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2,9-Dimethyl-1,10-phenanthrolinium dioxido(pyridine-2,6-dicarboxylato)-vanadate(V) monohydrate

Hossein Aghabozorg* and Elahe Sadr-khanlou

Department of Chemistry, Teacher Training University, Tehran, Iran
Correspondence e-mail: aghabozorg@saba.tmu.ac.ir

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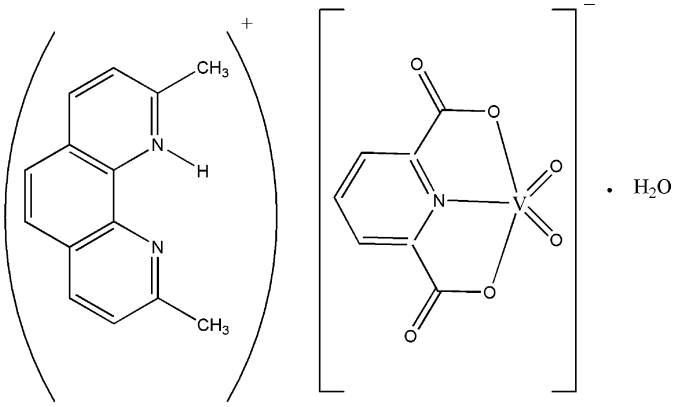
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.102; data-to-parameter ratio = 15.1.

In the title compound, $(C_{14}H_{13}N_2)[V(C_7H_3NO_4)O_2]\cdot H_2O$, the V^V atom has a distorted trigonal–bipyramidal coordination environment formed by one pyridyl N atom and two O atoms of the $[VO_2]^+$ group occupying the equatorial plane, and two carboxylate O atoms occupying the axial positions. O—H···O, N—H···O, O—H···N and C—H···O hydrogen bonds, together with π–π stacking interactions [average centroid-to-centroid distance 3.644 (12) Å], seem to be effective in the stabilization of the crystal structure, resulting in the formation of a three-dimensional supramolecular structure.

Related literature

For related literature, see: Ranjbar (2004).



Experimental

Crystal data

$(C_{14}H_{13}N_2)[V(C_7H_3NO_4)O_2]\cdot H_2O$
M_r = 475.32
Triclinic, $P\overline{1}$
a = 7.1194 (12) Å
b = 10.6843 (19) Å
c = 13.480 (2) Å
α = 81.166 (3)°
β = 82.397 (3)°
γ = 81.959 (4)°
V = 996.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 120 (2) K
0.05 × 0.05 × 0.02 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.973, T_{max} = 0.989
7763 measured reflections
4398 independent reflections
3677 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.102
S = 0.96
4398 reflections
291 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

V1—O2V	1.6158 (14)	V1—O1	2.0132 (13)
V1—O1V	1.6228 (14)	V1—N1	2.1107 (15)
V1—O3	1.9842 (13)		
O2V—V1—O1V	109.64 (7)	O3—V1—O1	147.22 (6)
O2V—V1—O3	101.07 (6)	O2V—V1—N1	117.60 (6)
O1V—V1—O3	99.96 (6)	O1V—V1—N1	132.68 (7)
O2V—V1—O1	99.69 (6)	O3—V1—N1	74.31 (5)
O1V—V1—O1	96.56 (6)	O1—V1—N1	73.66 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1AA···O1S^{i}	0.88	1.87	2.714 (2)	162
O1S—H1S···O1	0.85	1.89	2.728 (2)	168
O1S—H2S···N2A^{i}	0.85	2.49	2.964 (2)	116
C2A—H2AA···O2^{ii}	0.95	2.39	3.241 (2)	149
C5—H5···O4^{iii}	0.95	2.34	3.238 (2)	157

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Financial support by the Teacher Training University is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2236).

References

Bruker (1998). *SAINT-Plus* (Version 6.01), *SMART* (Version 5.059), *SADABS* (Version 2.01) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Ranjbar, M. (2004). *Anal. Sci.* **20**, x135–x136.

supplementary materials

Acta Cryst. (2007). E**63**, m1753 [**doi:10.1107/S1600536807023987**]

2,9-Dimethyl-1,10-phenanthrolinium dioxido(pyridine-2,6-dicarboxylato)vanadate(V) monohydrate

H. Aghabozorg and E. Sadr-khanlou

Comment

In recent years, the fundamental coordination chemistry and characterization of vanadium compounds have attracted considerable attention, due to their actions in biological systems. A five coordinated vanadium(V) complex with the same ligand was previously reported (Ranjbar, 2004) in which one ($C_7H_3NO_4$) anion was attached to a metal center. We herein report the crystal structure of the title compound, (I).

