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(1-Formyl-2-naphtholato-κ^2O,O')dioxo(1,10-phenanthroline-κ^2N,N')vanadium(V) dichloromethane 0.25-solvate

Jian-Fang Dong, Lian-Zhi Li,* Hui Cui, Da-Qi Wang and Tao Xu

School of Chemistry and Chemical Engineering,
Liaocheng University, Shandong 252059,
People's Republic of China

Correspondence e-mail:
lilianzhi1963@yahoo.com.cn

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In the molecule of the title compound, $[V(C_{11}H_7O_2)O_2(C_{12}H_8N_2)]\cdot 0.25CH_2Cl_2$, the central V^V atom has an octahedral environment, with vanadyl and deprotonated phenol O atoms and two phenanthroline N atoms in the equatorial plane and vanadyl and aldehyde carbonyl O atoms in axial positions. In the crystal structure, intermolecular C—H···O hydrogen bonds and π–π stacking interactions result in the formation of a two-dimensional network.

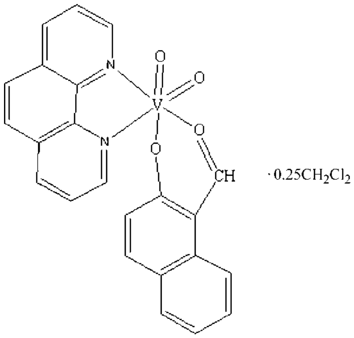
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.012 Å
Disorder in solvent or counterion
R factor = 0.093
wR factor = 0.302
Data-to-parameter ratio = 12.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The significant role of vanadium, in a variety of chemical and biological systems, has stimulated the development of vanadium chemistry (Hirao, 1997). However, compared with other transition metal complexes, fewer vanadium complexes have been synthesized and characterized. It is necessary to synthesize a wide variety of vanadium complexes for an understanding of their functions and applications. Vanadium in oxidation states III, IV, and V readily forms V—O bonds and also easily binds N and S. We report here the synthesis and crystal structure of the title complex, (I).



(I)

In the molecule of (I), (Fig. 1), the ligand bond lengths and angles (Table 1) are generally within normal ranges (Allen *et al.*, 1987). The oxidation state is V^V, as a result of the oxidation of V^{IV} in the presence of air (Li *et al.*, 2004). The title complex has two vanadyl O atoms with two V═O double bonds of V1═O4 [1.608 (5) Å] and V1═O3 [1.627 (6) Å], and owing to their strong repulsion the O3═V1═O4 [106.5 (3)°] angle is increased from the standard octahedral value, being close to the corresponding angles in $[V^VO_2(HSHED)]_2$ (HSHED is *N*-salicylidene-*N'*-(2-hydroxyethyl)ethylenediamine; Li *et al.*, 1988) and VO_2EDTA (Scheidt *et al.*, 1971). The central V^V atom has a distorted octahedral environment, in which the O4 vanadyl and O1 aldehyde carbonyl atoms occupy the axial positions, while the O3 vanadyl and O2 deprotonated phenol

