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Key indicators

Single-crystal X-ray study
T = 295 K
Mean $\sigma(C-C)$ = 0.004 Å
R factor = 0.046
wR factor = 0.143
Data-to-parameter ratio = 16.4

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

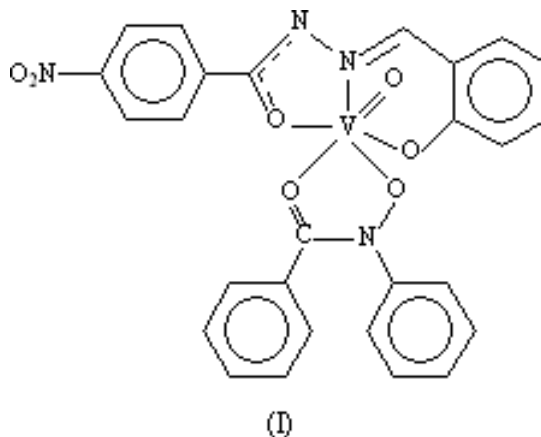
**(N-Hydroxy-N-phenylbenzamidato- κ^2O,O')-
oxo[salicylaldehyde (4-nitrobenzoyl)hydrazon-
ato- κ^3O,N,O']vanadium(V)**

The vanadium(V) atom in $[V(C_{14}H_9N_3O_4)(C_{13}H_{10}NO_2)O]$ is coordinated by an oxo O atom, the N and two O atoms of the tridentate hydrazonate, and the two O atoms of the bidentate hydroxamate in a distorted octahedral geometry.

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Comment

Bis(acetylacetonato)vanadium(IV) reacts with *N*-benzoyl-*N*-phenylhydroxylamine and benzoylacetone 4-methoxybenzoylhydrazone to form (benzoylacetone 4-methoxybenzoylhydrazonato)(*N*-benzoylhydroxylaminato)oxovanadium(IV) (Gao *et al.*, 2005). A similar reaction with salicylaldehyde(4-nitrobenzoyl)hydrazone, which has an acidic H atom in the form of a hydroxy group, furnished the corresponding compound, but the metal atom has been oxidized to the 5+ oxidation state, (I). The V^V atom is coordinated by an oxo O atom, one N and two O atoms of the tridentate hydrazonate, and the two O atoms of the bidentate hydroxamate in a distorted octahedral geometry.



Experimental

Salicylaldehyde(4-nitrophenoxycetyl)hydrazone was synthesized by condensing salicylaldehyde with an equimolar quantity of (4-nitrobenzoyl)hydrazine in ethanol. An ethanol solution (15 ml) of VO(acac)₂ (0.26 g, 2 mmol) was added to an ethanol solution (15 ml) of salicylaldehyde(4-nitrobenzoyl)hydrazone (0.56 g, 2 mmol). The mixture was heated for 1 h. The addition of an ethanol solution (15 ml) of *N*-benzoyl-*N*-phenylhydroxylamine (0.41 g, 2 mmol) gave a red solution. The mixture was filtered, and the dark red compound was collected and then dissolved in dichloromethane. Crystals were obtained by the diffusion of *n*-hexane into the filtrate over a period of several days. Analysis found: C 57.62, H 3.47, N 9.99%; calculated for C₂₇H₁₉N₄O₇V: C 57.66, H 3.41, N 9.96%.

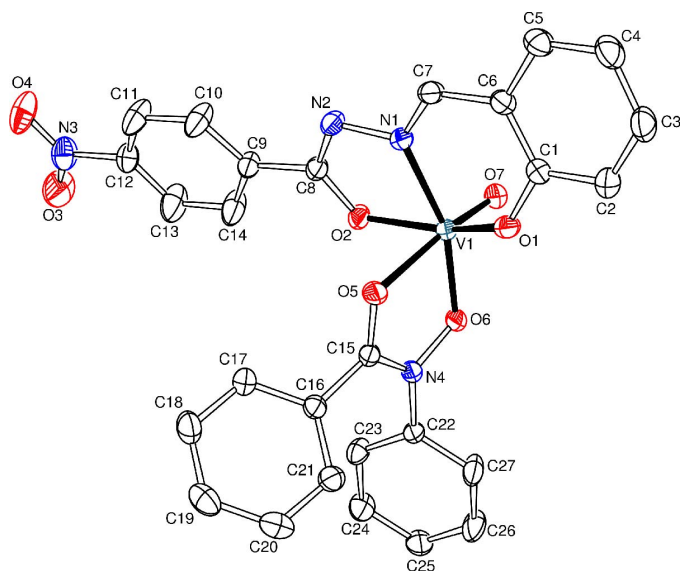


Figure 1
ORTEP (Burnett & Johnson, 1996) molecular view of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

Crystal data

$[\text{V}(\text{C}_{14}\text{H}_9\text{N}_3\text{O}_4)(\text{C}_{13}\text{HNO}_3)\text{O}]$

$M_r = 562.40$

Triclinic, $P\bar{1}$

$a = 10.263 (2) \text{ \AA}$

$b = 10.737 (2) \text{ \AA}$

$c = 12.623 (3) \text{ \AA}$

$\alpha = 86.48 (3)^\circ$

$\beta = 86.20 (3)^\circ$

$\gamma = 66.81 (3)^\circ$

$V = 1274.8 (5) \text{ \AA}^3$

$Z = 2$

$D_x = 1.465 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

Cell parameters from 11 507

reflections

$\theta = 3.2\text{--}27.5^\circ$

$\mu = 0.44 \text{ mm}^{-1}$

$T = 295 (2) \text{ K}$

Prism, red

$0.38 \times 0.26 \times 0.18 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID IP
diffractometer

ω scans

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.842$, $T_{\max} = 0.923$

12 599 measured reflections

5774 independent reflections

4630 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 27.5^\circ$

$h = -13 \rightarrow 12$

$k = -13 \rightarrow 13$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.143$

$S = 1.09$

5774 reflections

352 parameters

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0859P)^2 + 0.1841P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.65 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.24 \text{ e \AA}^{-3}$

Extinction correction: none

The C—C distances in aromatic rings were restrained to be equal with an effective standard deviation of 0.01, as the free refinement led to a wide spread of these distances. Some of the anisotropic displacement parameters have large components, suggesting the occurrence of disorder; however, any attempts to construct chemically reasonable models failed. H atoms were placed in calculated positions (C—H = 0.93 Å) and were allowed to ride on their parent C atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *RAPID-AUTO* (Rigaku Corporation, 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: *ORTEP* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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