The molecule of (I) contains one $(C_{14}H_{13}N_2)^+$ cation, one water molecule and one $[V(C_7H_3NO_4)(O_2)]^-$ anion, where the vanadium(V) atom has a distorted trigonal bipyramidal coordination environment (Fig. 1, Table 1). The coordinated pyridyl nitrogen atom (N1) and two oxygen atoms (O1V, O2V) of the VO_2^+ group occupy the distorted equatorial plane, while the two carboxylate oxygen atoms (O1 and O3) occupy the axial positions around the central atom.

The V1—O1V [1.6228 (14) Å] and V1—O2V [1.6158 (14) Å] bonds are shorter than V1—O1 [2.0132 (13) Å] and V1—O3 [1.9842 (13) Å] bonds, due to the formation of double bonds. The water molecule resides between two ionic units making a bridge-like hydrogen bond. Beside the classic hydrogen bonds, there are also C—H···O type hydrogen bonds (Table 2), which are responsible for stabilization of the crystal network. Each vanadium(V) complex is attached to the neighboring complex and four $(C_{14}H_{13}N_2)^+$ units *via* C—H···O interactions. These interactions, coming inconcert, make an infinite layers which could be described by $R^2_2(10)$, $C^2_2(15)$ and $R^3_3(13)$ graph set descriptors (Fig. 2). Furthermore, considering the average values for intercentroid [3.644 (12) Å] and interplanar [3.297 (16) Å] distances for $(C_{14}H_{13}N_2)^+$ ions [symmetry codes: x, y, z; $x-1, y, z$; $-x, -y, -z+2$; $-x-1, -y, -z+2$], the π-π stacking interaction between cations can be established. Thus, the three-dimensional supramolecular structure for (I) is confirmed.

Experimental

A pale yellow colored vanadium(V) complex of cation and anion as a proton transfer agent, the title compound, (I), was isolated at pH = 3.0 by stirring an aqueous mixture of the ligand [2,9-dimethyl-1,10-phenathroline (21.2 mg) and pyridine-2,6-dicarboxylic acid (13.9 mg) in water (10 ml)] with 0.5 molar equivalent of vanadium(III) chloride (8.1 mg) at room temperature (yield; 1.79 mg, 73%, m.p. 450 K). Slow evaporation of the solvent during two weeks resulted in X-ray quality crystals. Elemental analysis revealed that one molecule of water is associated as solvate.

Refinement

H atoms were positioned geometrically, with N—H = 0.88 Å (for NH), O—H = 0.85 Å (for OH_2) and C—H = 0.95 and 0.98 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,N,O)$, where $x = 1.5$ for water and methyl H, and $x = 1.2$ for all other H atoms.

Figures

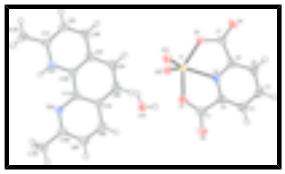


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

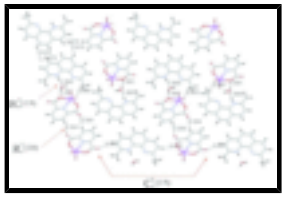


Fig. 2. Graph set descriptors made by different *X*—H···O interactions.

2,9-Dimethyl-1,10-phenanthrolinium dioxo(pyridine-2,6-dicarboxylato)vanadate(V) monohydrate

