Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Piperazinium aquabis(oxalato)oxidovanadate(IV) dihydrate

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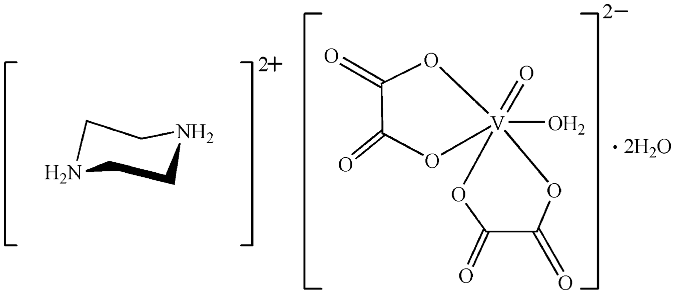
Received 30 April 2007; accepted 14 May 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.103; data-to-parameter ratio = 24.0.

The title compound, $(C_4H_{12}N_2)[V(C_2O_4)_2O(H_2O)]\cdot 2H_2O$, has been obtained by the reaction of vanadium(III) chloride with piperazinediium oxalate in aqueous solution. It shows distorted octahedral geometry [V=O = 1.6010 (12) Å and V—O = 1.9932 (12)–2.2176 (12) Å]]. The structure exhibits intermolecular N—H···O and C—H···O hydrogen bonds. The uncoordinated water molecules contribute to the formation of the supramolecular structure *via* O—H···O hydrogen bonds.

Related literature

The geometry of the three-dimensional network formed by hydrogen-bonded cations and anions is very similar to that observed in piperazinium aquabis(oxalato)oxovanadate(IV) sesquihydrate (Lin *et al.*, 2004). New proton-transfer compounds, which contain pyridine-2,6-dicarboxylic acid ($pydcH_2$), pyridine-2,6-diamine (pyda) and creatine, are discussed by Aghabozorg *et al.* (2005), Moghimi *et al.* (2005) and Soleimannejad *et al.* (2005). A five-coordinate vanadium(V) complex with a (pydcH)(pydaH) proton-transfer compound was previously reported by Ranjbar (2004). Another five-coordinate self-assembled complex, (dmpH)-$[V(pydc)(O)_2]\cdot H_2O$ (dmp is 2,9-dimethyl-1,10-phenanthroline) was reported by Aghabozorg & Sadr-khanlou (2007).



Experimental

Crystal data

$(C_4H_{12}N_2)[V(C_2O_4)_2O(H_2O)]\cdot 2H_2O$
M_r = 385.18
Monoclinic, $P2_1/n$
a = 10.0302 (6) Å
b = 12.4995 (8) Å
c = 12.4665 (8) Å
β = 111.320 (1)°
V = 1455.99 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.75 mm^{-1}
T = 100 (2) K
0.60 × 0.12 × 0.06 mm

Data collection

Bruker SMART APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.662, T_{max} = 0.956
16128 measured reflections
4983 independent reflections
3891 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.02
4983 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O5	0.92	1.92	2.787 (2)	157
N1—H1*B*···O6^{i}	0.92	1.91	2.818 (2)	170
N2—H2*A*···O1^{ii}	0.92	1.87	2.772 (2)	166
N2—H2*B*···O2^{iii}	0.92	1.89	2.805 (2)	170
O10—H10*A*···O2^{iv}	0.85	1.83	2.674 (2)	172
O10—H10*B*···O1*W*	0.85	1.77	2.607 (2)	172
O1*W*—H1*WA*···O2*W*	0.85	1.93	2.745 (2)	160
O1*W*—H1*WB*···O4^{v}	0.85	1.88	2.720 (2)	170
O2*W*—H2*WA*···O6^{iv}	0.85	2.20	3.004 (2)	158
O2*W*—H2*WB*···O8^{vi}	0.85	2.11	2.911 (2)	156
C6—H6*A*···O9^{vii}	0.99	2.53	3.192 (2)	124
C6—H6*B*···O4^{iii}	0.99	2.48	3.087 (2)	119
C8—H8*B*···O9^{viii}	0.99	2.58	3.442 (2)	146

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x-1, y, z$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (v) $-x+1, -y+2, -z+1$; (vi) $-x+2, -y+2, -z+2$; (vii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (viii) $-x+1, -y+2, -z+2$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

doi:10.1107/S1600536807023616

The financial support of Azad University, Qom Branch, and the Teacher Training University, Tehran, is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2241).

