metal-organic compounds

(4-Bromo-2-{[2-(morpholin-4-yl)ethyl-imino]methyl}phenolato)dioxido-vanadium(V)

Chen-Yi Wang,* Xiang Wu, Feng Cao and Cai-Jun Yuan

Department of Chemistry, Huzhou University, Huzhou 313000, People's Republic of China

Correspondence e-mail: chenyi_wang@163.com

In the title mononuclear dioxidovanadium(V) complex, [V(C$_{13}$H$_{16}$BrN$_2$O$_2$)O$_2$], the V^V atom is five-coordinated by one phenolate O, one imine N and one morpholine N atom of the Schiff base ligand, and by two oxide O atoms, forming a distorted square-pyramidal geometry. In the crystal, weak C—H···O interactions and a short Br···Br contact [3.4597 (12) Å] are observed.

Related literature

For related Schiff base complexes that we have reported recently, see: Wang (2009, 2011); Wang & Ye (2011). For similar oxidovanadium(V) complexes, see: Xie *et al.* (2004); Gao *et al.* (2005); Hartung *et al.* (2007); Romanowski *et al.* (2009).
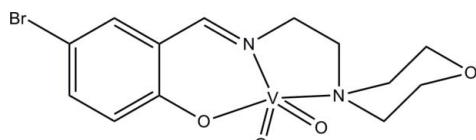
Experimental

Crystal data

[V(C$_{13}$H$_{16}$BrN$_2$O$_2$)O$_2$]	V = 1468.0 (5) Å^3
M_r = 395.13	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 21.372 (3) Å	μ = 3.41 mm^{-1}
b = 6.0892 (15) Å	T = 298 K
c = 11.372 (3) Å	0.17 × 0.13 × 0.13 mm
β = 97.248 (2)°	

Data collection

Bruker SMART CCD area-detector diffractometer	11267 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3204 independent reflections
T_{min} = 0.595, T_{max} = 0.665	2058 reflections with $I > 2\sigma(I)$
	R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044	190 parameters
$wR(F^2)$ = 0.096	H-atom parameters constrained
S = 1.02	$\Delta\rho_{max}$ = 0.61 e Å^{-3}
3204 reflections	$\Delta\rho_{min}$ = −0.62 e Å^{-3}

Table 1
Selected bond lengths (Å).

V1—O4	1.611 (2)	V1—N1	2.142 (3)
V1—O3	1.622 (3)	V1—N2	2.159 (3)
V1—O1	1.907 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1^i	0.93	2.53	3.241 (4)	133
C11—H11A···O2^{ii}	0.97	2.57	3.479 (5)	156

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); 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); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2767).

References

Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Gao, S., Huo, L.-H., Deng, Z.-P. & Zhao, H. (2005). *Acta Cryst.* E**61**, m978–m980.

Hartung, J., Ludwig, A., Svoboda, I. & Fuess, H. (2007). *Acta Cryst.* E**63**, m1422–m1423.

Romanowski, G., Wera, M. & Sikorski, A. (2009). *Acta Cryst.* E**65**, m190.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Wang, C.-Y. (2009). *J. Coord. Chem.* **62**, 2860–2868.

Wang, C.-Y. (2011). *Acta Cryst.* E**67**, m1085–m1086.

Wang, C.-Y. & Ye, J.-Y. (2011). *Russ. J. Coord. Chem.* **37**, 235–241.

Xie, M.-J., Ping, Y.-S., Zheng, L.-D., Hui, J.-Z. & Peng, C. (2004). *Acta Cryst.* E**60**, m1382–m1383.

supplementary materials

(4-Bromo-2-{[2-(morpholin-4-yl)ethylimino]methyl}phenolato)dioxidovanadium(V)

C.-Y. Wang, X. Wu, F. Cao and C.-J. Yuan

Comment

As part of our investigations into new Schiff base complexes (Wang & Ye, 2011; Wang, 2009, 2011), we have synthesized the title compound, a new mononuclear dioxovanadium(V) complex (Fig. 1). The V^V atom in the complex is five-coordinated by one phenolate O, one imine N and one morpholine N atom of the Schiff base ligand, and by two oxo O atoms, forming a distorted square pyramidal coordination. The V–O and V–N bond lengths (Table 1) are typical and are comparable with those observed in other similar oxovanadium(V) complexes with Schiff bases (Xie *et al.*, 2004; Gao *et al.*, 2005; Hartung *et al.*, 2007; Romanowski *et al.*, 2009).

