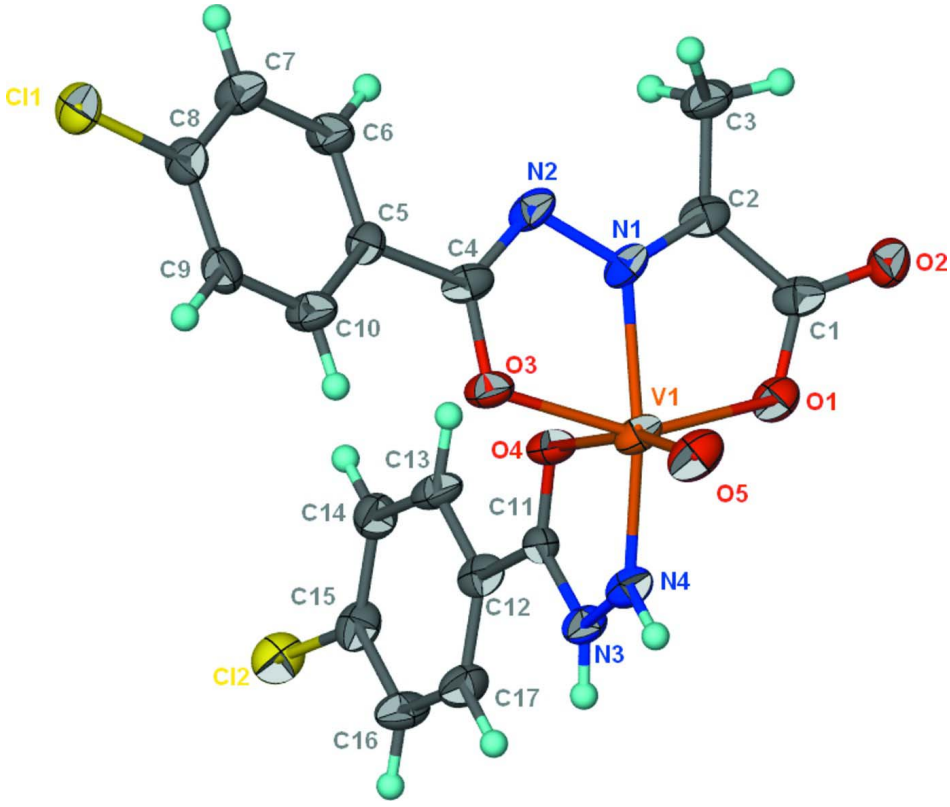

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $VO(C_7H_6ClN_2O)(C_{10}H_7ClN_2O_3)$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

(4-Chlorobenzohydrazidato-κ^2N',O)[2-(4- chlorobenzoylhydrazono-κ^2N,O)propionato(2-)-κO]oxidovanadium(V)