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supplementary materials

Acta Cryst. (2007). E**63**, m1754-m1755 [**doi:10.1107/S1600536807023616**]

Piperazinium aquabis(oxalato)oxidovanadate(IV) dihydrate

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Comment

Hydrogen bonding plays a key role in chemical, catalytic and biochemical processes, as well as in supramolecular chemistry and crystal engineering. In order to study the hydrogen-bonding patterns in proton-transfer compounds, our research group has selected pyridine-2,6-dicarboxylic acid (pydcH_2) and 1,10-phenanthroline-2,9-dicarboxylic acid (phendcH_2) as proton donors, and pyridine-2,6-diamine (pyda), creatinine (creat) and 1,10-phenanthroline (phen) as proton acceptors. This has resulted in the formation of new proton-transfer systems, such as (pydaH)(pydcH) (Aghabozorg *et al.*, 2005), (creatH)(pydcH) (Moghimi *et al.*, 2005) and (creatH)(phendcH) (Soleimannejad *et al.*, 2005).

The molecular structure of the title compound, (I), is iven in Fig. 1. The asymmetric unit of the $(\text{pipzH}_2)[\text{V(O)(ox)}_2]\cdot 2H_2O$ compound contains $[\text{V(O)(ox)}_2]^{2-}$ complex anions, two water molecules and $(\text{pipzH}_2)^{2+}$ cations.

In this article, our goal was the generation of the self-assembling coordination compound using a self-assembling ligand. The structure of the V(IV) complex $[VO(C_2O_4)_2(H_2O)]^{2-}[C_4H_{12}N_2]^{2+}\cdot 1.5H_2O$, was reported recently (Lin *et al.*, 2004), but the goal and method of synthesis were completely different from ours. The data collection temperature in our work was 100 K, whereas, the previous work was conducted at room temperature. In the present study the measured reflections was 3891, while in the earlier work it was 2183. It is necessary to mention that the structure of the V(IV) complex $[VO(C_2O_4)_2(H_2O)]^{2-}[C_4H_{12}N_2]^{2+}\cdot 2H_2O$, (I), contains two uncoordinated water molecules without any disordering atoms but in the previous work, all uncoordinated water molecules were disorder. Therefore, The uncoordinated water molecules contribute to the formation of supramolecular structure *via* O—H···O hydrogen bonds.

The V1—O9 bond length [1.6014 (12) Å] is shorter than the other V—O bond lengths probably due to the formation of double bond. O1 and O9 atoms occupy the axial positions, [O1—V1—O9 is 170.19 (5)°], while O3, O5, O7 and O10 atoms form the equatorial plane. Therefore the coordination around the central atom is distorted octahedral. The O1—V1—O7—C3 and O5—V1—O3—C2 torsion angles are −93.97 (11)° and −84.46 (12)°, respectively, indicating that two dianionic $(\text{ox})^{2-}$ units are almost perpendicular to each other. A remarkable feature in the crystal structure of compound (I) is the presence of O—H···O, O—H···N and C—H···O hydrogen bonds with D···A distances ranging from 2.60 (2) Å to 3.442 (2) Å (Table 1). Hydrogen bonds result in the formation of a supramolecular structure (Fig. 2). Ion pairing and van der Waals interactions are also effective in the packing.

Experimental

A solution of VCl_3 (80 mg, 0.5 mmol) in water (20 ml) was added to an aqueous solution of piperazinediium oxalate (176 mg, 1.0 mmol in water (20 ml) in a 2:1 molar ratio. Blue crystals of (I) were obtained after a few days at room temperature.