Crystal data

$(C_{14}H_{13}N_2)[V(C_7H_3NO_4)O_2]\cdot H_2O$	$Z = 2$
$M_r = 475.32$	$F_{000} = 488$
Triclinic, $P\overline{1}$	$D_x = 1.584$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.1194$ (12) Å	Cell parameters from 921 reflections
$b = 10.6843$ (19) Å	$\theta = 2.3$–27.8°
$c = 13.480$ (2) Å	$\mu = 0.55$ mm^{-1}
$\alpha = 81.166$ (3)°	$T = 120$ (2) K
$\beta = 82.397$ (3)°	Prism, yellow
$\gamma = 81.959$ (4)°	0.05 × 0.05 × 0.02 mm
$V = 996.8$ (3) Å^3	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	4398 independent reflections
Radiation source: fine-focus sealed tube	3677 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 120(2)$ K	$\theta_{max} = 27.1^\circ$
φ and ω scans	$\theta_{min} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$h = -9\rightarrow8$
$T_{min} = 0.973$, $T_{max} = 0.989$	$k = -13\rightarrow13$
7763 measured reflections	$l = -17\rightarrow17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.5169P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.96$	$(\Delta/\sigma)_{max} = 0.001$
4398 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
291 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.23632 (4)	0.53331 (3)	0.71259 (2)	0.02141 (11)
O1V	0.4234 (2)	0.50716 (13)	0.77320 (10)	0.0315 (3)
O2V	0.0597 (2)	0.47362 (12)	0.78294 (10)	0.0287 (3)
O1	0.3179 (2)	0.39575 (12)	0.62365 (10)	0.0271 (3)
O2	0.3574 (2)	0.33281 (14)	0.47038 (11)	0.0366 (3)
O3	0.1639 (2)	0.71802 (12)	0.72049 (9)	0.0275 (3)
O4	0.0581 (2)	0.91366 (12)	0.64817 (11)	0.0314 (3)
N1	0.1910 (2)	0.61833 (13)	0.56431 (11)	0.0198 (3)
C1	0.3060 (3)	0.41282 (17)	0.52667 (14)	0.0253 (4)
C2	0.2222 (2)	0.54682 (17)	0.48962 (13)	0.0226 (4)
C3	0.1809 (3)	0.59740 (19)	0.39317 (14)	0.0281 (4)
H3A	0.2057	0.5470	0.3396	0.034*
C4	0.1022 (3)	0.7240 (2)	0.37720 (14)	0.0317 (4)
H4A	0.0684	0.7603	0.3122	0.038*
C5	0.0721 (3)	0.79869 (18)	0.45546 (14)	0.0275 (4)
H5A	0.0199	0.8859	0.4448	0.033*
C6	0.1212 (2)	0.74101 (17)	0.54953 (13)	0.0224 (4)
C7	0.1099 (3)	0.80042 (17)	0.64506 (14)	0.0234 (4)
N1A	0.3456 (2)	0.04478 (13)	1.20580 (11)	0.0196 (3)
H1AA	0.3654	−0.0387	1.2212	0.024*
N2A	0.2998 (2)	−0.11965 (13)	1.07341 (11)	0.0200 (3)
C1A	0.3719 (3)	0.11637 (17)	1.27465 (13)	0.0228 (4)
C2A	0.3398 (3)	0.24933 (18)	1.25092 (14)	0.0264 (4)
H2AA	0.3576	0.3023	1.2986	0.032*