Experimental

5-Bromosalicylaldehyde (1.0 mmol, 0.201 g), 2-morpholin-4-ylethylamine (1.0 mmol, 0.130 g) and VO(acac)$_2$ (1.0 mmol, 0.265 g) were dissolved in MeOH (30 ml). The mixture was stirred at room temperature for 10 min to give a yellow solution. After keeping the solution in air for a week, yellow block-shaped crystals were formed at the bottom of the vessel.

Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with $U_{iso}(H)$ set at $1.2U_{eq}(C)$.

Figures
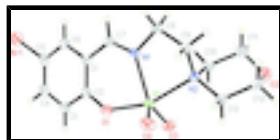
Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

(4-Bromo-2-{[2-(morpholin-4-yl)ethylimino]methyl}phenolato)dioxidovanadium(V)

Crystal data

[V(C$_{13}$H$_{16}$BrN$_2$O$_2$)O$_2$]	$F(000) = 792$
$M_r = 395.13$	$D_x = 1.788$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2133 reflections
$a = 21.372$ (3) Å	$\theta = 2.7$–$25.1°$
$b = 6.0892$ (15) Å	$\mu = 3.41$ mm^{-1}
$c = 11.372$ (3) Å	$T = 298$ K

supplementary materials

$\beta = 97.248\ (2)°$

$V = 1468.0\ (5)\ \text{Å}^3$

$Z = 4$

Block, yellow

$0.17 \times 0.13 \times 0.13$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	3204 independent reflections
Radiation source: fine-focus sealed tube	2058 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.051$
ω scans	$\theta_{max} = 27.0°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -27 \rightarrow 27$
$T_{min} = 0.595$, $T_{max} = 0.665$	$k = -7 \rightarrow 7$
11267 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
3204 reflections	$(\Delta/\sigma)_{max} < 0.001$
190 parameters	$\Delta\rho_{max} = 0.61$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.62$ e Å^{-3}

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	0.28158 (3)	0.31216 (10)	0.45502 (5)	0.02930 (17)
Br1	0.01304 (2)	−0.30291 (9)	0.10991 (4)	0.06124 (19)
N1	0.23304 (13)	0.4015 (4)	0.2854 (2)	0.0255 (6)