Refinement

All hydrogen atoms were found in difference Fourier synthesis, placed in idealized positions (C—H 0.99 Å, N—H 0.92 Å, O—H 0.85 Å) and refined in isotropic approximatiom as riding with U_{iso}(H) = 1.2 U_{eq}(parent atom). The highest residual peak [1.26 e $Å^{-3}$] is situated 0.83 Å at atom V1.

Figures

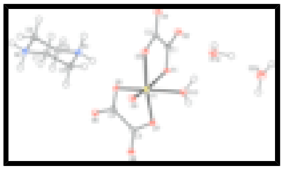

Fig. 1.
The asymmetric unit of $(pipzH_2)[V(O)(ox)_2]\cdot 2H_2O$, with displacement ellipsoids drawn at the 50% probability level.

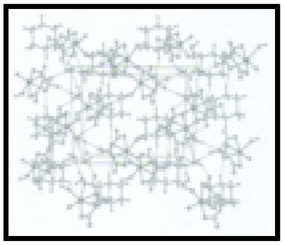

Fig. 2
The crystal packing of $(pipzH_2)[V(O)(ox)_2]\cdot 2H_2O$, hydrogen bonds are shown as dashed lines.

Piperazinium aquabis(oxalato)oxidovanadate(IV) dihydrate

Crystal data

$(C_4H_{12}N_2)[V(C_2O_4)_2O(H_2O)]\cdot 2H_2O$	F_{000} = 796
M_r = 385.18	D_x = 1.757 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3837 reflections
a = 10.0302 (6) Å	θ = 2.3–32.9º
b = 12.4995 (8) Å	μ = 0.75 mm^{-1}
c = 12.4665 (8) Å	T = 100 (2) K
β = 111.3200 (10)º	Plate, blue
V = 1455.99 (16) $Å^3$	0.60 × 0.12 × 0.06 mm
Z = 4	

Data collection

Bruker SMART APEX II CCD area-detector diffractometer	4983 independent reflections
Radiation source: fine-focus sealed tube	3891 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 100(2) K	θ_{max} = 32.0º
ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan	$h = -14 \rightarrow 14$

(APEX2; Bruker, 2005)
$T_{\min} = 0.662$, $T_{\max} = 0.956$
16128 measured reflections

$k = -18 \rightarrow 17$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.103$
$S = 1.02$
4983 reflections
208 parameters
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.8P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.75$ e Å^{-3}
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 $F^2 > 2\text{sigma}(F^2)$ 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*	$U_{\text{iso}}*/U_{\text{eq}}$
V1	0.61163 (3)	0.93971 (2)	0.85255 (2)	0.00815 (7)
O1	0.69720 (12)	0.80761 (10)	0.77764 (10)	0.0112 (2)
O2	0.64764 (13)	0.71346 (10)	0.61456 (10)	0.0135 (2)
O3	0.46500 (12)	0.92058 (9)	0.69221 (10)	0.0118 (2)
O4	0.40975 (13)	0.84269 (10)	0.52017 (10)	0.0140 (2)
O5	0.53646 (12)	0.81507 (9)	0.91695 (10)	0.0109 (2)
O6	0.58704 (13)	0.71576 (10)	1.07578 (10)	0.0138 (2)
O7	0.78005 (12)	0.91467 (9)	0.99668 (10)	0.0111 (2)
O8	0.83600 (12)	0.83993 (10)	1.17084 (10)	0.0136 (2)
O9	0.52951 (13)	1.03699 (10)	0.88501 (10)	0.0139 (2)
O10	0.74368 (13)	1.02457 (10)	0.79444 (10)	0.0133 (2)
H10A	0.7735	1.0872	0.8175	0.016*
H10B	0.7438	1.0175	0.7266	0.016*
C1	0.62181 (16)	0.78455 (13)	0.67431 (14)	0.0099 (3)
C2	0.48584 (16)	0.85416 (13)	0.62224 (13)	0.0098 (3)
C3	0.75798 (16)	0.85130 (13)	1.06993 (13)	0.0097 (3)

