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(*N*-Benzoyl-*N*-phenylhydroxaminato-κ^2O,O')-(salicylaldehyde isonicotinoylhydrazonato-κ^3O,N,N')vanadium(IV) hexane hemisolvate

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Key indicators

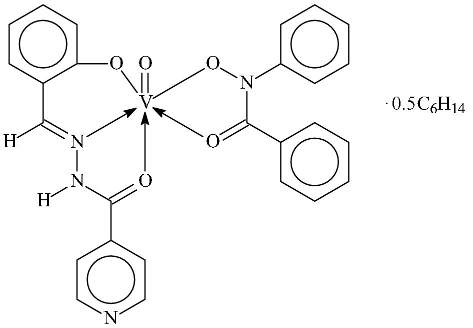
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.141
Data-to-parameter ratio = 17.3

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

The O atoms of the terdentate salicylaldehyde isonicotinoylhydrazone monoanion in the title compound, $[V(C_{13}H_{10}NO_2)(C_{13}H_{10}N_3O_2)O]\cdot0.5C_6H_{14}$, span two opposite sites of the octahedron surrounding the V^{IV} atom. Two other sites of the polyhedron are occupied by the O atoms of the chelating *N*-benzoyl-*N*-phenylhydroxamine monoanion; the sixth position is occupied by the oxo O atom. The hexane solvent molecule lies on an inversion center.

Comment

Salicylaldehydebenzoylhydrazone and *N*-phenylbenzoylhydroxamine are ligands that have been extensively used to bind to metal atoms. For vanadium, in particular, the terdentate ligand is structurally characterized in the V^VO_2 (Plass *et al.*, 2000), $V^V{}_2O_3$ (Sundheim & Mattes, 1993) and $V^VO(C_6H_5NHNH)$ (Sundheim *et al.*, 1994) complexes. The bidentate ligand is characterized in the oxovanadium derivatives of two other Schiff bases, salicyldehyde-L-alanine and acetylacetonebenzoylhydrazone.



(I)

The study of mixed-chelate vanadium complexes (Chen *et al.*, 1999) is extended to the present study, which describes an oxovanadium(IV) unit whose metal atom is chelated by salicylaldehyde isonicotinoylhydrazonate and *N*-benzoyl-*N*-phenylhydroxaminate ions in a six-coordinate geometry. The compound crystallizes as a hexane hemisolvate, (I) (Fig. 1). The salicylaldehyde isonicotinoylhydrazone monoanion, which spans two opposite sites of the octahedron surrounding the metal atom [O1—V1—O2 = 153.0 (1)°], is essentially planar. The metal is chelated by the *N*-benzoyl-*N*-phenylhydroxamine monoanion. The sixth coordination site is occupied by the oxo atom, which appears to exert a *trans* influence, as the opposite V—O bond [2.192 (2) Å] is much longer than the other V—O bonds. The hexane solvent molecule lies on an inversion center. There are no important intermolecular interactions in the crystal structure.

Experimental

A methanol solution (15 ml) of vanadyl(IV) bis(acetylacetonate) (0.26 g, 1 mmol) was added dropwise to a methanol solution (15 ml) of salicylaldehyde isonicotinoylhydrazone (0.24 g, 1 mmol). The mixture was refluxed with stirring for 1.5 h. A methanol solution (15 ml) of *N*-phenyl–*N*-benzoylhydroxylamine (0.22 g, 1 mmol) was added to the cool solution. A dark-red solid separated after 0.5 h. After filtration crystals were grown by diffusion of *n*-hexane into the filtrate over a period of several days.

Crystal data

$[V(C_{13}H_{10}NO_2)(C_{13}H_{10}N_3O_2)O]\cdot 0.5C_6H_{14}$
$M_r = 562.49$
Triclinic, $P\overline{1}$
$a = 10.071$ (2) Å
$b = 12.016$ (2) Å
$c = 12.966$ (3) Å
$\alpha = 102.92$ (3)°
$\beta = 98.52$ (3)°
$\gamma = 113.49$ (3)°
$V = 1352.3$ (5) Å^3
$Z = 2$
$D_x = 1.379$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10 306 reflections
$\theta = 3.3$–27.4°
$\mu = 0.41$ mm^{-1}
$T = 295$ (2) K
Prism, red
0.39 × 0.26 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.725$, $T_{\max} = 0.926$
12 805 measured reflections
6103 independent reflections
3780 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 27.5°$
$h = -11 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.141$
$S = 1.01$
6103 reflections
352 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.0547P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.44$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

V1—O1	1.858 (2)	V1—O4	1.863 (2)
V1—O2	1.947 (2)	V1—O5	1.582 (2)
V1—O3	2.192 (2)		
O1—V1—O2	153.0 (1)	O2—V1—N1	74.6 (1)
O1—V1—O3	84.4 (1)	O3—V1—O4	75.2 (1)
O1—V1—O4	104.6 (1)	O3—V1—O5	171.8 (1)
O1—V1—O5	98.5 (1)	O3—V1—N1	89.0 (1)
O1—V1—N1	83.4 (1)	O4—V1—O5	96.7 (1)
O2—V1—O3	79.8 (1)	O4—V1—N1	161.2 (1)
O2—V1—O4	92.4 (1)	O5—V1—N1	98.9 (1)
O2—V1—O5	100.2 (1)		

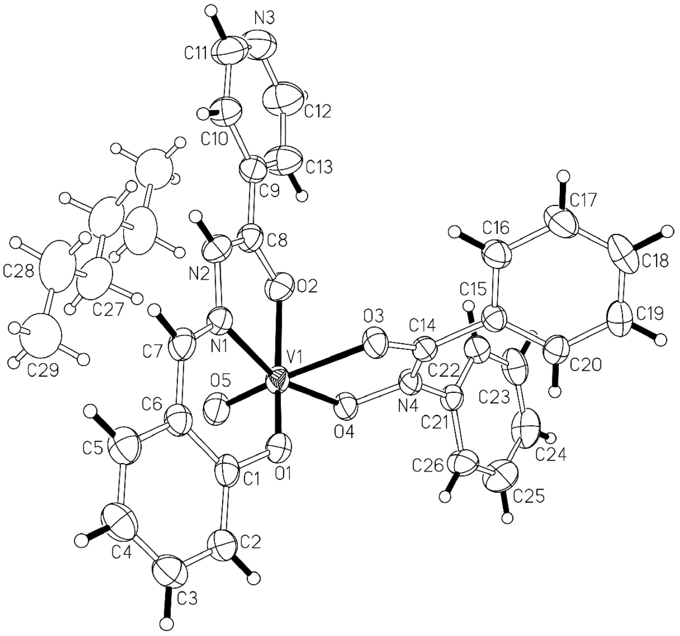


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii. Unlabeled atoms in the solvent molecule are related to the labeled atoms by $(1 - x, 2 - y, 1 - z)$.

The hexane molecule was refined with a C—C distance restraint of 1.54 (1) Å. The H atoms were placed in calculated positions [C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for aromatic, C—H = 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for methylene, C—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl, and N—H = 0.86 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm N})$ for the hydrazone H atom], and were included in the refinement in the riding-model approximation.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 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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