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Methoxy[3-methoxysalicylaldehyde (4-methoxybenzoyl)hydrazonato(2−)-κ^3O,O',N]oxovanadium(V)

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Key indicators

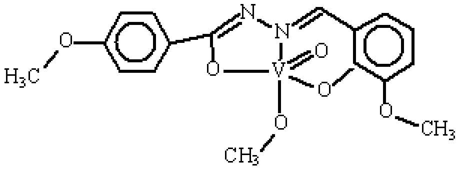
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.093
Data-to-parameter ratio = 16.6

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

The V^V atom of the title complex, [VOL(CH$_3$O)] [L is 3-methoxysalicylaldehyde (4-methoxybenzoyl)hydrazonate(2−), $C_{16}H_{14}N_2O_4$], has a square pyramidal O$_4$N configuration, with two O atoms and one N atom from the tridentate hydrazone ligand and one O atom from the methoxy group forming the basal plane. The oxo atom is located at the apical position. A layer structure is formed by π–π stacking interactions.

Comment

The study of the biological properties of vanadium complexes has been an interesting field in recent years (Butler & Walker, 1993). Most hydrazone ligands are coordinated to the metal centre through their O and N atoms, giving coordination environments similar to those of the biological system; therefore, it is important to study the VO^{3+} hydrazone complexes. We have previously reported the structures of some vanadium(V) hydrazone complexes with alkoxide, in which the hydrazone ligands are formed by condensing benzoylhydrazine with salicylaldehyde (Chen *et al.*, 1999; Gao *et al.*, 1998). In these complexes, the V^V atom shows a six-coordinate O$_5$N configuration. We report here the synthesis and structure of the title complex, VOL(OCH$_3$) [L is 3-methoxysalicylaldehyde (4-methoxybenzoyl)hydrazonate(2−), $C_{16}H_{14}N_2O_4$], (I), in which the V^V atom is in a five-coordinate environment.



(I)

As shown in Fig. 1, the V atom is coordinated by four O atoms and one N atom to form a square pyramidal O$_4$N configuration. The basal plane is defined by atoms O2, O3 and N1 of the fully deprotonated tridentate hydrazone ligand, and atom O4 of the methoxy group (r.m.s. deviation = 0.09 Å). Oxo atom O1 is located at the apical position. The V1—O1 bond distance is 1.619 (3) Å. The V atom deviates 0.471 (2) Å out of the basal plane towards the oxo O atom. The bond distances in the basal plane follow the order alkoxide oxygen < phenoxide oxygen < enolic oxygen < imine nitrogen. The five-membered chelate ring containing atoms O3, C9, N1, N2 and V1 is planar, with an r.m.s. deviation of 0.066 Å, while the six-membered chelate ring containing atoms O2, C1, C6, C7,

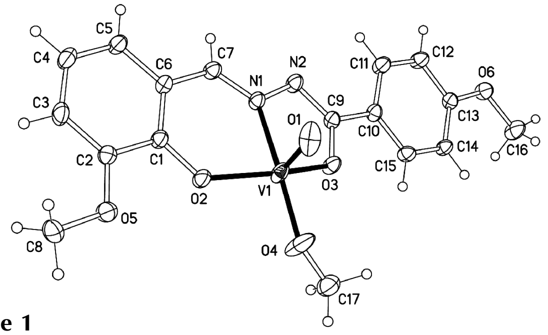


Figure 1
A view of the title compound, with 30% probability displacement ellipsoids.

N1 and V1 is not planar (r.m.s. deviation = 0.144 Å). There are π–π stacking interactions between adjacent benzene rings, with a separation of 3.713 Å. These π–π stacking interactions lead to the formation of a two-dimensional layer structure (Fig. 2).