Crystal data

$[V(C_7H_6ClN_2O)(C_{10}H_7ClN_2O_3)O]$
$M_r = 475.15$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 26.628$ (2) Å
$b = 5.7109$ (3) Å
$c = 24.772$ (1) Å
$\beta = 100.396$ (3)°
$V = 3705.2$ (4) Å^3
$Z = 8$
$F(000) = 1920$
$D_x = 1.704$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 957 reflections
$\theta = 2.5$–$23.1°$
$\mu = 0.86$ mm^{-1}
$T = 119$ K
Prism, orange
0.40 × 0.04 × 0.04 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.724$, $T_{max} = 0.966$
11303 measured reflections
4189 independent reflections
2154 reflections with $I > 2\sigma(I)$
$R_{int} = 0.174$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -34 \rightarrow 34$
$k = -7 \rightarrow 7$
$l = -32 \rightarrow 30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.175$
$S = 0.97$
4189 reflections
239 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
$w = 1/[\sigma^2(F_o^2) + (0.0714P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.40$ e Å^{-3}
$\Delta\rho_{min} = -1.20$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.66692 (3)	0.32434 (15)	0.27882 (4)	0.0216 (3)
Cl1	0.44158 (5)	0.7483 (2)	0.46938 (6)	0.0289 (4)
Cl2	0.84139 (5)	−0.6026 (2)	0.53131 (6)	0.0294 (4)
O1	0.67515 (13)	0.1122 (6)	0.21738 (17)	0.0236 (9)
O2	0.63441 (13)	−0.1245 (6)	0.15164 (17)	0.0281 (9)
O3	0.62899 (12)	0.4195 (6)	0.33748 (16)	0.0237 (9)
O4	0.69433 (12)	0.0349 (6)	0.33560 (16)	0.0222 (9)
O5	0.65919 (13)	0.5607 (6)	0.24415 (18)	0.0296 (10)
N1	0.60013 (15)	0.1326 (7)	0.2650 (2)	0.0203 (10)
N2	0.56469 (15)	0.1678 (7)	0.2986 (2)	0.0236 (11)
N3	0.76603 (15)	0.2341 (7)	0.34041 (19)	0.0206 (10)
H3	0.7990	0.2555	0.3516	0.025*
N4	0.73671 (15)	0.3820 (7)	0.3060 (2)	0.0215 (10)
H4	0.7506	0.5106	0.2958	0.026*
C1	0.63701 (18)	−0.0159 (9)	0.1950 (3)	0.0228 (12)
C2	0.59319 (18)	−0.0213 (9)	0.2266 (2)	0.0225 (12)
C3	0.55037 (18)	−0.1879 (9)	0.2136 (3)	0.0280 (14)
H3A	0.5521	−0.3016	0.2435	0.042*
H3B	0.5526	−0.2702	0.1794	0.042*
H3C	0.5180	−0.1025	0.2092	0.042*
C4	0.58356 (18)	0.3289 (8)	0.3341 (3)	0.0221 (12)
C5	0.55047 (11)	0.4234 (5)	0.37140 (15)	0.0196 (12)
C6	0.50488 (12)	0.3112 (5)	0.37506 (15)	0.0239 (13)
H6	0.4965	0.1671	0.3565	0.029*
C7	0.47159 (10)	0.4100 (5)	0.40592 (17)	0.0249 (13)
H7A	0.4404	0.3333	0.4084	0.030*
C8	0.48390 (11)	0.6209 (5)	0.43310 (15)	0.0227 (12)
C9	0.52949 (12)	0.7331 (5)	0.42944 (15)	0.0239 (12)
H9	0.5379	0.8772	0.4480	0.029*
C10	0.56278 (10)	0.6343 (5)	0.39859 (16)	0.0254 (13)
H10	0.5939	0.7110	0.3961	0.031*
C11	0.74052 (18)	0.0506 (8)	0.3562 (2)	0.0186 (12)
C12	0.76740 (11)	−0.1137 (5)	0.39758 (13)	0.0222 (13)
C13	0.73990 (8)	−0.3013 (5)	0.41306 (14)	0.0245 (13)