N2	0.34664 (13)	0.5238 (4)	0.3783 (2)	0.0253 (7)
O1	0.21270 (13)	0.1134 (5)	0.4562 (2)	0.0482 (7)
O2	0.47871 (12)	0.5987 (5)	0.3519 (3)	0.0527 (8)
O3	0.33864 (12)	0.1419 (4)	0.4953 (2)	0.0442 (7)
O4	0.27296 (13)	0.4752 (4)	0.5634 (2)	0.0473 (7)
C1	0.15474 (16)	0.1138 (6)	0.2635 (3)	0.0290 (8)
C2	0.17008 (17)	0.0235 (6)	0.3773 (3)	0.0329 (9)
C3	0.13930 (18)	−0.1665 (6)	0.4067 (3)	0.0407 (10)
H3	0.1501	−0.2298	0.4809	0.049*
C4	0.09347 (19)	−0.2617 (6)	0.3282 (4)	0.0431 (10)
H4	0.0730	−0.3876	0.3496	0.052*
C5	0.07759 (18)	−0.1697 (7)	0.2164 (3)	0.0401 (10)
C6	0.10726 (17)	0.0147 (6)	0.1835 (3)	0.0372 (9)
H6	0.0962	0.0748	0.1085	0.045*
C7	0.18586 (17)	0.3060 (6)	0.2271 (3)	0.0307 (8)
H7	0.1702	0.3667	0.1542	0.037*
C8	0.25689 (16)	0.5992 (6)	0.2292 (3)	0.0301 (8)
H8A	0.2228	0.7031	0.2088	0.036*
H8B	0.2741	0.5587	0.1573	0.036*
C9	0.30743 (16)	0.7010 (5)	0.3164 (3)	0.0304 (8)
H9A	0.3337	0.7968	0.2751	0.036*
H9B	0.2883	0.7882	0.3737	0.036*
C10	0.38229 (17)	0.3950 (6)	0.2964 (3)	0.0351 (9)
H10A	0.3534	0.3487	0.2283	0.042*
H10B	0.3994	0.2640	0.3371	0.042*
C11	0.43528 (19)	0.5240 (7)	0.2543 (3)	0.0494 (12)
H11A	0.4573	0.4323	0.2031	0.059*
H11B	0.4181	0.6492	0.2084	0.059*
C12	0.44707 (19)	0.7346 (7)	0.4274 (4)	0.0459 (11)
H12A	0.4307	0.8634	0.3837	0.055*
H12B	0.4770	0.7837	0.4934	0.055*
C13	0.39376 (17)	0.6159 (6)	0.4741 (3)	0.0362 (9)
H13A	0.4109	0.4971	0.5252	0.043*
H13B	0.3725	0.7166	0.5219	0.043*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.0394 (4)	0.0279 (4)	0.0198 (3)	−0.0050 (3)	0.0006 (3)	0.0003 (3)
Br1	0.0583 (3)	0.0709 (4)	0.0544 (3)	−0.0337 (3)	0.0067 (2)	−0.0207 (2)
N1	0.0354 (17)	0.0205 (16)	0.0202 (15)	−0.0021 (13)	0.0015 (13)	0.0013 (12)
N2	0.0338 (17)	0.0198 (16)	0.0215 (14)	−0.0002 (13)	0.0006 (12)	−0.0009 (12)
O1	0.0541 (18)	0.0570 (19)	0.0305 (15)	−0.0253 (15)	−0.0059 (13)	0.0126 (14)
O2	0.0331 (16)	0.068 (2)	0.0572 (18)	−0.0001 (15)	0.0048 (14)	0.0167 (17)
O3	0.0503 (17)	0.0344 (16)	0.0459 (16)	0.0026 (13)	−0.0017 (13)	0.0139 (13)
O4	0.0660 (19)	0.0461 (18)	0.0318 (14)	−0.0093 (15)	0.0135 (13)	−0.0113 (13)
C1	0.034 (2)	0.025 (2)	0.029 (2)	−0.0045 (16)	0.0059 (16)	−0.0027 (16)
C2	0.033 (2)	0.034 (2)	0.033 (2)	−0.0049 (18)	0.0085 (17)	−0.0014 (17)

C3	0.045 (2)	0.045 (3)	0.033 (2)	−0.011 (2)	0.0064 (18)	0.0057 (19)
C4	0.048 (3)	0.033 (2)	0.051 (3)	−0.0123 (19)	0.018 (2)	−0.003 (2)
C5	0.037 (2)	0.045 (3)	0.039 (2)	−0.0175 (19)	0.0094 (18)	−0.019 (2)
C6	0.039 (2)	0.041 (3)	0.031 (2)	−0.0065 (19)	0.0039 (17)	−0.0050 (18)
C7	0.037 (2)	0.037 (2)	0.0181 (17)	0.0017 (18)	0.0026 (15)	−0.0014 (17)
C8	0.037 (2)	0.024 (2)	0.0287 (19)	0.0028 (16)	0.0010 (16)	0.0062 (16)
C9	0.033 (2)	0.022 (2)	0.035 (2)	0.0019 (16)	0.0016 (16)	−0.0003 (17)
C10	0.045 (2)	0.030 (2)	0.030 (2)	0.0120 (18)	0.0030 (17)	0.0002 (17)
C11	0.047 (3)	0.065 (3)	0.039 (2)	0.011 (2)	0.017 (2)	0.010 (2)
C12	0.041 (2)	0.043 (3)	0.051 (3)	−0.008 (2)	−0.008 (2)	0.007 (2)
C13	0.041 (2)	0.038 (2)	0.027 (2)	−0.0040 (19)	−0.0072 (17)	−0.0010 (17)

Geometric parameters (Å, °)