Experimental

$VO(acac)_2$ (acac$^-$ is acetylacetonate) was synthesized according to the method described by Rowe & Jones (1957). The ligand *N*-(3-methoxysalicylaldehyde)-*N*′-(4-methoxybenzoyl)hydrazone was synthesized by condensing 3-methoxysalicylaldehyde with equimolar 4-methoxybenzoylhydrazine in ethanol. A methanol solution (15 ml) of $VO(acac)_2$ (0.26 g, 1 mmol) was added dropwise to a methanol solution (15 ml) containing *N*-(3-methoxysalicylaldehyde)-*N*′-(4-methoxybenzoyl)hydrazone (0.30 g, 1 mmol). The resulting mixture was refluxed with stirring for 30 min, cooled slowly to room temperature and filtered. Deep-red crystals were obtained from the solution over a period of several days. Analysis calculated for $C_{17}H_{17}N_2O_6V$: C 51.53, H 4.32, N 7.07%; found: C 51.70, H 4.19, N 6.95%.

Crystal data

$[VO(C_{16}H_{14}N_2O_4)(CH_3O)]$
$M_r = 396.27$
Orthorhombic, $P2_12_12_1$
$a = 7.531$ (3) Å
$b = 11.787$ (4) Å
$c = 19.786$ (6) Å
$V = 1756.4$ (11) Å^3
$Z = 4$
$D_x = 1.499$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 15 303 reflections
$\theta = 3.4$–27.5°
$\mu = 0.60$ mm^{-1}
$T = 296$ (2) K
Prism, red
0.34 × 0.26 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.823$, $T_{\max} = 0.899$
15 391 measured reflections

3957 independent reflections
3658 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.045$
$\theta_{\max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -15 \rightarrow 12$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.093$
$S = 1.00$
3957 reflections
239 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.3631P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Absolute structure: Flack (1983), 1643 Friedel pairs
Flack parameter = 0.49 (2)

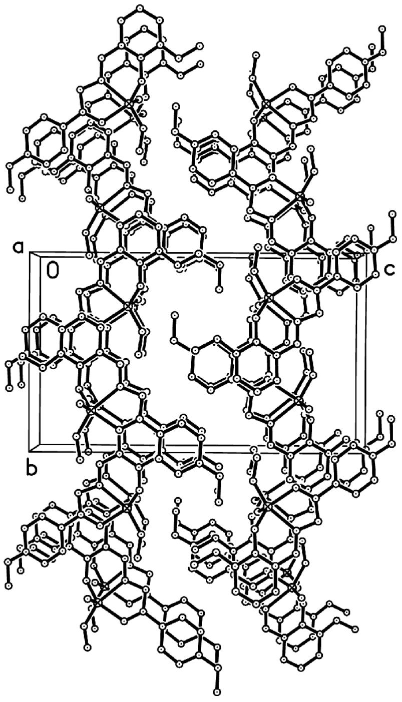


Figure 2
Packing diagram of the title complex, viewed along the *a* axis.

Table 1
Selected geometric parameters (Å, °).

V1—N1	2.100 (2)	V1—O4	1.747 (2)
V1—O1	1.616 (2)	N1—C7	1.291 (3)
V1—O2	1.848 (2)	N2—C9	1.302 (3)
V1—O3	1.931 (2)		
O1—V1—N1	96.2 (1)	O2—V1—O3	141.50 (8)
O1—V1—O2	107.76 (9)	O3—V1—N1	73.76 (6)
O1—V1—O3	105.42 (9)	O4—V1—N1	154.2 (1)
O1—V1—O4	106.6 (1)	O4—V1—O2	100.2 (1)
O2—V1—N1	83.71 (7)	O4—V1—O3	88.40 (8)

H atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.96 Å (methyl), and U(H) = $1.2U_{\rm eq}(C_{\rm aromatic})$ or $1.5U_{\rm eq}(C_{\rm methyl})$, and included in the final cycles of refinement in a riding model. The crystal is a racemic twin. The ratio of the two components refined to 0.49 (2)/0.51 (2).

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku Corporation, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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