C4	0.61588 (16)	0.78749 (13)	1.01984 (13)	0.0099 (3)
N1	0.24180 (15)	0.78431 (11)	0.81510 (12)	0.0111 (3)
H1A	0.3395	0.7798	0.8356	0.013*
H1B	0.2015	0.7859	0.7360	0.013*
N2	−0.00704 (14)	0.79569 (11)	0.87667 (12)	0.0112 (3)
H2A	−0.1047	0.8001	0.8562	0.013*
H2B	0.0334	0.7932	0.9557	0.013*
C5	0.18936 (18)	0.68788 (14)	0.85840 (15)	0.0142 (3)
H5A	0.2377	0.6827	0.9430	0.017*
H5B	0.2122	0.6226	0.8235	0.017*
C6	0.02939 (18)	0.69603 (14)	0.82766 (15)	0.0141 (3)
H6A	−0.0191	0.6964	0.7429	0.017*
H6B	−0.0050	0.6330	0.8583	0.017*
C7	0.04559 (18)	0.89219 (14)	0.83438 (15)	0.0137 (3)
H7A	0.0239	0.9569	0.8708	0.016*
H7B	−0.0044	0.8986	0.7500	0.016*
C8	0.20546 (17)	0.88496 (13)	0.86239 (15)	0.0126 (3)
H8A	0.2375	0.9473	0.8291	0.015*
H8B	0.2561	0.8867	0.9469	0.015*
O1W	0.74471 (15)	0.98447 (11)	0.58954 (12)	0.0207 (3)
H1WA	0.8212	1.0015	0.5792	0.025*
H1WB	0.6957	1.0349	0.5479	0.025*
O2W	1.01439 (16)	1.05116 (12)	0.61212 (13)	0.0271 (3)
H2WA	0.9990	1.0873	0.5510	0.033*
H2WB	1.0365	1.0950	0.6680	0.033*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.00938 (12)	0.00690 (12)	0.00767 (12)	0.00021 (9)	0.00249 (9)	−0.00027 (9)
O1	0.0105 (5)	0.0120 (5)	0.0095 (5)	0.0015 (4)	0.0016 (4)	−0.0012 (4)
O2	0.0161 (5)	0.0123 (6)	0.0107 (5)	0.0052 (4)	0.0031 (4)	−0.0010 (4)
O3	0.0129 (5)	0.0104 (5)	0.0105 (5)	0.0030 (4)	0.0021 (4)	−0.0013 (4)
O4	0.0146 (5)	0.0152 (6)	0.0094 (5)	0.0032 (5)	0.0010 (4)	−0.0013 (4)
O5	0.0100 (5)	0.0115 (5)	0.0098 (5)	−0.0017 (4)	0.0020 (4)	0.0007 (4)
O6	0.0165 (5)	0.0118 (6)	0.0124 (5)	−0.0026 (4)	0.0045 (4)	0.0013 (4)
O7	0.0115 (5)	0.0102 (5)	0.0100 (5)	−0.0020 (4)	0.0019 (4)	0.0009 (4)
O8	0.0137 (5)	0.0155 (6)	0.0095 (5)	−0.0007 (4)	0.0017 (4)	0.0012 (4)
O9	0.0182 (6)	0.0126 (6)	0.0113 (5)	0.0031 (5)	0.0057 (4)	−0.0007 (4)
O10	0.0182 (6)	0.0104 (5)	0.0122 (5)	−0.0039 (5)	0.0066 (4)	−0.0007 (4)
C1	0.0106 (6)	0.0085 (7)	0.0110 (7)	−0.0001 (5)	0.0043 (5)	0.0004 (5)
C2	0.0108 (6)	0.0085 (7)	0.0098 (7)	0.0003 (5)	0.0034 (5)	0.0008 (5)
C3	0.0115 (6)	0.0065 (6)	0.0116 (7)	0.0005 (5)	0.0047 (5)	−0.0009 (5)
C4	0.0108 (6)	0.0093 (7)	0.0103 (7)	−0.0009 (5)	0.0045 (5)	−0.0023 (5)
N1	0.0112 (6)	0.0112 (6)	0.0111 (6)	−0.0017 (5)	0.0042 (5)	−0.0026 (5)
N2	0.0098 (6)	0.0130 (6)	0.0105 (6)	−0.0007 (5)	0.0035 (5)	0.0009 (5)
C5	0.0152 (7)	0.0103 (7)	0.0188 (8)	0.0008 (6)	0.0082 (6)	0.0009 (6)
C6	0.0152 (7)	0.0123 (7)	0.0162 (7)	−0.0043 (6)	0.0072 (6)	−0.0028 (6)

