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(Benzohydrazidato-κ^2N',O)[2-(benzoylhydrazono-κ^2N,O)propionato-κO]-oxidovanadium(V)

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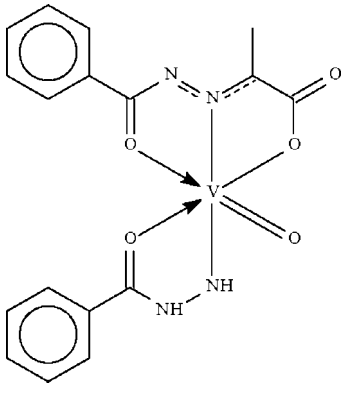
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.091; data-to-parameter ratio = 15.8.

The V^V atom in the title compound, $[VO(C_7H_7N_2O)(C_{10}H_8N_2O_3)]$, is N,O-chelated by the benzohydrazidate anion and O,N,O'-chelated by the 2-(benzoylhydrazono)propionate dianion. The distorted octahedral trans-N_2O_4 coordination geometry is completed by the vandadyl O atom. Molecules are linked by N—H···O hydrogen bonds into a supramolecular chain structure parallel to [010].

Related literature

For other benzoylhydrazido–oxovanadium compounds, see: Kopka & Mattes (1995); Sundheim *et al.* (1994).



Experimental

Crystal data

$[V(C_7H_7N_2O)(C_{10}H_8N_2O_3)O]$
M_r = 406.27
Monoclinic, $P2_1/n$
a = 10.9424 (2) Å
b = 6.2384 (1) Å
c = 25.7215 (5) Å
β = 96.603 (1)°
V = 1744.18 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 123 K
0.35 × 0.10 × 0.03 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.816, T_{max} = 0.982
11614 measured reflections
4010 independent reflections
3330 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.00
4010 reflections
253 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O4^{i}	0.87 (1)	1.97 (1)	2.823 (2)	164 (2)
N4—H4···O3^{i}	0.88 (1)	2.05 (1)	2.861 (2)	154 (2)

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the University of Malaya (grant Nos. FS339/2008 A and PS206/2008 A) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2396).

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supporting information

Acta Cryst. (2009). E**65**, m422 [doi:10.1107/S1600536809009520]

(Benzohydrazidato-κ^2N',O)[2-(benzoylhydrazono-κ^2N,O)propionato-κO]oxidovanadium(V)

Hon Wee Wong, Kong Mun Lo and Seik Weng Ng

S1. Experimental

2-[Benzoylhydrazono]propionic acid (0.66 g, 3.2 mmol), prepared from the condensation reaction of benzhydrazide and pyruvic acid, was dissolved in of ethanol (50 ml). It was then mixed with vanadyl sulfate (0.26 g, 1.6 mmol) in distilled water (20 ml) and the mixture was heated for 5 h. Upon slow evaporation of the filtrate, red crystals formed.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2-1.5*U*(C).

The nitrogen-bound H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H 0.88±0.01 Å; their U_{iso} values were freely refined.

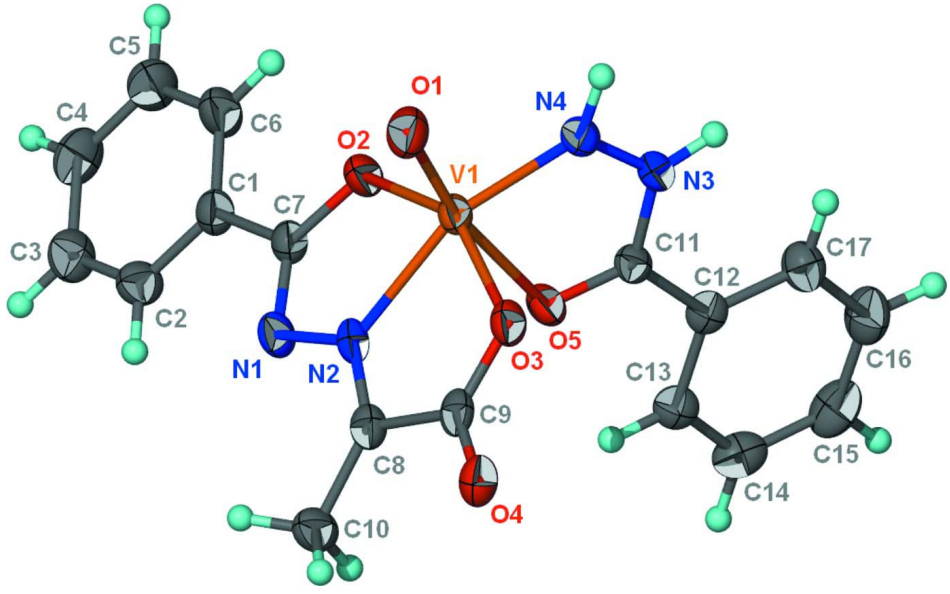

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $VO(C_7H_7N_2O)(C_{10}H_8N_2O_3)$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

