Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

{2-[2-(*N*,*N*′-Dimethylamino)ethyliminomethyl]-phenolato-κ^3N,N',O}dioxovanadium(V)

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Received 7 July 2004
Accepted 5 August 2004
Online 11 September 2004

Key indicators

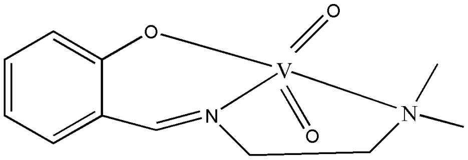
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.034
wR factor = 0.098
Data-to-parameter ratio = 13.7

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

Oxovanadium(V) complexes of *N*,*N*′-dimethylethylenediamine(salicylaldehyde) have been prepared and characterized. The structure of the title compound, [$VO_2(C_{11}H_{15}N_2O)$], reveals that the vanadium(V) centre is five-coordinate, with a distorted square-pyramidal environment.

Comment

The significant role of vanadium in a variety of chemical and biological systems has stimulated the development of vanadium chemistry (Hirao, 1997; Wever & Kustin, 1990; Van de Velde *et al.*, 2000). The interaction of simple vanadium species with ligands having pharmacological activity, particularly those with antitumour and insulin-mimetic properties, is of growing interest. A more detailed physicochemical characterization of vanadium compounds with pharmacologically interesting ligands will help in furthering the inorganic pharmacology of vanadium (Baran, 2000). In the meantime, Schiff base ligands show a wide range of biological activities. We report here a new five-coordinate vanadium(V) complex with the *N*,*N*′-dimethylethylenediamine(salicylaldehyde) ligand.



(I)

The title complex, (I), consists of a discrete monomeric molecule. The vanadium(V) ion is five-coordinate, in a distorted square-pyramidal environment. The basal square plane is composed of the *N*,*N*′-dimethylethylenediamine(salicylaldehyde) molecule, which acts as a tridentate ligand through its atoms N1 and N2 and the deprotonated phenol atom O1, and oxo ligand O3. The apical position is occupied by a second oxo ligand, O2. The V1—O2 and V1—O3 distances of 1.6097 (19) and 1.622 (2) Å are somewhat longer than the value of 1.585 (7) Å reported for VO(salen) (Bruins & Weaver, 1970; Mokry & Carrano, 1993) complexes but compare well with the values of 1.605 (2) and 1.647 (2) Å reported by Plass *et al.* (2000) and Ligtenbarg *et al.* (1999) for dioxovanadium complexes. The average V—O bond length and O—V—O angle are similar to those reported previously for the *cis*-VO_2 moiety in other complexes (Melchior *et al.*, 1999). The metal ion lies 0.476 (2) Å from the mean plane of the basal atoms, in the direction of the axial oxo ligand. The τ value for this Schiff base complex is 0.23, indicating a signifi-

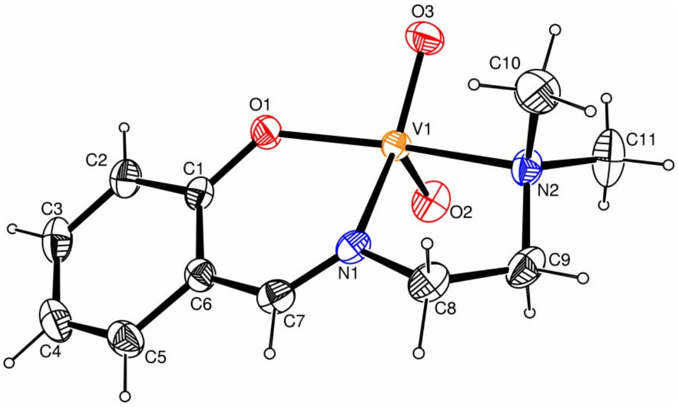


Figure 1
Molecular view of the title complex, with the atomic labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

cant distortion toward the trigonal bipyramidal form (Cornman *et al.*, 1997).

Experimental

A methanol solution (10 ml) of *N*,*N*′-dimethylethylenediamine (0.264 g, 3 mmol) was added to a methanol solution (10 ml) of salicylaldehyde (0.366 g, 3 mmol) and the solution was refluxed for 1 h. The ligand was not isolated and the mixture was added to an aqueous solution of NH_4VO_3 (0.35 g, 3 mmol) and Et_3N (0.301 g, 3 mmol). After refluxing for 2 h, the resulting yellow solution was collected. A few days later, deep-yellow crystals suitable for X-ray analysis were obtained.

Crystal data

$[V(C_{11}H_{15}N_2O)O_2]$
$M_r = 274.19$
Monoclinic, $P2_1/n$
$a = 6.3250$ (19) Å
$b = 15.945$ (5) Å
$c = 11.995$ (4) Å
$\beta = 90.645$ (5)°
$V = 1209.6$ (7) Å^3
$Z = 4$
$D_x = 1.506$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 946 reflections
$\theta = 3.1$–25.9°
$\mu = 0.82$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.22 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.813$, $T_{max} = 0.877$
6160 measured reflections
2140 independent reflections
1736 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 25.0°$
$h = -7 \rightarrow 7$
$k = -18 \rightarrow 18$
$l = -6 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.098$
$S = 1.05$
2140 reflections
156 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.3272P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

V1—O2	1.6097 (19)	V1—N1	2.138 (2)
V1—O3	1.622 (2)	V1—N2	2.188 (2)
V1—O1	1.9190 (18)		
O2—V1—O3	109.54 (11)	O1—V1—N1	83.72 (7)
O2—V1—O1	105.57 (9)	O2—V1—N2	94.70 (9)
O3—V1—O1	98.07 (9)	O3—V1—N2	88.87 (9)
O2—V1—N1	107.57 (9)	O1—V1—N2	154.78 (8)
O3—V1—N1	140.78 (10)	N1—V1—N2	75.84 (8)
O2—V1—O1—C1	70.1 (2)		

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.93–0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996) and *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 1998).

This work was financially supported by the National Science Foundation of China (No. 20171026) and Tianjin Natural Science Foundation (No. 013605811).

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Acta Cryst. (2004). E**60**, m1382–m1383 Xie Ming-Jin *et al.* • $[V(C_{11}H_{15}N_2O)O_2]$ **m1383**