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Dioxo(pyridine-2-carboxylato)(pyridinium-2-carboxylato)vanadium(V) monohydrate

Lim Eng Khoon,[a] Teoh Siang Guan[a] and Bohari M. Yamin[b]*

[a]School of Chemical Sciences, Universiti Sains Malaysia, 11800 Minden, Penang, Malaysia, and [b]School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail: bohari@pkrisc.cc.ukm.my

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.092
Data-to-parameter ratio = 12.2

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

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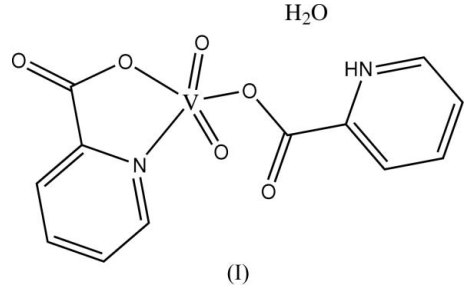
In the title compound, $[VO_2(C_6H_4NO_2)(C_6H_5NO_2)]\cdot H_2O$, one of the two pyridine-2-carboxylate ligands chelates to the V atom in a bidentate manner, *via* the carboxylate O atom and the N atom of the pyridine ring. The second pyridine-2-carboxylate ligand coordinates to the V atom only *via* the carboxylate O atom, while the N atom of the pyridine ring is protonated. The coordination geometry of the V atom is distorted trigonal–bipyramidal. The crystal packing is stabilized by intermolecular N—H···O, C—H···O and O—H···O hydrogen bonds involving the solvent water molecule.

Comment

Current interest in dioxovanadium compounds is very much driven by their promising pharmaceutical properties and biological activity. Bis(kojato) oxovanadium(IV) and bis-(maltolato)oxovanadium(IV) are two examples demonstrating insulin-mimetic properties (Yuen *et al.*, 1997; Winter *et al.*, 2005).



(I)

The title compound, (I), is a dioxovanadium complex with two pyridine-2-carboxylate ligands; one is chelated to the V atom in a bidentate manner *via* atom N2 of the pyridine ring and atom O4 of the carboxylate, and the second is linked through the carboxylate atom O2 in a monodentate fashion (Fig. 1). Atom N1 of the uncoordinated pyridine ring is protonated. The coordination geometry of atom V1 is highly distorted trigonal–bipyrimidal (Table 1). The mean values of the V═O(oxo) and V—O(carboxylate) bond lengths in (I) of 1.6045 (18) and 1.9873 (16) Å, respectively, are close to those observed in $[VO_2(C_{13}H_9ClN_3O]$, (II) [1.607 (4) and 1.972 (3) Å, respectively; Pal & Pal, 2001]. However, the V—N(pyridyl) bond length of 2.1252 (18) Å in (I) is slightly longer than the value of 1.972 (4) Å in (II). The bidentate chelate ligand and V atom form an essentially planar V1/O4/N2/O3/C7–C12 fragment, with a maximum deviation of 0.036 (2) Å for atom O3, and this makes a dihedral angle of 22.61 (8)° with the fragment N1/C1–C6.

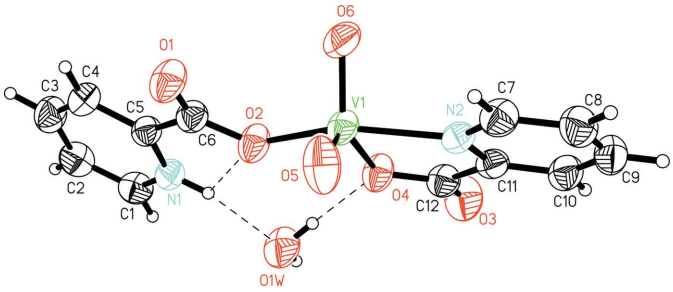


Figure 1
The molecular structure of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. Hydrogen bonds are shown by dashed lines.