V1—O4	1.611 (2)	C4—C5	1.392 (5)
V1—O3	1.622 (3)	C4—H4	0.9300
V1—O1	1.907 (3)	C5—C6	1.365 (5)
V1—N1	2.142 (3)	C6—H6	0.9300
V1—N2	2.159 (3)	C7—H7	0.9300
Br1—C5	1.899 (4)	C8—C9	1.504 (5)
N1—C7	1.275 (4)	C8—H8A	0.9700
N1—C8	1.483 (4)	C8—H8B	0.9700
N2—C9	1.487 (4)	C9—H9A	0.9700
N2—C13	1.497 (4)	C9—H9B	0.9700
N2—C10	1.497 (4)	C10—C11	1.505 (5)
O1—C2	1.314 (4)	C10—H10A	0.9700
O2—C12	1.423 (5)	C10—H10B	0.9700
O2—C11	1.428 (4)	C11—H11A	0.9700
C1—C2	1.406 (5)	C11—H11B	0.9700
C1—C6	1.410 (5)	C12—C13	1.502 (5)
C1—C7	1.433 (5)	C12—H12A	0.9700
C2—C3	1.392 (5)	C12—H12B	0.9700
C3—C4	1.368 (5)	C13—H13A	0.9700
C3—H3	0.9300	C13—H13B	0.9700
O4—V1—O3	109.41 (14)	N1—C7—C1	126.0 (3)
O4—V1—O1	102.89 (13)	N1—C7—H7	117.0
O3—V1—O1	98.38 (13)	C1—C7—H7	117.0
O4—V1—N1	116.30 (13)	N1—C8—C9	107.9 (3)
O3—V1—N1	132.70 (12)	N1—C8—H8A	110.1
O1—V1—N1	83.17 (11)	C9—C8—H8A	110.1
O4—V1—N2	94.75 (12)	N1—C8—H8B	110.1
O3—V1—N2	89.77 (12)	C9—C8—H8B	110.1
O1—V1—N2	156.74 (10)	H8A—C8—H8B	108.4
N1—V1—N2	75.39 (10)	N2—C9—C8	109.1 (3)
C7—N1—C8	116.0 (3)	N2—C9—H9A	109.9
C7—N1—V1	127.9 (2)	C8—C9—H9A	109.9
C8—N1—V1	116.1 (2)	N2—C9—H9B	109.9
C9—N2—C13	111.1 (3)	C8—C9—H9B	109.9
C9—N2—C10	112.7 (3)	H9A—C9—H9B	108.3

C13—N2—C10	107.5 (3)	N2—C10—C11	112.7 (3)
C9—N2—V1	105.7 (2)	N2—C10—H10A	109.0
C13—N2—V1	109.74 (19)	C11—C10—H10A	109.0
C10—N2—V1	110.0 (2)	N2—C10—H10B	109.0
C2—O1—V1	136.7 (2)	C11—C10—H10B	109.0
C12—O2—C11	110.0 (3)	H10A—C10—H10B	107.8
C2—C1—C6	119.5 (3)	O2—C11—C10	111.1 (3)
C2—C1—C7	121.5 (3)	O2—C11—H11A	109.4
C6—C1—C7	119.0 (3)	C10—C11—H11A	109.4
O1—C2—C3	119.3 (3)	O2—C11—H11B	109.4
O1—C2—C1	121.8 (3)	C10—C11—H11B	109.4
C3—C2—C1	118.9 (3)	H11A—C11—H11B	108.0
C4—C3—C2	121.1 (4)	O2—C12—C13	111.9 (3)
C4—C3—H3	119.4	O2—C12—H12A	109.2
C2—C3—H3	119.4	C13—C12—H12A	109.2
C3—C4—C5	119.8 (4)	O2—C12—H12B	109.2
C3—C4—H4	120.1	C13—C12—H12B	109.2
C5—C4—H4	120.1	H12A—C12—H12B	107.9
C6—C5—C4	120.9 (4)	N2—C13—C12	113.1 (3)
C6—C5—Br1	120.1 (3)	N2—C13—H13A	109.0
C4—C5—Br1	119.0 (3)	C12—C13—H13A	109.0
C5—C6—C1	119.7 (4)	N2—C13—H13B	109.0
C5—C6—H6	120.1	C12—C13—H13B	109.0
C1—C6—H6	120.1	H13A—C13—H13B	107.8

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1[i]	0.93	2.53	3.241 (4)	133
C11—H11A···O2[ii]	0.97	2.57	3.479 (5)	156

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

Fig. 1