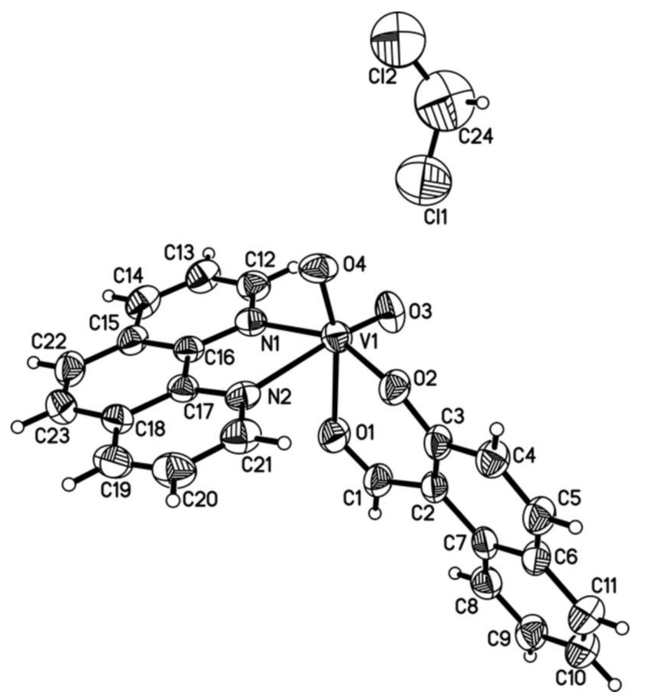


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

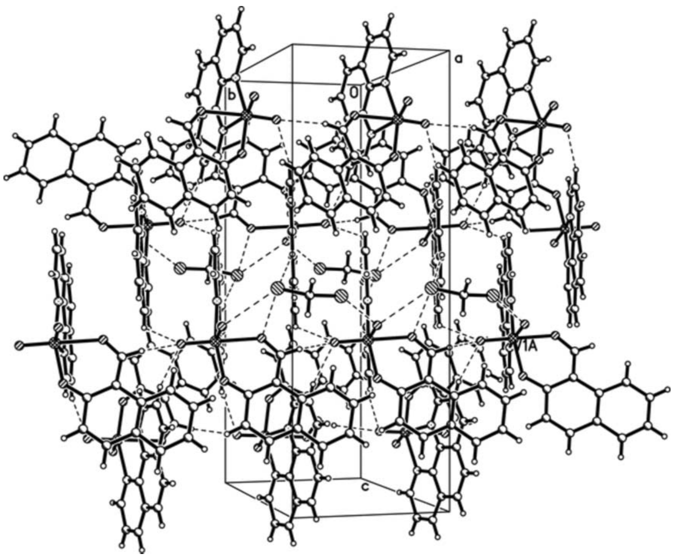


Figure 2
A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

atoms and N1 and N2 phenanthroline atoms form the equatorial plane.

The aldehyde ligand is oriented with respect to the equatorial plane at a dihedral angle of 77.92 (15)°. The chelation of the ligands leads to small O2—V1—O1 [82.9 (2)°] and N1—V1—N2 [73.4 (2)°] angles.

The 1,10-phenanthroline ligands related by a center of symmetry have a centroid–centroid separation of 3.693 (13) Å [the perpendicular distance is 3.554 (11) Å] for rings N1/C12–C16; the slip angle is 15.77 (24)°. These values indicate a significant π–π interaction (Tong *et al.*, 1999).

In the crystal structure, (Fig. 2), the intermolecular C—H···O hydrogen bonds (Table 2) and the π–π stacking interactions cause the formation of a two-dimensional network.

Experimental

2-Hydroxy-1-naphthaldehyde (172.19 mg, 1 mmol) and 1,10-phenanthroline (198.22 mg, 1 mmol) were dissolved in methanol (15 ml) and added dropwise to a solution of vanadyl sulfate hydrate (225.4 mg, 1mmol) in water (4 ml) with stirring. The mixture was refluxed for 4 h with stirring and then filtered. The precipitate was dissolved in dichloromethane (10 ml) and held at room temperature for 7 d, whereupon yellow crystals suitable for X-ray diffraction were obtained.

Crystal data

$[V(C_{11}H_7O_2)O_2(C_{12}H_8N_2)]\cdot 0.25CH_2Cl_2$
$M_r = 455.54$
Monoclinic, $P2_1/c$
$a = 13.860$ (2) Å
$b = 7.3946$ (14) Å
$c = 21.063$ (2) Å
$\beta = 90.105$ (2)°
$V = 2158.8$ (6) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.55$ mm^{-1}
$T = 298$ (2) K
0.23 × 0.18 × 0.15 mm

Data collection

Bruker CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.883$, $T_{\max} = 0.922$
10232 measured reflections
3770 independent reflections
1689 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.067$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.093$
$wR(F^2) = 0.302$
$S = 0.98$
3770 reflections
298 parameters
24 restraints
H-atom parameters constrained
$\Delta\rho_{\max} = 0.96$ e Å^{-3}
$\Delta\rho_{\min} = -0.72$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

V1—O1	2.182 (6)	V1—O4	1.608 (5)
V1—O2	1.896 (6)	V1—N1	2.153 (6)
V1—O3	1.627 (6)	V1—N2	2.287 (6)
O2—V1—O1	82.9 (2)	O3—V1—N1	92.8 (3)
O3—V1—O1	87.6 (3)	O4—V1—N1	93.3 (2)
O4—V1—O1	164.8 (3)	O1—V1—N2	75.5 (2)
N1—V1—O1	80.1 (2)	O2—V1—N2	85.8 (2)
O3—V1—O2	103.7 (3)	O3—V1—N2	159.5 (3)
O4—V1—O2	98.8 (2)	O4—V1—N2	89.6 (3)
O4—V1—O3	106.5 (3)	N1—V1—N2	73.4 (2)
O2—V1—N1	155.8 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O4$^{\rm i}$	0.93	2.41	3.199 (10)	143
C14—H14···O4$^{\rm ii}$	0.93	2.57	3.219 (11)	126
C20—H20···O4$^{\rm iii}$	0.93	2.32	3.237 (11)	166

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

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H atoms were positioned geometrically with C—H = 0.93 and 0.97 Å for aromatic and methylene H atoms, and constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) or 1.5$U_{\rm eq}$($C_{\rm methyl}$).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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