(Benzohydrazidato-κ^2N',O)[2-(benzoylhydrazono- κ^2N,O)propionato-κO]oxidovanadium(V)

Crystal data

$[V(C_7H_7N_2O)(C_{10}H_8N_2O_3)O]$
$M_r = 406.27$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.9424$ (2) Å
$b = 6.2384$ (1) Å
$c = 25.7215$ (5) Å
$\beta = 96.603$ (1)°
$V = 1744.18$ (5) Å^3
$Z = 4$
$F(000) = 832$
$D_x = 1.547$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3550 reflections
$\theta = 2.9$–$28.0°$
$\mu = 0.61$ mm^{-1}
$T = 123$ K
Prism, red
0.35 × 0.10 × 0.03 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.816$, $T_{max} = 0.982$
11614 measured reflections
4010 independent reflections
3330 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -14 \rightarrow 14$
$k = -8 \rightarrow 7$
$l = -32 \rightarrow 33$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.00$
4010 reflections
253 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.9013P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.63939 (3)	0.51362 (5)	0.667678 (11)	0.02025 (10)
O1	0.71456 (13)	0.3013 (2)	0.65899 (5)	0.0310 (3)
O2	0.47660 (11)	0.4125 (2)	0.63552 (5)	0.0252 (3)
O3	0.77761 (11)	0.7223 (2)	0.66978 (4)	0.0227 (3)
O4	0.87206 (11)	0.9694 (2)	0.62563 (5)	0.0250 (3)
O5	0.53800 (11)	0.7850 (2)	0.69541 (4)	0.0229 (3)
N1	0.49712 (13)	0.6388 (3)	0.56603 (5)	0.0230 (3)
N2	0.60460 (13)	0.6815 (2)	0.59808 (5)	0.0200 (3)
N3	0.57784 (13)	0.6090 (2)	0.76970 (5)	0.0204 (3)
H3	0.582 (2)	0.584 (4)	0.8033 (4)	0.039 (6)*
N4	0.63345 (14)	0.4678 (2)	0.73974 (6)	0.0213 (3)
H4	0.6650 (17)	0.363 (3)	0.7597 (7)	0.029 (6)*