C7	0.0131 (7)	0.0120 (8)	0.0154 (7)	0.0002 (6)	0.0046 (6)	0.0022 (6)
C8	0.0127 (7)	0.0096 (7)	0.0160 (7)	−0.0024 (6)	0.0057 (6)	−0.0032 (6)
O1W	0.0223 (6)	0.0201 (7)	0.0199 (6)	0.0026 (5)	0.0078 (5)	0.0044 (5)
O2W	0.0272 (7)	0.0257 (8)	0.0260 (7)	−0.0023 (6)	0.0067 (6)	0.0034 (6)

Geometric parameters (Å, °)

V1—O9	1.6010 (12)	N1—H1A	0.9200
V1—O7	1.9932 (12)	N1—H1B	0.9200
V1—O3	2.0194 (12)	N2—C7	1.488 (2)
V1—O5	2.0200 (12)	N2—C6	1.490 (2)
V1—O10	2.0253 (12)	N2—H2A	0.9200
V1—O1	2.2176 (12)	N2—H2B	0.9200
O1—C1	1.2672 (19)	C5—C6	1.512 (2)
O2—C1	1.2458 (19)	C5—H5A	0.9900
O3—C2	1.2753 (19)	C5—H5B	0.9900
O4—C2	1.2313 (19)	C6—H6A	0.9900
O5—C4	1.2867 (19)	C6—H6B	0.9900
O6—C4	1.2331 (19)	C7—C8	1.515 (2)
O7—C3	1.2873 (19)	C7—H7A	0.9900
O8—C3	1.2245 (19)	C7—H7B	0.9900
O10—H10A	0.8500	C8—H8A	0.9900
O10—H10B	0.8501	C8—H8B	0.9900
C1—C2	1.547 (2)	O1W—H1WA	0.8500
C3—C4	1.553 (2)	O1W—H1WB	0.8500
N1—C8	1.490 (2)	O2W—H2WA	0.8500
N1—C5	1.492 (2)	O2W—H2WB	0.8501
O9—V1—O7	103.69 (6)	C5—N1—H1A	109.3
O9—V1—O3	94.80 (6)	C8—N1—H1B	109.3
O7—V1—O3	161.31 (5)	C5—N1—H1B	109.3
O9—V1—O5	100.64 (6)	H1A—N1—H1B	107.9
O7—V1—O5	80.63 (5)	C7—N2—C6	111.16 (12)
O3—V1—O5	93.40 (5)	C7—N2—H2A	109.4
O9—V1—O10	98.98 (6)	C6—N2—H2A	109.4
O7—V1—O10	87.39 (5)	C7—N2—H2B	109.4
O3—V1—O10	92.50 (5)	C6—N2—H2B	109.4
O5—V1—O10	158.94 (5)	H2A—N2—H2B	108.0
O9—V1—O1	170.19 (5)	N1—C5—C6	109.58 (14)
O7—V1—O1	86.09 (5)	N1—C5—H5A	109.8
O3—V1—O1	75.47 (4)	C6—C5—H5A	109.8
O5—V1—O1	81.41 (5)	N1—C5—H5B	109.8
O10—V1—O1	80.55 (5)	C6—C5—H5B	109.8
C1—O1—V1	114.72 (10)	H5A—C5—H5B	108.2
C2—O3—V1	120.84 (10)	N2—C6—C5	110.38 (13)
C4—O5—V1	114.68 (10)	N2—C6—H6A	109.6
C3—O7—V1	115.35 (10)	C5—C6—H6A	109.6
V1—O10—H10A	123.9	N2—C6—H6B	109.6
V1—O10—H10B	122.6	C5—C6—H6B	109.6
H10A—O10—H10B	107.7	H6A—C6—H6B	108.1