C3A	0.2831 (3)	0.30303 (17)	1.15923 (14)	0.0253 (4)
H3AA	0.2614	0.3933	1.1438	0.030*
C4A	0.2563 (2)	0.22627 (16)	1.08750 (13)	0.0212 (3)
C5A	0.2000 (3)	0.27714 (17)	0.98954 (14)	0.0240 (4)
H5AA	0.1763	0.3669	0.9714	0.029*
C6A	0.1804 (3)	0.19858 (17)	0.92258 (13)	0.0239 (4)
H6AA	0.1436	0.2342	0.8580	0.029*
C7A	0.2141 (2)	0.06288 (17)	0.94763 (13)	0.0206 (3)
C8A	0.1947 (2)	−0.02266 (18)	0.88038 (13)	0.0242 (4)
H8AA	0.1590	0.0090	0.8149	0.029*
C9A	0.2276 (3)	−0.15098 (18)	0.91015 (14)	0.0249 (4)
H9AA	0.2154	−0.2090	0.8653	0.030*
C10A	0.2799 (2)	−0.19738 (17)	1.00801 (14)	0.0224 (4)
C11A	0.2681 (2)	0.00837 (16)	1.04333 (12)	0.0185 (3)
C12A	0.2899 (2)	0.09300 (16)	1.11330 (13)	0.0193 (3)
C13A	0.4363 (3)	0.05058 (19)	1.37274 (14)	0.0307 (4)
H13A	0.3685	−0.0242	1.3960	0.046*
H13B	0.4086	0.1095	1.4233	0.046*
H13C	0.5740	0.0234	1.3633	0.046*
C14A	0.3109 (3)	−0.33859 (17)	1.04256 (15)	0.0279 (4)
H14A	0.4198	−0.3582	1.0822	0.042*
H14B	0.3369	−0.3843	0.9836	0.042*
H14C	0.1962	−0.3653	1.0844	0.042*
O1S	0.5167 (2)	0.20247 (13)	0.73847 (10)	0.0354 (4)
H1S	0.4510	0.2544	0.6982	0.053*
H2S	0.5160	0.2402	0.7897	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.02635 (19)	0.01819 (16)	0.02035 (16)	0.00025 (12)	−0.00783 (12)	−0.00297 (11)
O1V	0.0338 (8)	0.0301 (7)	0.0333 (7)	−0.0017 (6)	−0.0154 (6)	−0.0052 (6)
O2V	0.0318 (8)	0.0257 (7)	0.0278 (7)	−0.0021 (6)	−0.0038 (6)	−0.0020 (5)
O1	0.0326 (7)	0.0213 (6)	0.0281 (7)	0.0028 (5)	−0.0091 (6)	−0.0064 (5)
O2	0.0482 (9)	0.0301 (7)	0.0334 (7)	−0.0019 (7)	−0.0013 (7)	−0.0154 (6)
O3	0.0386 (8)	0.0202 (6)	0.0246 (6)	0.0012 (5)	−0.0092 (6)	−0.0054 (5)
O4	0.0360 (8)	0.0195 (6)	0.0380 (7)	0.0012 (6)	−0.0054 (6)	−0.0046 (5)
N1	0.0191 (7)	0.0201 (7)	0.0214 (7)	−0.0036 (6)	−0.0049 (6)	−0.0032 (5)
C1	0.0242 (9)	0.0250 (9)	0.0283 (9)	−0.0043 (7)	−0.0031 (7)	−0.0075 (7)
C2	0.0199 (9)	0.0254 (9)	0.0244 (8)	−0.0067 (7)	−0.0018 (7)	−0.0062 (7)
C3	0.0279 (10)	0.0358 (10)	0.0227 (9)	−0.0096 (8)	−0.0033 (7)	−0.0051 (7)
C4	0.0318 (11)	0.0405 (11)	0.0233 (9)	−0.0092 (9)	−0.0099 (8)	0.0042 (8)
C5	0.0260 (10)	0.0262 (9)	0.0294 (9)	−0.0035 (7)	−0.0071 (8)	0.0023 (7)
C6	0.0178 (9)	0.0213 (8)	0.0281 (9)	−0.0040 (7)	−0.0033 (7)	−0.0015 (7)
C7	0.0210 (9)	0.0217 (8)	0.0279 (9)	−0.0024 (7)	−0.0036 (7)	−0.0037 (7)
N1A	0.0195 (7)	0.0168 (7)	0.0226 (7)	−0.0020 (5)	−0.0025 (6)	−0.0031 (5)
N2A	0.0169 (7)	0.0185 (7)	0.0245 (7)	−0.0010 (5)	−0.0023 (6)	−0.0036 (5)
C1A	0.0190 (9)	0.0273 (9)	0.0232 (8)	−0.0037 (7)	−0.0009 (7)	−0.0073 (7)

