Crystal structure of (*N*-(2-hydroxyl-1-naphthal)ethylenediamine)(isonicotinyl hydrazide)oxovanadium(V), $[VO(C_6H_5N_3O)(C_{13}H_{13}N_2O)]$

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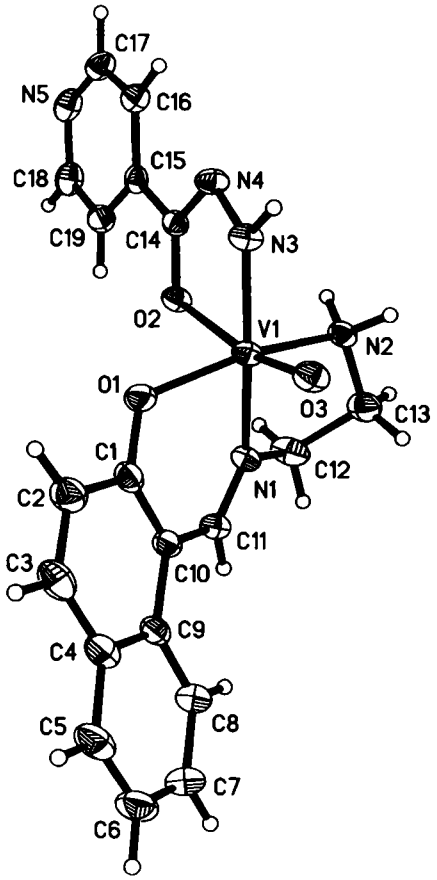

Abstract

$C_{19}H_{18}N_5O_3V$, monoclinic, $P12_1/c1$ (no. 14), $a = 14.935(2)$ Å, $b = 7.130(1)$ Å, $c = 17.573(2)$ Å, $\beta = 97.084(2)°$, $V = 1856.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.035$, $wR_{ref}(F^2) = 0.097$, $T = 273$ K.

Source of material

The ligand 2-hydroxyl-1-naphthaldehyde isonicotinyl hydrazone was prepared according to [1] and characterized by IR and ^{1}H NMR. 0.13 g $VO(acac)_2$ was dissolved in 10.0 ml CH_3OH, and was added dropwise to a methanol solution of 0.15 g 2-hydroxyl-1-naphthaldehyde isonicotinyl hydrazone under continuous stirring. The reaction lasted for two hours, and a mixture of 2.5 ml CH_3OH and 5.5 ml ethylenediamine was added. After filtration, the brownish filtrate was layered with Et_2O, allowed to stand at room temperature. Brownish-red crystals of the vanadium(V) complex suitable for X-ray diffraction were obtained after five days. V(IV) was oxidized to V(V) by air oxygen.

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Discussion

The discovery of the insulin-like *in vitro* and *in vivo* activity of vanadium compounds has stimulated research on vanadium compounds that may have important application in the treatment of diabetes mellitus [2,3]. Detailed physicochemical characterization of vanadium compounds will help in further understanding of pharmacology of vanadium.

It can be seen from the molecular structure that 2-hydroxyl-1-naphthaldehyde dissociated from the original hydrazone combined with ethylenediamine to form a new Schiff base, which coordinates vanadium in a tridentate mode. An octahedral coordination sphere N_3O_3 exists around the vanadium center. The equatorial plane in the octahedron is defined by N3 from isonicotinyl hydrazide as well as O1, N1 and N2 from the newly formed Schiff base 2-hydroxyl-1-naphthal-*N*-ethylenediamine with a mean deviation of 0.04 Å. The metal atom is 0.270 Å out of this plane towards the apical oxo oxygen O3. The V1—O2 bond distance is 2.141(2) Å, which is lying *trans* to V1=O3 and lengthened due to the *trans* influence of the oxo atom. Along the axis of the octahedron, the angle O2–V1–O3 is 167.64(7)°, and two axis angles in the equational plane of the octahedron are very similar, N1–V1–N3 of 158.58(8)° and N2–V1–O1 of 158.27(7)°. The bite angles of the tridentate Schiff base ligand are 77.41(7)° for the five-membered ring and 83.40(7)° for the six-membered ring. These two chelate rings deviate slightly from planarity with the mean deviations being 0.16 Å (five-membered ring) and 0.18 Å (six-membered ring). The bond distances and angles for the isonicotinyl hydrazide moiety have normal values. The crystal structure is stabilized by a net of hydrogen bonds involving the imine group NH and one neighboring ligand N5(x,$-y+½$,$z-½$) (d(N3···N5′) = 3.144 Å, ∠N3–H3A···N5′ = 159.0°), the amine NH_2 group which is hydrogen-bonded to N5($-x+1$,$-y+1$,$-z+1$) with d(N2···N5″) = 3.156 Å, ∠N2–H2A···N5″ = 166.4° and to N4($-x+1$,$y+½$,$-z+½$) with d(N2–H2B···N4‴) = 2.972 Å, ∠N2–H2B···N5‴ = 145.7°. The naphthalene nuclei of adjacent vanadium units are packed in pairs which satisfies well the requirement the intermolecular π-π stacking interactions with a mean centroid-centroid distance of 3.61 Å.