H13	0.7054	−0.3249	0.3958	0.029*
C14	0.76286 (11)	−0.4543 (5)	0.45377 (15)	0.0241 (12)
H14	0.7441	−0.5825	0.4644	0.029*
C15	0.81333 (11)	−0.4198 (5)	0.47899 (13)	0.0251 (13)
C16	0.84083 (9)	−0.2323 (6)	0.46350 (14)	0.0273 (13)
H16	0.8753	−0.2087	0.4807	0.033*
C17	0.81787 (10)	−0.0792 (5)	0.42280 (15)	0.0251 (13)
H17	0.8367	0.0489	0.4122	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.0156 (4)	0.0218 (5)	0.0292 (7)	0.0012 (4)	0.0087 (4)	0.0014 (4)
Cl1	0.0244 (6)	0.0351 (8)	0.0291 (9)	0.0057 (5)	0.0097 (6)	−0.0082 (6)
Cl2	0.0273 (7)	0.0282 (7)	0.0332 (10)	0.0029 (6)	0.0066 (6)	0.0046 (7)
O1	0.0189 (17)	0.0246 (19)	0.028 (2)	0.0046 (15)	0.0074 (17)	0.0015 (17)
O2	0.0217 (18)	0.040 (2)	0.024 (3)	0.0046 (17)	0.0079 (17)	−0.004 (2)
O3	0.0151 (16)	0.0256 (19)	0.031 (3)	−0.0022 (14)	0.0067 (17)	−0.0060 (18)
O4	0.0150 (16)	0.0225 (18)	0.029 (3)	−0.0032 (14)	0.0048 (17)	−0.0022 (17)
O5	0.0269 (19)	0.025 (2)	0.039 (3)	0.0037 (16)	0.013 (2)	0.0060 (19)
N1	0.018 (2)	0.019 (2)	0.027 (3)	0.0043 (17)	0.011 (2)	0.003 (2)
N2	0.018 (2)	0.024 (2)	0.032 (3)	0.0009 (18)	0.014 (2)	−0.004 (2)
N3	0.0137 (19)	0.025 (2)	0.023 (3)	0.0018 (17)	0.0037 (18)	0.005 (2)
N4	0.020 (2)	0.018 (2)	0.028 (3)	−0.0018 (17)	0.009 (2)	0.000 (2)
C1	0.016 (2)	0.020 (3)	0.033 (4)	0.003 (2)	0.004 (2)	−0.001 (3)
C2	0.016 (2)	0.021 (3)	0.030 (4)	0.004 (2)	0.004 (2)	0.003 (3)
C3	0.018 (2)	0.032 (3)	0.036 (4)	−0.004 (2)	0.010 (3)	−0.007 (3)
C4	0.018 (2)	0.017 (2)	0.032 (4)	0.000 (2)	0.005 (2)	0.004 (3)
C5	0.019 (2)	0.020 (3)	0.021 (3)	0.002 (2)	0.006 (2)	−0.003 (2)
C6	0.022 (2)	0.021 (3)	0.029 (4)	−0.003 (2)	0.006 (2)	−0.007 (3)
C7	0.021 (3)	0.021 (3)	0.034 (4)	0.000 (2)	0.009 (3)	−0.001 (3)
C8	0.022 (2)	0.023 (3)	0.023 (3)	0.005 (2)	0.006 (2)	−0.004 (2)
C9	0.026 (3)	0.026 (3)	0.019 (3)	0.000 (2)	0.003 (2)	−0.005 (2)
C10	0.017 (2)	0.028 (3)	0.033 (4)	−0.001 (2)	0.006 (2)	−0.003 (3)
C11	0.017 (2)	0.022 (3)	0.017 (3)	−0.003 (2)	0.005 (2)	−0.004 (2)
C12	0.019 (2)	0.018 (3)	0.031 (4)	0.002 (2)	0.011 (2)	−0.008 (2)
C13	0.016 (2)	0.024 (3)	0.036 (4)	−0.003 (2)	0.011 (2)	−0.006 (3)
C14	0.026 (3)	0.024 (3)	0.023 (3)	−0.002 (2)	0.006 (2)	0.000 (3)
C15	0.023 (3)	0.027 (3)	0.027 (4)	0.007 (2)	0.009 (3)	0.002 (3)
C16	0.014 (2)	0.032 (3)	0.035 (4)	−0.001 (2)	0.003 (2)	0.003 (3)
C17	0.023 (3)	0.024 (3)	0.029 (4)	−0.003 (2)	0.007 (3)	0.005 (3)

Geometric parameters (Å, º)