C2A	0.0261 (10)	0.0247 (9)	0.0307 (9)	−0.0048 (7)	−0.0007 (7)	−0.0116 (7)
C3A	0.0226 (9)	0.0187 (8)	0.0343 (10)	−0.0018 (7)	0.0008 (7)	−0.0067 (7)
C4A	0.0169 (8)	0.0188 (8)	0.0270 (9)	−0.0009 (6)	−0.0002 (7)	−0.0034 (7)
C5A	0.0196 (9)	0.0187 (8)	0.0312 (9)	−0.0005 (7)	−0.0022 (7)	0.0018 (7)
C6A	0.0192 (9)	0.0266 (9)	0.0229 (8)	0.0000 (7)	−0.0028 (7)	0.0034 (7)
C7A	0.0143 (8)	0.0248 (9)	0.0215 (8)	−0.0009 (7)	−0.0008 (6)	−0.0020 (7)
C8A	0.0182 (9)	0.0340 (10)	0.0203 (8)	−0.0026 (7)	−0.0014 (7)	−0.0048 (7)
C9A	0.0183 (9)	0.0308 (9)	0.0273 (9)	−0.0030 (7)	0.0003 (7)	−0.0117 (7)
C10A	0.0156 (8)	0.0233 (9)	0.0290 (9)	−0.0023 (7)	−0.0001 (7)	−0.0084 (7)
C11A	0.0135 (8)	0.0202 (8)	0.0211 (8)	−0.0008 (6)	−0.0006 (6)	−0.0032 (6)
C12A	0.0148 (8)	0.0204 (8)	0.0221 (8)	−0.0019 (6)	−0.0014 (6)	−0.0024 (6)
C13A	0.0365 (11)	0.0322 (10)	0.0254 (9)	−0.0052 (8)	−0.0079 (8)	−0.0059 (8)
C14A	0.0260 (10)	0.0214 (9)	0.0376 (10)	−0.0017 (7)	−0.0045 (8)	−0.0086 (7)
O1S	0.0530 (10)	0.0245 (7)	0.0245 (7)	0.0098 (6)	−0.0066 (6)	−0.0009 (5)

Geometric parameters (Å, °)

V1—O2V	1.6158 (14)	C2A—C3A	1.367 (3)
V1—O1V	1.6228 (14)	C2A—H2AA	0.9500
V1—O3	1.9842 (13)	C3A—C4A	1.407 (2)
V1—O1	2.0132 (13)	C3A—H3AA	0.9500
V1—N1	2.1107 (15)	C4A—C12A	1.407 (2)
O1—C1	1.304 (2)	C4A—C5A	1.435 (2)
O2—C1	1.216 (2)	C5A—C6A	1.356 (3)
O3—C7	1.300 (2)	C5A—H5AA	0.9500
O4—C7	1.220 (2)	C6A—C7A	1.431 (2)
N1—C6	1.330 (2)	C6A—H6AA	0.9500
N1—C2	1.333 (2)	C7A—C11A	1.410 (2)
C1—C2	1.508 (3)	C7A—C8A	1.414 (2)
C2—C3	1.382 (3)	C8A—C9A	1.364 (3)
C3—C4	1.385 (3)	C8A—H8AA	0.9500
C3—H3A	0.9500	C9A—C10A	1.415 (3)
C4—C5	1.396 (3)	C9A—H9AA	0.9500
C4—H4A	0.9500	C10A—C14A	1.503 (2)
C5—C6	1.388 (3)	C11A—C12A	1.439 (2)
C5—H5A	0.9500	C13A—H13A	0.9800
C6—C7	1.509 (2)	C13A—H13B	0.9800
N1A—C1A	1.336 (2)	C13A—H13C	0.9800
N1A—C12A	1.361 (2)	C14A—H14A	0.9800
N1A—H1AA	0.8800	C14A—H14B	0.9800
N2A—C10A	1.333 (2)	C14A—H14C	0.9800
N2A—C11A	1.362 (2)	O1S—H1S	0.8500
C1A—C2A	1.401 (3)	O1S—H2S	0.8500
C1A—C13A	1.496 (3)		
O2V—V1—O1V	109.64 (7)	C1A—C2A—H2AA	119.9
O2V—V1—O3	101.07 (6)	C2A—C3A—C4A	120.83 (16)
O1V—V1—O3	99.96 (6)	C2A—C3A—H3AA	119.6
O2V—V1—O1	99.69 (6)	C4A—C3A—H3AA	119.6
O1V—V1—O1	96.56 (6)	C3A—C4A—C12A	117.84 (16)