Table 1. Data collection and handling.

Crystal:	brownish-red block, size 0.2 × 0.2 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.66 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9676, 3271
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2612
$N(param)_{refined}$:	253
Programs:	SHELXS-97 [5], SHELXL-97 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2A)	4e	0.4064	0.8144	0.2236	0.044
H(2B)	4e	0.4022	0.7628	0.3091	0.044
H(3A)	4e	0.4494	0.3873	0.1956	0.045
H(2)	4e	0.1195	0.0860	0.1334	0.063
H(3)	4e	−0.0147	0.1136	0.0587	0.066
H(5)	4e	−0.1448	0.3036	0.0015	0.070
H(6)	4e	−0.2257	0.5732	−0.0027	0.079
H(7)	4e	−0.1768	0.8199	0.0780	0.082
H(8)	4e	−0.0440	0.8020	0.1568	0.065

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(11)	4e	0.0710	0.7631	0.2350	0.046
H(12A)	4e	0.2367	0.8225	0.3504	0.063
H(12B)	4e	0.1748	0.9520	0.2933	0.063
H(13A)	4e	0.2883	0.9951	0.2175	0.063
H(13B)	4e	0.3323	1.0329	0.3022	0.063
H(16)	4e	0.5613	0.2195	0.4162	0.048
H(17)	4e	0.6060	0.1450	0.5415	0.055
H(18)	4e	0.3803	0.3157	0.6030	0.057
H(19)	4e	0.3274	0.3979	0.4800	0.049

