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Key indicators

Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
Disorder in main residue
R factor = 0.036
wR factor = 0.110
Data-to-parameter ratio = 12.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(Benzoylacetone 4-methoxybenzoylhydrazonato- $\kappa^3\text{O},\text{N},\text{O}'$)(*N*-benzoylhydroxylaminato- $\kappa^2\text{O},\text{O}'$)-oxovanadium(IV)**

The V atom in the title compound, $[\text{V}(\text{C}_{13}\text{H}_{10}\text{NO}_2)(\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_3)\text{O}]$, is *O,O'*-chelated by an *N*-benzoylhydroxylaminato ligand and *O,N,O'*-chelated by a benzoylacetone 4-methoxybenzoylhydrazonato ligand. The oxo O atom occupies the sixth coordination site of the octahedral coordination.

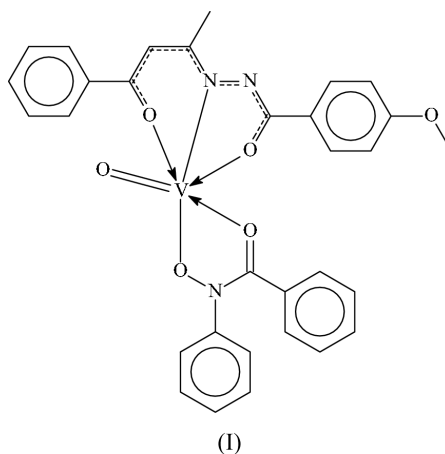
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Comment

As a continuation of studies on the hydrazone derivatives of vanadium, *viz.* vanadium(V) 4-methoxybenzoylhydrazonate (Huo *et al.*, 2004) and vanadium(IV) isonicotinoylhydrazonate (Gao *et al.*, 2004), the structure of (benzoylacetone 4-methoxybenzoylhydrazonato- $\kappa^3\text{O},\text{N},\text{O}'$)(*N*-benzoylhydroxylaminato- $\kappa^2\text{O},\text{O}'$)oxovanadium(IV), (I), has been determined.



The V atom in (I) (Fig. 1) is chelated by the terdentate and bidentate ligands, and the sixth coordination site of the octahedron is occupied by the oxo O atom. The oxo O atom is *trans* to the carbonyl O atom of the *N*-benzoyl-*N*-hydroxylaminato group; this O atom is farther from the V atom than the O atoms that are positioned *cis* to the oxo O atom.

Experimental

$\text{VO}(\text{acac})_2$ was synthesized according to a literature method (Rowe & Jones, 1957). Benzoylacetone 4-methoxybenzoylhydrazonate was synthesized by condensing benzoylacetone with an equimolar quantity of 4-methoxybenzoylhydrazine in ethanol. A methanol solution (15 ml) of $\text{VO}(\text{acac})_2$ (0.26 g, 1 mmol) was added to a methanol solution (15 ml) of benzoylacetone 4-methoxybenzoylhydrazonate (0.31 g, 1 mmol). The mixture was refluxed for 1 h and then cooled. A methanol solution (10 ml) of *N*-benzoyl-*N*-hydroxylamine (0.21 g, 1 mmol) was added to afford a deep-red solid. The product was collected and dissolved in dichloromethane. Crystals were isolated after several days by slow diffusion of hexane into the filtered di-

chloromethane solution. Elemental analysis calculated for $C_{31}H_{26}N_3O_6V$: C 63.38, H 4.46, N 7.15%; found: C 63.42, H 4.41, N 7.19%.

Crystal data

$[V(C_{13}H_{10}NO_2)(C_{18}H_{16}N_2O_3)O]$
 $M_r = 587.49$
 Monoclinic, $P2_1/c$
 $a = 10.247(2) \text{ \AA}$
 $b = 12.387(3) \text{ \AA}$
 $c = 21.858(2) \text{ \AA}$
 $\beta = 90.59(1)^\circ$
 $V = 2774.3(8) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.407 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 24313 reflections
 $\theta = 3.2\text{--}27.5^\circ$
 $\mu = 0.41 \text{ mm}^{-1}$
 $T = 295(2) \text{ K}$
 Block, red
 $0.36 \times 0.25 \times 0.19 \text{ mm}$

Data collection

Rigaku R-Axis RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{min} = 0.791, T_{max} = 0.927$
 26700 measured reflections

6307 independent reflections
 4958 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.019$
 $\theta_{max} = 27.5^\circ$
 $h = -12 \rightarrow 13$
 $k = -16 \rightarrow 16$
 $l = -28 \rightarrow 28$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.110$
 $S = 1.07$
 6307 reflections
 499 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 0.1309P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.26 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.23 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

V1—O1	1.590 (1)	V1—O5	2.203 (1)
V1—O2	1.880 (1)	V1—O6	1.879 (1)
V1—O3	1.920 (1)	V1—N1	2.050 (2)
O1—V1—O2	98.31 (6)	O2—V1—N1	84.80 (5)
O1—V1—O3	102.20 (6)	O3—V1—O5	80.04 (5)
O1—V1—O5	171.41 (6)	O3—V1—O6	87.61 (5)
O1—V1—O6	96.77 (6)	O3—V1—N1	75.40 (5)
O1—V1—N1	100.26 (6)	O5—V1—O6	74.95 (4)
O2—V1—O3	153.61 (6)	O5—V1—N1	88.32 (5)
O2—V1—O5	82.15 (5)	O6—V1—N1	158.07 (5)
O2—V1—O6	106.36 (5)		

The hydrazone anion is disordered with respect to the phenyl and anisyl groups; as the disorder refined to an almost 50:50 ratio, the occupancy of the disordered atoms was set at 0.5. The C4—C5 and C4—C5' distances were restrained to be within 0.01 \AA of each other, as were the C11—C12/C11—C12', C15—O4/C15'—O4' and C18—O4/C18'—O4' pairs of distances. The C—C distances in the aromatic rings of the disordered atoms were restrained to be 1.39 (1) \AA . It was not necessary to restrain the displacement parameters of the primed and unprimed atoms. The methyl H atoms were allowed to rotate to

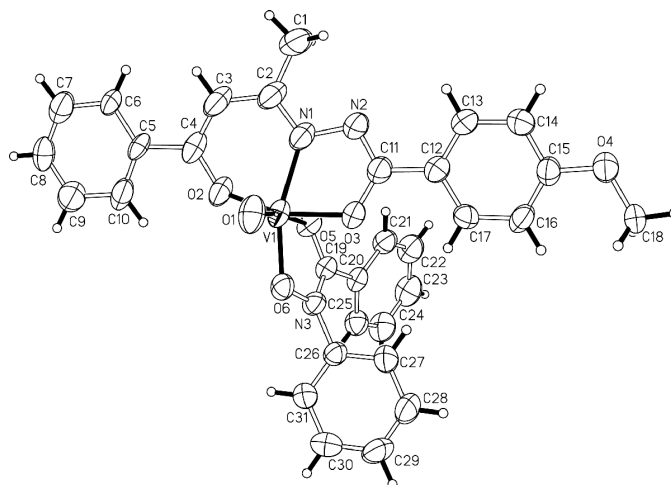


Figure 1

ORTEPII plot (Johnson, 1976) of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. Only one disorder component is shown.

fit the electron density [$C-H = 0.9 \text{ \AA}$ and $U_{iso}(H) = 1.5U_{eq}(C)$]; the other H atoms were positioned geometrically [$C-H = 0.93 \text{ \AA}$ $U_{iso}(H) = 1.2U_{eq}(C)$]. All H atoms were included in the refinement in the riding-model approximation.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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