O3—V1—O1	147.22 (6)	C3A—C4A—C5A	123.32 (16)
O2V—V1—N1	117.60 (6)	C12A—C4A—C5A	118.82 (16)
O1V—V1—N1	132.68 (7)	C6A—C5A—C4A	120.78 (16)
O3—V1—N1	74.31 (5)	C6A—C5A—H5AA	119.6
O1—V1—N1	73.66 (5)	C4A—C5A—H5AA	119.6
C1—O1—V1	123.69 (12)	C5A—C6A—C7A	121.08 (16)
C7—O3—V1	123.61 (11)	C5A—C6A—H6AA	119.5
C6—N1—C2	121.89 (15)	C7A—C6A—H6AA	119.5
C6—N1—V1	118.44 (11)	C11A—C7A—C8A	116.76 (16)
C2—N1—V1	119.56 (12)	C11A—C7A—C6A	120.21 (16)
O2—C1—O1	125.46 (18)	C8A—C7A—C6A	123.02 (16)
O2—C1—C2	122.44 (17)	C9A—C8A—C7A	119.59 (16)
O1—C1—C2	112.09 (15)	C9A—C8A—H8AA	120.2
N1—C2—C3	121.01 (17)	C7A—C8A—H8AA	120.2
N1—C2—C1	110.77 (15)	C8A—C9A—C10A	119.85 (16)
C3—C2—C1	128.21 (16)	C8A—C9A—H9AA	120.1
C2—C3—C4	117.85 (17)	C10A—C9A—H9AA	120.1
C2—C3—H3A	121.1	N2A—C10A—C9A	122.26 (16)
C4—C3—H3A	121.1	N2A—C10A—C14A	117.51 (16)
C3—C4—C5	120.81 (17)	C9A—C10A—C14A	120.22 (16)
C3—C4—H4A	119.6	N2A—C11A—C7A	123.74 (15)
C5—C4—H4A	119.6	N2A—C11A—C12A	118.19 (15)
C6—C5—C4	117.57 (17)	C7A—C11A—C12A	118.07 (15)
C6—C5—H5A	121.2	N1A—C12A—C4A	118.81 (15)
C4—C5—H5A	121.2	N1A—C12A—C11A	120.16 (15)
N1—C6—C5	120.82 (16)	C4A—C12A—C11A	121.03 (15)
N1—C6—C7	110.84 (15)	C1A—C13A—H13A	109.5
C5—C6—C7	128.34 (16)	C1A—C13A—H13B	109.5
O4—C7—O3	125.37 (17)	H13A—C13A—H13B	109.5
O4—C7—C6	122.36 (17)	C1A—C13A—H13C	109.5
O3—C7—C6	112.26 (15)	H13A—C13A—H13C	109.5
C1A—N1A—C12A	124.06 (15)	H13B—C13A—H13C	109.5
C1A—N1A—H1AA	118.0	C10A—C14A—H14A	109.5
C12A—N1A—H1AA	118.0	C10A—C14A—H14B	109.5
C10A—N2A—C11A	117.80 (15)	H14A—C14A—H14B	109.5
N1A—C1A—C2A	118.35 (16)	C10A—C14A—H14C	109.5
N1A—C1A—C13A	118.39 (16)	H14A—C14A—H14C	109.5
C2A—C1A—C13A	123.25 (16)	H14B—C14A—H14C	109.5
C3A—C2A—C1A	120.10 (16)	H1S—O1S—H2S	105.4
C3A—C2A—H2AA	119.9		
O2V—V1—O1—C1	114.46 (15)	C5—C6—C7—O4	−2.4 (3)
O1V—V1—O1—C1	−134.26 (15)	N1—C6—C7—O3	−2.3 (2)
O3—V1—O1—C1	−14.2 (2)	C5—C6—C7—O3	178.65 (18)
N1—V1—O1—C1	−1.63 (14)	C12A—N1A—C1A—C2A	0.2 (3)
O2V—V1—O3—C7	−110.14 (15)	C12A—N1A—C1A—C13A	−179.04 (16)
O1V—V1—O3—C7	137.39 (15)	N1A—C1A—C2A—C3A	0.0 (3)
O1—V1—O3—C7	18.2 (2)	C13A—C1A—C2A—C3A	179.25 (17)
N1—V1—O3—C7	5.67 (14)	C1A—C2A—C3A—C4A	−0.1 (3)
O2V—V1—N1—C6	87.64 (14)	C2A—C3A—C4A—C12A	0.0 (3)