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	4e	0.30064(2)	0.53157(6)	0.22010(2)	0.0266(2)	0.0422(3)	0.0306(2)	0.0015(2)	0.0005(2)	−0.0031(2)
O(1)	4e	0.2097(1)	0.3356(2)	0.20968(9)	0.035(1)	0.0395(9)	0.053(1)	−0.0001(7)	−0.0061(8)	0.0006(8)
O(2)	4e	0.3277(1)	0.4523(2)	0.33818(8)	0.0295(9)	0.047(1)	0.0317(9)	0.0022(7)	0.0047(7)	0.0004(7)
O(3)	4e	0.3030(1)	0.5815(2)	0.13163(9)	0.038(1)	0.064(1)	0.0328(9)	0.0008(8)	0.0001(7)	0.0006(8)
N(1)	4e	0.1949(1)	0.6959(3)	0.2495(1)	0.029(1)	0.041(1)	0.037(1)	−0.0015(8)	0.0017(8)	−0.0043(8)
N(2)	4e	0.3676(1)	0.7822(3)	0.2615(1)	0.029(1)	0.048(1)	0.034(1)	−0.0042(9)	−0.0005(8)	0.0002(8)
N(3)	4e	0.4129(1)	0.4056(3)	0.2315(1)	0.033(1)	0.052(1)	0.027(1)	0.0062(9)	0.0024(8)	−0.0042(8)
N(4)	4e	0.4579(1)	0.3464(3)	0.2995(1)	0.032(1)	0.050(1)	0.030(1)	0.0074(9)	0.0000(8)	−0.0024(8)
N(5)	4e	0.4983(2)	0.2219(3)	0.5850(1)	0.065(2)	0.041(1)	0.034(1)	−0.004(1)	−0.004(1)	0.0032(9)
C(1)	4e	0.1281(2)	0.3571(3)	0.1730(1)	0.034(1)	0.043(1)	0.043(1)	−0.010(1)	0.002(1)	0.000(1)
C(2)	4e	0.0892(2)	0.2002(4)	0.1309(2)	0.046(2)	0.049(2)	0.061(2)	−0.005(1)	−0.001(1)	−0.008(1)
C(3)	4e	0.0082(2)	0.2168(4)	0.0871(2)	0.049(2)	0.062(2)	0.052(2)	−0.019(1)	0.002(1)	−0.016(1)
C(4)	4e	−0.0426(2)	0.3840(4)	0.0828(1)	0.038(1)	0.064(2)	0.040(1)	−0.011(1)	0.004(1)	0.000(1)
C(5)	4e	−0.1242(2)	0.4032(5)	0.0331(2)	0.042(2)	0.087(2)	0.043(2)	−0.017(2)	−0.002(1)	−0.001(1)
C(6)	4e	−0.1725(2)	0.5624(5)	0.0306(2)	0.041(2)	0.096(3)	0.056(2)	−0.008(2)	−0.012(1)	0.014(2)
C(7)	4e	−0.1423(2)	0.7112(5)	0.0785(2)	0.045(2)	0.076(2)	0.080(2)	0.006(2)	−0.013(2)	0.021(2)
C(8)	4e	−0.0631(2)	0.7001(4)	0.1260(2)	0.039(2)	0.060(2)	0.061(2)	0.001(1)	−0.004(1)	0.007(1)
C(9)	4e	−0.0095(2)	0.5363(4)	0.1292(1)	0.029(1)	0.054(2)	0.039(1)	−0.007(1)	0.004(1)	0.005(1)
C(10)	4e	0.0774(2)	0.5213(3)	0.1748(1)	0.030(1)	0.046(1)	0.035(1)	−0.003(1)	0.002(1)	0.004(1)
C(11)	4e	0.1120(2)	0.6733(3)	0.2225(1)	0.031(1)	0.044(1)	0.039(1)	0.003(1)	0.003(1)	−0.000(1)
C(12)	4e	0.2227(2)	0.8593(4)	0.2971(2)	0.040(2)	0.054(2)	0.062(2)	0.002(1)	0.002(1)	−0.026(1)
C(13)	4e	0.3046(2)	0.9380(3)	0.2674(2)	0.041(2)	0.041(2)	0.072(2)	−0.005(1)	−0.006(1)	−0.010(1)
C(14)	4e	0.4050(2)	0.3753(3)	0.3537(1)	0.029(1)	0.034(1)	0.033(1)	−0.005(1)	−0.0009(9)	−0.0012(9)
C(15)	4e	0.4383(2)	0.3193(3)	0.4331(1)	0.037(1)	0.029(1)	0.033(1)	−0.009(1)	−0.000(1)	−0.0010(9)
C(16)	4e	0.5229(2)	0.2412(3)	0.4532(1)	0.042(1)	0.040(1)	0.037(1)	−0.000(1)	0.001(1)	−0.000(1)
C(17)	4e	0.5489(2)	0.1966(3)	0.5288(1)	0.047(2)	0.042(1)	0.044(2)	0.001(1)	−0.010(1)	0.004(1)
C(18)	4e	0.4173(2)	0.2963(4)	0.5648(1)	0.062(2)	0.048(2)	0.034(1)	−0.005(1)	0.009(1)	0.004(1)
C(19)	4e	0.3847(2)	0.3464(3)	0.4909(1)	0.039(1)	0.044(1)	0.038(1)	−0.002(1)	0.002(1)	0.003(1)

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References

1. Richardson, D. R.; Bernhardt, P. V.: Crystal and molecular structure of 2-hydroxy-1-naphthaldehyde isonicotinoyl hydrazone (NIH) and its iron(III) complex: an iron chelator with anti-tumour activity. J. Biol. Inorg. Chem. **4** (1999) 266-273.
2. Rehder, D.: Biological and medicinal aspects of vanadium. Inorg. Chem. Commun. **6** (2003) 604-617.
3. Thompson, K. H.; Chiles J.; Yuen, V. G.; Tse, J.; McNeill, J. H.; Orvig, C.: Comparison of anti-hyperglycemic effect amongst vanadium, molybdenum and other metal maltol complexes. J. Inorg. Biochem. **98** (2004) 683-690.
4. Pohlmann, A.; Nica, S.; Luong, T. K. K.; Plass, W.: Dioxovanadium(V) complexes with side chain substituted *N*-salicylidenehydrazides modelling supramolecular interactions in vanadium haloperoxidases. Inorg. Chem. Commun. **8** (2005) 289-292.
5. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
6. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.