Crystal structure of ethanolammonium dioxo-(*N*-(2-ethanolato)-2-oxy-3-methoxybenzaldiminato-*O,N,O'*)vanadate(V), $(NH_3CH_2CH_2OH)[VO_2\{(C_6H_4(OCH_3)(O)CHNCCH_2CH_2O\}]$

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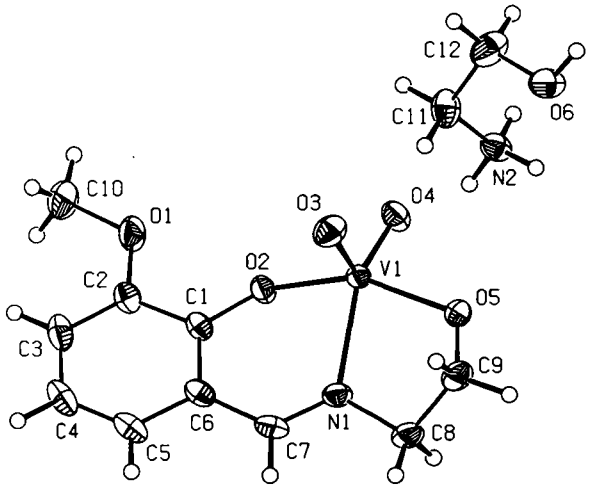

Abstract

$C_{12}H_{19}N_2O_6V$, monoclinic, $P12_1/c1$ (no. 14), a = 8.986(2) Å, b = 13.168(2) Å, c = 13.243(2) Å, β = 108.677(2)°, V = 1484.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.083, T = 294 K.

Source of material

Methanol solution of ammonium metavanadate (0.06 g, 0.5 mmol) and ethanolamine (0.3 ml) was added dropwise to the solution of *o*-vanillin-hydroxylamine (0.07 g, 0.5 mmol) dissolved in methanol with stirring. The reaction mixture was stirred for 15 h at RT, and the resulting yellow-brown solution was filtered off. The block-shaped yellow crystals of the title complex were deposited on the tube wall near the bottom end 24 h later.

Discussion

Vanadium is an ultramicrotrace bioelement with interesting biological activity [1]. Coordination complexes of vanadium which may have pharmacological relevance, such as BMOV, the most widely and intensively tested of the many proposed insulin mimetic vanadium complexes [2]. This motivated us to search for more pharmaceutical active vanadium complexes.

The X-ray structure analysis reveals that the title complex is a monomeric dioxovanadium(V) species with the formula $(HOCH_2CH_2NH_3)^+[VO_2(L)]^-$, and the protonated ethanolamine is a counter cation. The dioxovanadium(V) moiety is located in a quasi-square pyramidal environment. The basal plane is composed of the imine nitrogen atom (N1), phenolate oxygen atom (O2), vanadyl oxygen atom (O4) and ethanolate oxygen atom (O5), with the mean deviation from plane of 0.083 Å. The vanadium atom deviates from the plane by about 0.492 Å. Chelation gives rise to one five-membered (V1-O5-C9-C8-N1) and one six-membered (V1-O2-C1-C6-C7-N1) rings. Both of the rings are slightly folded. The ligand system is practically planar with the mean deviation from plane about 0.013 Å, and its angle with the coordination quasi-plane is about 17.9°. The apical position is occupied by the vanadyl oxygen atom O3. The short V—O3 and V—O4 bond distances of 1.609 Å and 1.655 Å, respectively, characterize the O=V=O double bonds [3]. The V—N1 bond length of 2.156 Å is similar to that in other analogues [4-5]. The N1—C7 distance of 1.284 Å confirms the coordination of the ethanolamine to the *o*-vanillin. The formative congeries array regularly and parallel the *b* axis. There are hydrogen bond interaction and π-π packing effect showing in the packing scheme, with all the hydrogen bond lengths (1.828 Å – 2.516 Å) in the normal range [6] and the distance of vicinal π-π plane about 3.302 Å.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.18 × 0.20 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.96 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7338, 2613
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2074
$N(param)_{refined}$:	193
Programs:	SHELXS-97 [7], SHELXL-97 [8]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	0.2619	0.6079	0.4726	0.052
H(2B)	4*e*	0.3233	0.5071	0.5070	0.052
H(2C)	4*e*	0.1724	0.5379	0.5154	0.052
H(6)	4*e*	−0.1733	0.5807	0.3525	0.075
H(3)	4*e*	0.8662	0.0621	0.4134	0.051
H(4)	4*e*	0.7968	−0.0820	0.4892	0.053
H(5)	4*e*	0.6671	−0.0669	0.6114	0.046
H(7)	4*e*	0.5627	0.0356	0.7246	0.041
H(8A)	4*e*	0.4962	0.2028	0.8550	0.048
H(8B)	4*e*	0.3985	0.1060	0.8023	0.048
H(9A)	4*e*	0.2051	0.2044	0.6871	0.047
H(9B)	4*e*	0.2372	0.2560	0.7994	0.047
H(10A)	4*e*	0.9921	0.2208	0.4067	0.090
H(10B)	4*e*	0.9023	0.3176	0.3496	0.090
H(10C)	4*e*	0.8331	0.2093	0.3136	0.090
H(11A)	4*e*	0.1355	0.4319	0.3726	0.053
H(11B)	4*e*	0.2261	0.5112	0.3256	0.053
H(12A)	4*e*	0.0276	0.6311	0.3154	0.063
H(12B)	4*e*	−0.0380	0.5360	0.2433	0.063