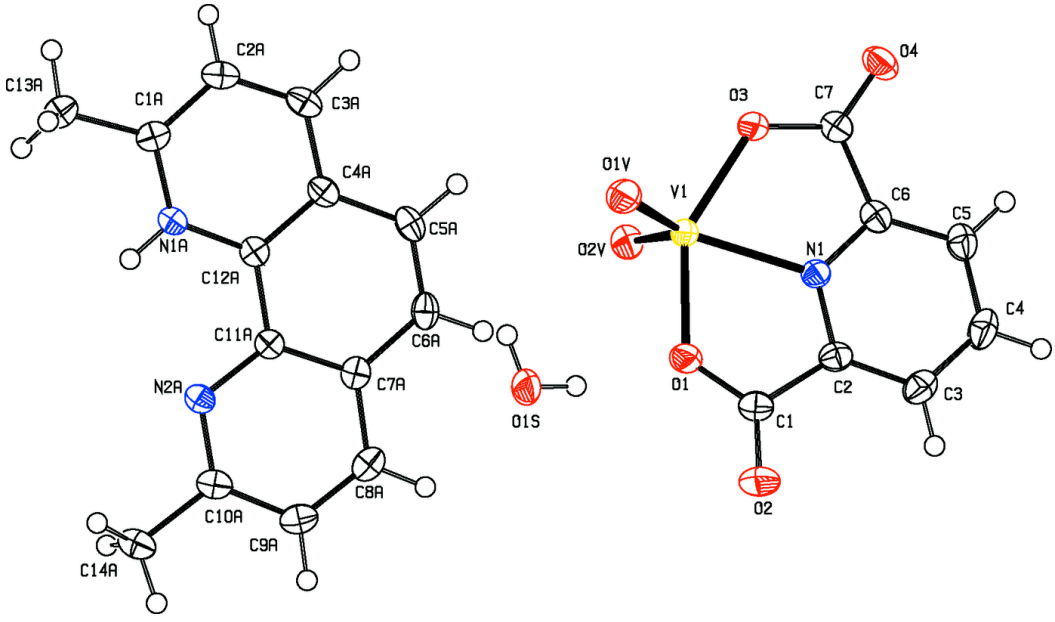
O1V—V1—N1—C6	−96.02 (14)	C2A—C3A—C4A—C5A	−178.93 (17)
O3—V1—N1—C6	−6.85 (12)	C3A—C4A—C5A—C6A	178.56 (17)
O1—V1—N1—C6	−179.80 (14)	C12A—C4A—C5A—C6A	−0.4 (3)
O2V—V1—N1—C2	−88.52 (14)	C4A—C5A—C6A—C7A	0.3 (3)
O1V—V1—N1—C2	87.82 (15)	C5A—C6A—C7A—C11A	0.2 (3)
O3—V1—N1—C2	176.99 (14)	C5A—C6A—C7A—C8A	179.76 (17)
O1—V1—N1—C2	4.04 (13)	C11A—C7A—C8A—C9A	0.2 (2)
V1—O1—C1—O2	178.41 (15)	C6A—C7A—C8A—C9A	−179.36 (16)
V1—O1—C1—C2	−0.6 (2)	C7A—C8A—C9A—C10A	0.3 (3)
C6—N1—C2—C3	−0.7 (3)	C11A—N2A—C10A—C9A	0.0 (2)
V1—N1—C2—C3	175.31 (13)	C11A—N2A—C10A—C14A	−178.67 (15)
C6—N1—C2—C1	178.57 (15)	C8A—C9A—C10A—N2A	−0.4 (3)
V1—N1—C2—C1	−5.41 (19)	C8A—C9A—C10A—C14A	178.24 (16)
O2—C1—C2—N1	−175.26 (18)	C10A—N2A—C11A—C7A	0.5 (2)
O1—C1—C2—N1	3.8 (2)	C10A—N2A—C11A—C12A	−179.80 (15)
O2—C1—C2—C3	4.0 (3)	C8A—C7A—C11A—N2A	−0.6 (2)
O1—C1—C2—C3	−176.96 (18)	C6A—C7A—C11A—N2A	178.95 (15)
N1—C2—C3—C4	−1.4 (3)	C8A—C7A—C11A—C12A	179.72 (15)
C1—C2—C3—C4	179.45 (17)	C6A—C7A—C11A—C12A	−0.7 (2)
C2—C3—C4—C5	2.2 (3)	C1A—N1A—C12A—C4A	−0.4 (3)
C3—C4—C5—C6	−0.9 (3)	C1A—N1A—C12A—C11A	179.01 (16)
C2—N1—C6—C5	2.1 (3)	C3A—C4A—C12A—N1A	0.2 (2)
V1—N1—C6—C5	−173.99 (13)	C5A—C4A—C12A—N1A	179.21 (15)
C2—N1—C6—C7	−177.03 (15)	C3A—C4A—C12A—C11A	−179.14 (15)
V1—N1—C6—C7	6.90 (18)	C5A—C4A—C12A—C11A	−0.2 (2)
C4—C5—C6—N1	−1.2 (3)	N2A—C11A—C12A—N1A	1.6 (2)
C4—C5—C6—C7	177.70 (17)	C7A—C11A—C12A—N1A	−178.66 (15)
V1—O3—C7—O4	177.30 (14)	N2A—C11A—C12A—C4A	−179.01 (15)
V1—O3—C7—C6	−3.7 (2)	C7A—C11A—C12A—C4A	0.7 (2)
N1—C6—C7—O4	176.67 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1A—H1AA···O1S^{i}	0.88	1.87	2.714 (2)	162
O1S—H1S···O1	0.85	1.89	2.728 (2)	168
O1S—H2S···N2A^{i}	0.85	2.49	2.964 (2)	116
C2A—H2AA···O2^{ii}	0.95	2.39	3.241 (2)	149
C5—H5···O4^{iii}	0.950	2.34	3.238 (2)	157