C1	0.32064 (16)	0.4069 (3)	0.56296 (7)	0.0235 (4)
C2	0.26918 (17)	0.4956 (3)	0.51595 (7)	0.0271 (4)
H2	0.3101	0.6096	0.5006	0.033*
C3	0.15882 (18)	0.4191 (3)	0.49134 (8)	0.0316 (4)
H3A	0.1242	0.4805	0.4592	0.038*
C4	0.09839 (18)	0.2527 (3)	0.51358 (8)	0.0346 (5)
H4A	0.0221	0.2011	0.4968	0.041*
C5	0.14961 (19)	0.1621 (4)	0.56019 (8)	0.0366 (5)
H5	0.1086	0.0477	0.5754	0.044*
C6	0.26067 (19)	0.2380 (3)	0.58478 (8)	0.0330 (5)
H6	0.2959	0.1747	0.6166	0.040*
C7	0.43810 (16)	0.4902 (3)	0.58978 (7)	0.0221 (4)
C8	0.67646 (16)	0.8356 (3)	0.58852 (7)	0.0217 (4)
C9	0.78589 (16)	0.8481 (3)	0.62982 (6)	0.0212 (4)
C10	0.65613 (17)	0.9934 (3)	0.54563 (7)	0.0273 (4)
H10A	0.6174	0.9223	0.5139	0.041*
H10B	0.7352	1.0546	0.5388	0.041*
H10C	0.6022	1.1080	0.5557	0.041*
C11	0.52823 (15)	0.7794 (3)	0.74341 (6)	0.0191 (3)
C12	0.46480 (15)	0.9478 (3)	0.77016 (7)	0.0207 (4)
C13	0.41422 (15)	1.1174 (3)	0.73968 (7)	0.0235 (4)
H13	0.4241	1.1237	0.7035	0.028*
C14	0.34966 (17)	1.2765 (3)	0.76211 (8)	0.0297 (4)
H14	0.3142	1.3913	0.7413	0.036*
C15	0.33666 (18)	1.2683 (3)	0.81518 (8)	0.0341 (5)
H15	0.2914	1.3766	0.8305	0.041*
C16	0.38955 (18)	1.1029 (4)	0.84576 (8)	0.0335 (5)
H16	0.3821	1.1002	0.8822	0.040*
C17	0.45334 (17)	0.9412 (3)	0.82367 (7)	0.0275 (4)
H17	0.4889	0.8270	0.8447	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.02461 (16)	0.02017 (17)	0.01501 (15)	0.00332 (12)	−0.00179 (11)	−0.00063 (11)
O1	0.0386 (8)	0.0277 (7)	0.0250 (7)	0.0083 (6)	−0.0030 (6)	−0.0049 (6)
O2	0.0300 (7)	0.0264 (7)	0.0178 (6)	−0.0041 (6)	−0.0026 (5)	0.0027 (5)
O3	0.0242 (6)	0.0258 (7)	0.0172 (6)	0.0014 (5)	−0.0019 (5)	−0.0025 (5)
O4	0.0224 (6)	0.0308 (7)	0.0215 (6)	−0.0022 (5)	0.0014 (5)	−0.0059 (5)
O5	0.0279 (6)	0.0242 (7)	0.0165 (6)	0.0053 (5)	0.0026 (5)	0.0028 (5)
N1	0.0215 (7)	0.0289 (8)	0.0172 (7)	−0.0031 (6)	−0.0030 (6)	0.0004 (6)
N2	0.0206 (7)	0.0252 (8)	0.0137 (6)	0.0013 (6)	−0.0007 (5)	−0.0025 (6)
N3	0.0226 (7)	0.0220 (8)	0.0160 (7)	0.0002 (6)	0.0002 (6)	0.0015 (6)
N4	0.0241 (7)	0.0199 (8)	0.0190 (7)	0.0034 (6)	−0.0016 (6)	0.0014 (6)
C1	0.0243 (9)	0.0263 (9)	0.0196 (8)	−0.0021 (7)	0.0010 (7)	−0.0026 (7)
C2	0.0265 (9)	0.0267 (10)	0.0273 (9)	−0.0012 (8)	−0.0003 (7)	0.0026 (8)
C3	0.0283 (10)	0.0350 (11)	0.0292 (10)	0.0044 (9)	−0.0060 (8)	−0.0004 (9)
C4	0.0261 (10)	0.0374 (12)	0.0386 (11)	−0.0060 (9)	−0.0035 (8)	−0.0076 (9)

C5	0.0387 (11)	0.0377 (12)	0.0330 (11)	−0.0150 (10)	0.0019 (9)	−0.0001 (9)
C6	0.0381 (11)	0.0372 (12)	0.0224 (9)	−0.0108 (9)	−0.0021 (8)	0.0017 (8)
C7	0.0256 (9)	0.0230 (9)	0.0173 (8)	0.0005 (7)	0.0007 (7)	−0.0019 (7)
C8	0.0229 (8)	0.0244 (9)	0.0176 (8)	0.0020 (7)	0.0016 (7)	−0.0030 (7)
C9	0.0222 (8)	0.0236 (9)	0.0176 (8)	0.0049 (7)	0.0009 (6)	−0.0065 (7)
C10	0.0277 (9)	0.0282 (10)	0.0249 (9)	−0.0042 (8)	−0.0024 (7)	0.0036 (8)
C11	0.0180 (8)	0.0206 (8)	0.0180 (8)	−0.0017 (7)	−0.0004 (6)	0.0006 (7)
C12	0.0154 (8)	0.0232 (9)	0.0236 (9)	−0.0025 (7)	0.0036 (6)	−0.0017 (7)
C13	0.0186 (8)	0.0254 (9)	0.0266 (9)	−0.0025 (7)	0.0027 (7)	0.0008 (7)
C14	0.0227 (9)	0.0252 (10)	0.0408 (11)	0.0006 (8)	0.0023 (8)	0.0005 (8)
C15	0.0262 (10)	0.0337 (11)	0.0436 (12)	0.0012 (8)	0.0095 (9)	−0.0120 (9)
C16	0.0339 (10)	0.0403 (12)	0.0280 (10)	−0.0011 (9)	0.0109 (8)	−0.0078 (9)
C17	0.0267 (9)	0.0329 (10)	0.0235 (9)	0.0014 (8)	0.0053 (7)	−0.0009 (8)

Geometric parameters (Å, º)