V1—N1	2.064 (4)	C3—H3C	0.9800
V1—N4	1.888 (4)	C4—C5	1.489 (5)
V1—O1	1.988 (4)	C5—C6	1.3900
V1—O3	1.989 (3)	C5—C10	1.3900

V1—O4	2.207 (4)	C6—C7	1.3900
V1—O5	1.593 (4)	C6—H6	0.9500
Cl1—C8	1.724 (2)	C7—C8	1.3900
Cl2—C15	1.726 (3)	C7—H7A	0.9500
O1—C1	1.293 (6)	C8—C9	1.3900
O2—C1	1.231 (6)	C9—C10	1.3900
O3—C4	1.305 (5)	C9—H9	0.9500
O4—C11	1.247 (6)	C10—H10	0.9500
N1—C2	1.284 (7)	C11—C12	1.476 (6)
N1—N2	1.381 (5)	C12—C13	1.3900
N2—C4	1.308 (7)	C12—C17	1.3900
N3—N4	1.344 (6)	C13—C14	1.3900
N3—C11	1.345 (6)	C13—H13	0.9500
N3—H3	0.8800	C14—C15	1.3900
N4—H4	0.8800	C14—H14	0.9500
C1—C2	1.518 (7)	C15—C16	1.3900
C2—C3	1.475 (7)	C16—C17	1.3900
C3—H3A	0.9800	C16—H16	0.9500
C3—H3B	0.9800	C17—H17	0.9500

O5—V1—N4	93.89 (19)	O3—C4—C5	117.5 (4)
O5—V1—O1	97.17 (18)	N2—C4—C5	118.3 (4)
N4—V1—O1	98.17 (16)	C6—C5—C10	120.0
O5—V1—O3	97.41 (17)	C6—C5—C4	119.7 (3)
N4—V1—O3	106.77 (17)	C10—C5—C4	120.1 (3)
O1—V1—O3	150.05 (14)	C5—C6—C7	120.0
O5—V1—N1	109.67 (19)	C5—C6—H6	120.0
N4—V1—N1	156.21 (18)	C7—C6—H6	120.0
O1—V1—N1	76.15 (15)	C8—C7—C6	120.0
O3—V1—N1	74.34 (15)	C8—C7—H7A	120.0
O5—V1—O4	166.80 (18)	C6—C7—H7A	120.0
N4—V1—O4	73.27 (16)	C9—C8—C7	120.0
O1—V1—O4	87.83 (14)	C9—C8—Cl1	120.02 (18)
O3—V1—O4	83.82 (14)	C7—C8—Cl1	119.94 (18)
N1—V1—O4	83.36 (15)	C10—C9—C8	120.0
C1—O1—V1	119.1 (3)	C10—C9—H9	120.0
C4—O3—V1	115.6 (3)	C8—C9—H9	120.0
C11—O4—V1	113.3 (3)	C9—C10—C5	120.0
C2—N1—N2	121.6 (4)	C9—C10—H10	120.0
C2—N1—V1	119.4 (3)	C5—C10—H10	120.0
N2—N1—V1	119.0 (3)	O4—C11—N3	116.7 (5)
C4—N2—N1	106.6 (4)	O4—C11—C12	123.9 (4)
N4—N3—C11	114.1 (4)	N3—C11—C12	119.4 (4)
N4—N3—H3	123.0	C13—C12—C17	120.0
C11—N3—H3	123.0	C13—C12—C11	117.9 (3)
N3—N4—V1	122.6 (3)	C17—C12—C11	122.0 (3)
N3—N4—H4	118.7	C12—C13—C14	120.0
V1—N4—H4	118.7	C12—C13—H13	120.0

O2—C1—O1	125.1 (5)	C14—C13—H13	120.0
O2—C1—C2	120.8 (5)	C13—C14—C15	120.0
O1—C1—C2	114.1 (5)	C13—C14—H14	120.0
N1—C2—C3	127.3 (5)	C15—C14—H14	120.0
N1—C2—C1	110.3 (4)	C16—C15—C14	120.0
C3—C2—C1	122.4 (5)	C16—C15—Cl2	119.67 (19)
C2—C3—H3A	109.5	C14—C15—Cl2	120.29 (19)
C2—C3—H3B	109.5	C15—C16—C17	120.0
H3A—C3—H3B	109.5	C15—C16—H16	120.0
C2—C3—H3C	109.5	C17—C16—H16	120.0
H3A—C3—H3C	109.5	C16—C17—C12	120.0
H3B—C3—H3C	109.5	C16—C17—H17	120.0
O3—C4—N2	124.2 (5)	C12—C17—H17	120.0