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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)	4*e*	0.45166(4)	0.31047(3)	0.60904(3)	0.0265(2)	0.0238(2)	0.0274(2)	0.0027(2)	0.0122(2)	0.0041(2)
O(1)	4*e*	0.8064(2)	0.2543(1)	0.4486(2)	0.052(1)	0.040(1)	0.061(1)	0.0040(9)	0.040(1)	0.0010(9)
O(2)	4*e*	0.6444(2)	0.2687(1)	0.5813(1)	0.0340(9)	0.0241(8)	0.043(1)	0.0039(7)	0.0222(8)	0.0035(7)
O(3)	4*e*	0.3166(2)	0.2799(1)	0.5003(1)	0.039(1)	0.054(1)	0.0315(9)	−0.0019(9)	0.0086(8)	0.0001(8)
O(4)	4*e*	0.4914(2)	0.4330(1)	0.6065(1)	0.0359(9)	0.0261(9)	0.052(1)	0.0030(7)	0.0227(9)	0.0040(8)
O(5)	4*e*	0.3326(2)	0.3292(1)	0.7055(1)	0.041(1)	0.036(1)	0.0376(9)	0.0080(8)	0.0240(8)	0.0081(8)
N(1)	4*e*	0.5035(2)	0.1752(2)	0.7058(2)	0.032(1)	0.032(1)	0.033(1)	0.0021(9)	0.0130(9)	0.0101(9)
N(2)	4*e*	0.2366(2)	0.5431(2)	0.4766(2)	0.028(1)	0.034(1)	0.045(1)	0.0037(9)	0.015(1)	0.009(1)
O(6)	4*e*	−0.0997(2)	0.5408(2)	0.3727(2)	0.033(1)	0.065(1)	0.059(1)	0.0162(9)	0.025(1)	0.027(1)
C(1)	4*e*	0.6848(3)	0.1754(2)	0.5603(2)	0.024(1)	0.027(1)	0.036(1)	0.005(1)	0.007(1)	−0.001(1)
C(2)	4*e*	0.7713(3)	0.1642(2)	0.4883(2)	0.029(1)	0.034(1)	0.041(1)	0.004(1)	0.012(1)	−0.002(1)
C(3)	4*e*	0.8115(3)	0.0686(2)	0.4620(2)	0.035(1)	0.046(2)	0.047(2)	0.009(1)	0.014(1)	−0.011(1)
C(4)	4*e*	0.7704(3)	−0.0180(2)	0.5079(2)	0.037(2)	0.031(2)	0.057(2)	0.009(1)	0.005(1)	−0.010(1)
C(5)	4*e*	0.6919(3)	−0.0089(2)	0.5800(2)	0.032(1)	0.025(1)	0.052(2)	0.004(1)	0.004(1)	0.001(1)
C(6)	4*e*	0.6478(3)	0.0872(2)	0.6076(2)	0.025(1)	0.027(1)	0.039(1)	0.003(1)	0.004(1)	0.003(1)
C(7)	4*e*	0.5679(3)	0.0938(2)	0.6860(2)	0.032(1)	0.027(1)	0.040(2)	0.002(1)	0.007(1)	0.012(1)
C(8)	4*e*	0.4272(3)	0.1745(2)	0.7890(2)	0.045(2)	0.042(2)	0.039(1)	0.005(1)	0.023(1)	0.015(1)
C(9)	4*e*	0.2830(3)	0.2397(2)	0.7443(2)	0.040(2)	0.044(2)	0.039(2)	−0.001(1)	0.022(1)	0.007(1)
C(10)	4*e*	0.8904(4)	0.2502(3)	0.3734(3)	0.064(2)	0.066(2)	0.068(2)	0.010(2)	0.047(2)	0.009(2)
C(11)	4*e*	0.1572(3)	0.5037(2)	0.3685(2)	0.030(1)	0.055(2)	0.051(2)	0.001(1)	0.018(1)	−0.009(1)
C(12)	4*e*	0.0071(3)	0.5589(3)	0.3164(2)	0.031(1)	0.088(2)	0.042(2)	0.005(2)	0.015(1)	0.012(2)

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