O2—C1—O1	126.38 (15)	N2—C7—C8	110.62 (13)
O2—C1—C2	119.56 (14)	N2—C7—H7A	109.5
O1—C1—C2	114.06 (13)	C8—C7—H7A	109.5
O4—C2—O3	126.05 (15)	N2—C7—H7B	109.5
O4—C2—C1	119.26 (14)	C8—C7—H7B	109.5
O3—C2—C1	114.69 (13)	H7A—C7—H7B	108.1
O8—C3—O7	126.14 (15)	N1—C8—C7	110.49 (13)
O8—C3—C4	120.25 (14)	N1—C8—H8A	109.6
O7—C3—C4	113.60 (13)	C7—C8—H8A	109.6
O6—C4—O5	124.83 (15)	N1—C8—H8B	109.6
O6—C4—C3	121.06 (14)	C7—C8—H8B	109.6
O5—C4—C3	114.10 (13)	H8A—C8—H8B	108.1
C8—N1—C5	111.70 (13)	H1WA—O1W—H1WB	94.3
C8—N1—H1A	109.3	H2WA—O2W—H2WB	107.5

O7—V1—O1—C1	178.77 (11)	V1—O3—C2—O4	−173.53 (13)
O3—V1—O1—C1	1.87 (11)	V1—O3—C2—C1	5.61 (18)
O5—V1—O1—C1	97.68 (11)	O2—C1—C2—O4	−4.5 (2)
O10—V1—O1—C1	−93.22 (11)	O1—C1—C2—O4	175.62 (15)
O9—V1—O3—C2	174.56 (12)	O2—C1—C2—O3	176.33 (14)
O7—V1—O3—C2	−13.9 (2)	O1—C1—C2—O3	−3.6 (2)
O5—V1—O3—C2	−84.45 (12)	V1—O7—C3—O8	−165.78 (13)
O10—V1—O3—C2	75.33 (12)	V1—O7—C3—C4	13.14 (16)
O1—V1—O3—C2	−4.23 (12)	V1—O5—C4—O6	177.67 (13)
O9—V1—O5—C4	−94.07 (11)	V1—O5—C4—C3	−3.75 (16)
O7—V1—O5—C4	8.23 (11)	O8—C3—C4—O6	−8.6 (2)
O3—V1—O5—C4	170.40 (11)	O7—C3—C4—O6	172.46 (14)
O10—V1—O5—C4	64.38 (19)	O8—C3—C4—O5	172.80 (15)
O1—V1—O5—C4	95.66 (11)	O7—C3—C4—O5	−6.18 (19)
O9—V1—O7—C3	86.73 (12)	C8—N1—C5—C6	57.49 (18)
O3—V1—O7—C3	−84.57 (18)	C7—N2—C6—C5	58.11 (17)
O5—V1—O7—C3	−12.04 (11)	N1—C5—C6—N2	−57.67 (18)
O10—V1—O7—C3	−174.66 (11)	C6—N2—C7—C8	−56.69 (17)
O1—V1—O7—C3	−93.97 (11)	C5—N1—C8—C7	−56.48 (18)
V1—O1—C1—O2	−179.70 (13)	N2—C7—C8—N1	55.37 (18)
V1—O1—C1—C2	0.21 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O5	0.92	1.92	2.787 (2)	157
N1—H1B···O6^{i}	0.92	1.91	2.818 (2)	170
N2—H2A···O1^{ii}	0.92	1.87	2.772 (2)	166
N2—H2B···O2^{iii}	0.92	1.89	2.805 (2)	170
O10—H10A···O2^{iv}	0.85	1.83	2.674 (2)	172
O10—H10B···O1W	0.85	1.77	2.607 (2)	172
O1W—H1WA···O2W	0.85	1.93	2.745 (2)	160
O1W—H1WB···O4^{v}	0.85	1.88	2.720 (2)	170
O2W—H2WA···O6^{iv}	0.85	2.20	3.004 (2)	158