O5—V1—O1—C1	−101.5 (4)	O1—C1—C2—N1	9.8 (6)
N4—V1—O1—C1	163.5 (4)	O2—C1—C2—C3	12.0 (8)
O3—V1—O1—C1	17.1 (6)	O1—C1—C2—C3	−169.0 (5)
N1—V1—O1—C1	7.1 (4)	V1—O3—C4—N2	6.8 (7)
O4—V1—O1—C1	90.8 (4)	V1—O3—C4—C5	−170.4 (3)
O5—V1—O3—C4	104.1 (4)	N1—N2—C4—O3	−4.5 (7)
N4—V1—O3—C4	−159.6 (4)	N1—N2—C4—C5	172.6 (4)
O1—V1—O3—C4	−14.5 (5)	O3—C4—C5—C6	−170.8 (4)
N1—V1—O3—C4	−4.3 (4)	N2—C4—C5—C6	11.9 (6)
O4—V1—O3—C4	−89.1 (4)	O3—C4—C5—C10	15.1 (6)
O5—V1—O4—C11	−14.2 (9)	N2—C4—C5—C10	−162.2 (4)
N4—V1—O4—C11	−0.7 (3)	C10—C5—C6—C7	0.0
O1—V1—O4—C11	98.5 (3)	C4—C5—C6—C7	−174.2 (4)
O3—V1—O4—C11	−110.3 (3)	C5—C6—C7—C8	0.0
N1—V1—O4—C11	174.8 (4)	C6—C7—C8—C9	0.0
O5—V1—N1—C2	91.9 (4)	C6—C7—C8—Cl1	177.7 (3)
N4—V1—N1—C2	−79.5 (6)	C7—C8—C9—C10	0.0
O1—V1—N1—C2	−0.8 (4)	Cl1—C8—C9—C10	−177.7 (3)
O3—V1—N1—C2	−175.6 (4)	C8—C9—C10—C5	0.0
O4—V1—N1—C2	−90.2 (4)	C6—C5—C10—C9	0.0
O5—V1—N1—N2	−90.2 (4)	C4—C5—C10—C9	174.1 (4)
N4—V1—N1—N2	98.4 (5)	V1—O4—C11—N3	−1.1 (6)
O1—V1—N1—N2	177.0 (4)	V1—O4—C11—C12	176.7 (3)
O3—V1—N1—N2	2.2 (3)	N4—N3—C11—O4	3.1 (7)
O4—V1—N1—N2	87.6 (4)	N4—N3—C11—C12	−174.9 (4)
C2—N1—N2—C4	178.1 (5)	O4—C11—C12—C13	2.1 (6)
V1—N1—N2—C4	0.3 (5)	N3—C11—C12—C13	179.9 (3)
C11—N3—N4—V1	−4.0 (6)	O4—C11—C12—C17	−174.7 (4)
O5—V1—N4—N3	179.5 (4)	N3—C11—C12—C17	3.1 (5)
O1—V1—N4—N3	−82.7 (4)	C17—C12—C13—C14	0.0
O3—V1—N4—N3	80.5 (4)	C11—C12—C13—C14	−176.9 (3)
N1—V1—N4—N3	−8.6 (7)	C12—C13—C14—C15	0.0
O4—V1—N4—N3	2.5 (4)	C13—C14—C15—C16	0.0
V1—O1—C1—O2	167.7 (4)	C13—C14—C15—Cl2	177.6 (3)

V1—O1—C1—C2	−11.3 (6)	C14—C15—C16—C17	0.0
N2—N1—C2—C3	−3.4 (8)	Cl2—C15—C16—C17	−177.6 (3)
V1—N1—C2—C3	174.5 (4)	C15—C16—C17—C12	0.0
N2—N1—C2—C1	177.9 (4)	C13—C12—C17—C16	0.0
V1—N1—C2—C1	−4.3 (6)	C11—C12—C17—C16	176.8 (3)
O2—C1—C2—N1	−169.2 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O2^{i}	0.88	1.92	2.744 (5)	156
N4—H4···O1^{i}	0.88	2.14	2.840 (5)	136

Symmetry code: (i) −*x*+3/2, *y*+1/2, −*z*+1/2.