V1—O1	1.589 (1)	C3—H3A	0.9500
V1—O2	1.979 (1)	C4—C5	1.384 (3)
V1—O3	1.992 (1)	C4—H4A	0.9500
V1—O5	2.188 (1)	C5—C6	1.387 (3)
V1—N2	2.071 (1)	C5—H5	0.9500
V1—N4	1.884 (2)	C6—H6	0.9500
O2—C7	1.297 (2)	C8—C10	1.476 (3)
O3—C9	1.304 (2)	C8—C9	1.509 (2)
O4—C9	1.223 (2)	C10—H10A	0.9800
O5—C11	1.252 (2)	C10—H10B	0.9800
N1—C7	1.319 (2)	C10—H10C	0.9800
N1—N2	1.382 (2)	C11—C12	1.473 (2)
N2—C8	1.283 (2)	C12—C13	1.393 (3)
N3—C11	1.341 (2)	C12—C17	1.397 (2)
N3—N4	1.359 (2)	C13—C14	1.382 (3)
N3—H3	0.874 (9)	C13—H13	0.9500
N4—H4	0.879 (9)	C14—C15	1.389 (3)
C1—C2	1.389 (3)	C14—H14	0.9500
C1—C6	1.393 (3)	C15—C16	1.383 (3)
C1—C7	1.481 (2)	C15—H15	0.9500
C2—C3	1.382 (3)	C16—C17	1.385 (3)
C2—H2	0.9500	C16—H16	0.9500
C3—C4	1.389 (3)	C17—H17	0.9500
O1—V1—N4	95.10 (7)	C4—C5—H5	120.0
O1—V1—O2	97.53 (7)	C6—C5—H5	120.0
N4—V1—O2	103.39 (6)	C5—C6—C1	120.22 (19)
O1—V1—O3	98.34 (7)	C5—C6—H6	119.9
N4—V1—O3	100.63 (6)	C1—C6—H6	119.9
O2—V1—O3	149.76 (5)	O2—C7—N1	123.88 (16)
O1—V1—N2	110.29 (6)	O2—C7—C1	117.71 (16)
N4—V1—N2	154.60 (6)	N1—C7—C1	118.41 (15)

O2—V1—N2	74.50 (5)	N2—C8—C10	126.85 (16)
O3—V1—N2	75.90 (5)	N2—C8—C9	110.97 (15)
O1—V1—O5	168.87 (6)	C10—C8—C9	122.07 (16)
N4—V1—O5	73.77 (5)	O4—C9—O3	124.44 (16)
O2—V1—O5	85.25 (5)	O4—C9—C8	121.82 (16)
O3—V1—O5	84.11 (5)	O3—C9—C8	113.73 (15)
N2—V1—O5	80.84 (5)	C8—C10—H10A	109.5
C7—O2—V1	116.27 (11)	C8—C10—H10B	109.5
C9—O3—V1	119.54 (11)	H10A—C10—H10B	109.5
C11—O5—V1	113.82 (11)	C8—C10—H10C	109.5
C7—N1—N2	106.71 (14)	H10A—C10—H10C	109.5
C8—N2—N1	121.87 (15)	H10B—C10—H10C	109.5
C8—N2—V1	119.25 (12)	O5—C11—N3	116.36 (15)
N1—N2—V1	118.51 (11)	O5—C11—C12	122.63 (15)
C11—N3—N4	114.16 (14)	N3—C11—C12	121.00 (15)
C11—N3—H3	127.7 (16)	C13—C12—C17	120.13 (16)
N4—N3—H3	118.0 (16)	C13—C12—C11	117.19 (15)
N3—N4—V1	121.87 (11)	C17—C12—C11	122.68 (16)
N3—N4—H4	108.9 (13)	C14—C13—C12	119.97 (17)
V1—N4—H4	129.3 (13)	C14—C13—H13	120.0
C2—C1—C6	119.31 (17)	C12—C13—H13	120.0
C2—C1—C7	120.58 (16)	C13—C14—C15	119.90 (19)
C6—C1—C7	120.11 (17)	C13—C14—H14	120.0
C3—C2—C1	120.43 (18)	C15—C14—H14	120.0
C3—C2—H2	119.8	C16—C15—C14	120.17 (18)
C1—C2—H2	119.8	C16—C15—H15	119.9
C2—C3—C4	120.11 (19)	C14—C15—H15	119.9
C2—C3—H3A	119.9	C15—C16—C17	120.52 (18)
C4—C3—H3A	119.9	C15—C16—H16	119.7
C5—C4—C3	119.82 (19)	C17—C16—H16	119.7
C5—C4—H4A	120.1	C16—C17—C12	119.28 (18)
C3—C4—H4A	120.1	C16—C17—H17	120.4
C4—C5—C6	120.10 (19)	C12—C17—H17	120.4

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O4^{i}	0.87 (1)	1.97 (1)	2.823 (2)	164 (2)
N4—H4···O3^{i}	0.88 (1)	2.05 (1)	2.861 (2)	154 (2)

Symmetry code: (i) −*x*+3/2, *y*−1/2, −*z*+3/2.