O2W—H2WB···O8$^{\mathrm{vi}}$	0.85	2.11	2.911 (2)	156
C6—H6A···O9$^{\mathrm{vii}}$	0.99	2.53	3.192 (2)	124
C6—H6B···O4$^{\mathrm{iii}}$	0.99	2.48	3.087 (2)	119
C8—H8B···O9$^{\mathrm{viii}}$	0.99	2.58	3.442 (2)	146

Symmetry codes: (i) *x*−1/2, −*y*+3/2, *z*−1/2; (ii) *x*−1, *y*, *z*; (iii) *x*−1/2, −*y*+3/2, *z*+1/2; (iv) −*x*+3/2, *y*+1/2, −*z*+3/2; (v) −*x*+1, −*y*+2, −*z*+1; (vi) −*x*+2, −*y*+2, −*z*+2; (vii) −*x*+1/2, *y*−1/2, −*z*+3/2; (viii) −*x*+1, −*y*+2, −*z*+2.

Fig. 1

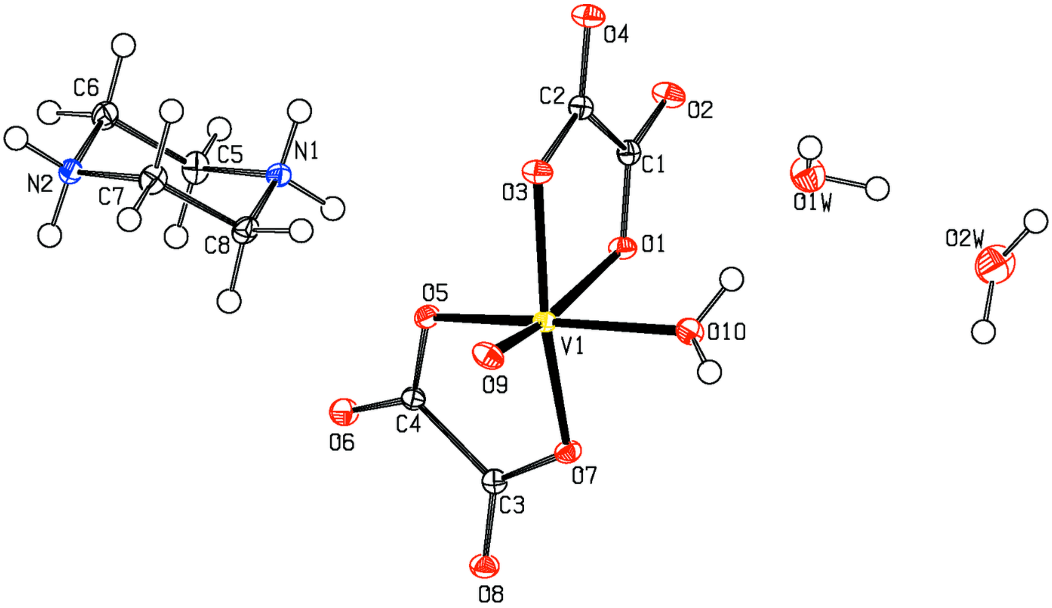

Fig. 2