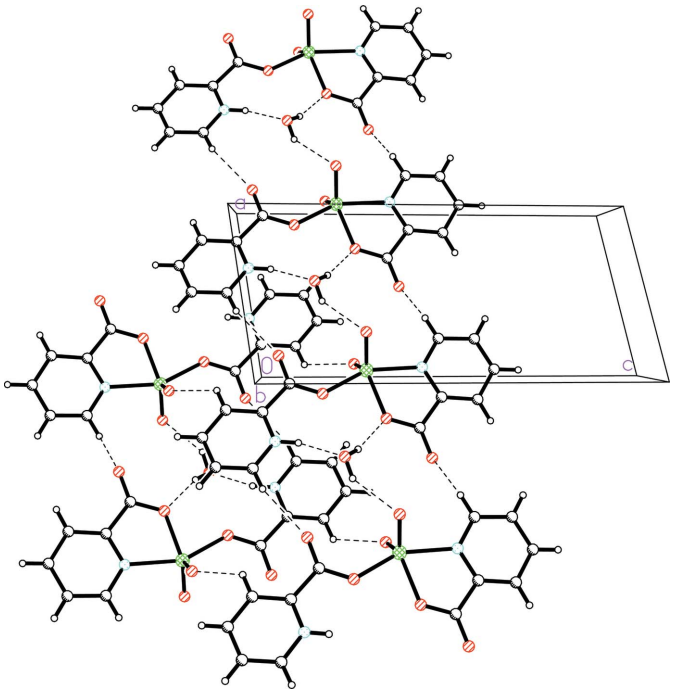


Figure 2
A packing diagram for (I), viewed down the *b* axis. Dashed lines denote intermolecular O—H···O, N—H···O and C—H···O hydrogen bonds.

The crystal packing (Fig. 2) is stabilized by intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds (Table 2) involving the solvent water molecules and forming ribbons extended along the *a* axis.

Experimental

Sodium metavanadate, $NaVO_3$ (1.00 g, 8.20 mmol), was dissolved in distilled water (50 ml). An aqueous solution (20 ml) of pyridine-2-carboxylic acid (2.20 g, 16.40 mmol) was then added to the vanadate solution and the mixture was acidified to pH 1–2.5 by dropwise addition of 2 *M* HCl. The resulting solution was stirred and refluxed for 3 h. The yellow solution was then filtered and allowed to cool to room temperature. Light-yellow crsytals of (I) were obtained after 3 d of slow evaporation at room temperature (yield 80%; decomposed at 594 K).

Crystal data

$[V(C_6H_4NO_2)(C_6H_5NO_2)O_2]\cdot H_2O$
M_r = 346.17
Triclinic, $P\overline{1}$
a = 7.3392 (15) Å
b = 7.4413 (16) Å
c = 14.592 (3) Å
α = 97.745 (3)°
β = 94.787 (3)°
γ = 118.655 (3)°
V = 682.8 (2) Å^3
Z = 2
D_x = 1.684 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2766 reflections
θ = 1.4–25.5°
μ = 0.77 mm^{-1}
T = 298 (2) K
Slab, yellow
0.48 × 0.14 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.710, T_{max} = 0.934
6777 measured reflections
2517 independent reflections
2173 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.5°
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.092
S = 1.04
2517 reflections
207 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.1867P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

V1—O6	1.6024 (18)	O1—C6	1.214 (3)
V1—O5	1.6066 (18)	O2—C6	1.281 (3)
V1—O2	1.9755 (16)	O3—C12	1.202 (3)
V1—O4	1.9990 (16)	O4—C12	1.303 (3)
V1—N2	2.1252 (18)		
O6—V1—O5	109.13 (12)	O2—V1—O4	78.70 (6)
O6—V1—O2	100.79 (8)	O6—V1—N2	94.65 (8)
O5—V1—O2	101.11 (8)	O5—V1—N2	93.24 (8)
O6—V1—O4	120.74 (10)	O2—V1—N2	154.05 (7)
O5—V1—O4	129.38 (10)	O4—V1—N2	75.49 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O2	0.86	2.30	2.643 (3)	104
O1W—H1WA···O4	0.84 (4)	2.01 (4)	2.828 (3)	164 (4)
N1—H1B···O1W	0.86	1.85	2.691 (3)	164
O1W—H1WB···O5^{i}	0.84 (3)	2.42 (4)	3.139 (3)	143 (4)
C1—H1A···O1^{i}	0.93	2.41	3.297 (4)	159
C4—H4A···O6^{ii}	0.93	2.46	3.121 (4)	128
C7—H7A···O3^{iii}	0.93	2.31	3.108 (4)	144

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, -y, -z$; (iii) $x+1, y, z$.

The H atoms of the solvent water molecule were located in a difference map and refined isotropically with bond-length restraints of O—H = 0.84 (3) Å. The remaining H atoms were located in a difference map, repositioned geometrically and refined as riding, with C—H = 0.93 and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq} of the parent atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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