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

Fig. 1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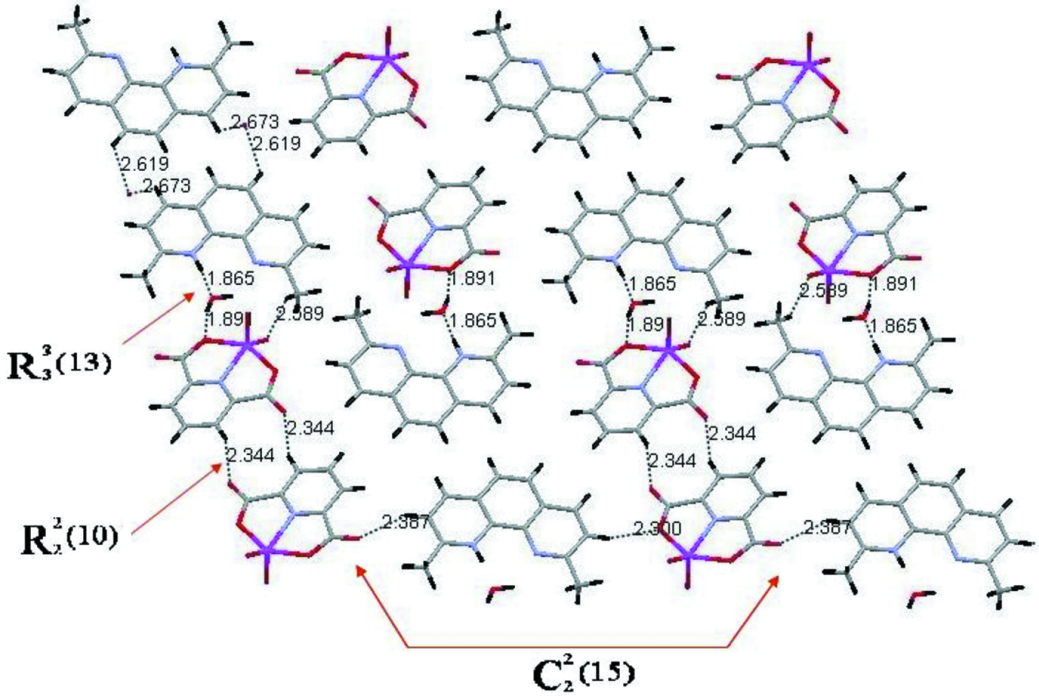
C13A
C1A
C3A
C4A
C5A
N1A
C12A
C11A
C6A
N2A
C7A
O1S
C8A
C10A
C9A
C14A
O4
O3
C7
O1V
V1
C6
C5
O2V
N1
C4
O1
C2
C3
C1
O2

Fig. 2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2.619
2.619
2.673
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2.589
1.891
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1.865
1.89
2.589
2.344
2.344
2.344
2.344
2.387
2.300
2.387
$R_3^3(13)$
$R_2^2(10)$
$C_2^2(15)$