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# Crystal structure of ammonium divanadium(IV,V) tellurium(IV) heptaoxide

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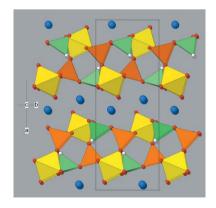
The polyhedral building blocks of the layered inorganic network in the mixed-valence title compound,  $(NH_4)(V^{IV}O_2)(V^VO_2)(TeO_3)$ , are vertex-sharing  $V^VO_4$  tetrahedra, distorted  $V^{IV}O_6$  octahedra and  $TeO_3$  pyramids, which are linked by V-O-V and V-O-Te bonds, forming double layers lying parallel to (100). The presumed  $Te^{IV}$  lone-pairs of electrons appear to be directed inwards into cavities in the double layers. The charge-balancing ammonium cations lie between the layers and probably interact with them  $via\ N-H\cdots O$  hydrogen bonds.

#### 1. Chemical context

An important feature of the crystal chemistry of tellurium(IV), electron configuration  $[Kr]4d^{10}5s^2$ , is the stereochemical activity of the  $5s^2$  lone-pair of electrons presumed to reside on the Te atom (Wells, 1962). This leads to distorted and unpredictable coordination polyhedra for the Te<sup>IV</sup> atom in the solid state (Zemann, 1968; Weber & Schleid, 2000), and its inherent asymmetry may promote the formation of noncentrosymmetric crystal structures with potentially interesting physical properties (Nguyen *et al.*, 2011). As part of our studies in this area (Johnston & Harrison, 2007), we now describe the synthesis and structure of the title mixed-valence compound,  $(NH_4)(V^{IV}O_2)(V^{V}O_2)(TeO_3)$ , (I). Some of the starting vanadium(V) was unexpectedly reduced, perhaps accompanied by oxidation of some of the ammonia.

#### 2. Structural commentary

The polyhedral building units of (I) are shown in Fig. 1. Atom V1 is bonded to four O-atom neighbours (O3<sup>i</sup>, O4, O6 and O7;





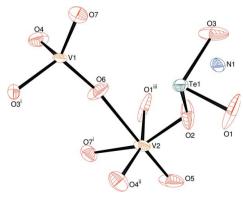


Figure 1
The asymmetric unit of (I) (50% displacement ellipsoids) expanded to show the coordination polyhedra of the V and Te atoms; see Table 1 for symmetry codes.

Table 1
Selected geometric parameters (Å, °).

8	1 ,	,	
V1-O7	1.631 (7)	V2-O1 <sup>iii</sup>	1.973 (16)
V1-O6	1.656 (5)	$V2-O7^{i}$	2.053 (7)
$V1-O3^{i}$	1.770 (5)	V2-O6	2.311 (5)
V1-O4	1.788 (9)	Te1-O1	1.748 (14)
V2-O5	1.612 (5)	Te1-O3	1.921 (5)
V2-O2	1.935 (15)	Te1-O2	1.931 (14)
V2-O4 <sup>ii</sup>	1.961 (7)		
Te1-O1-V2 <sup>iv</sup>	125.5 (9)	V1-O4-V2 <sup>vi</sup>	145.3 (4)
Te1-O2-V2	120.3 (8)	V1 - O6 - V2	167.7 (6)
$V1^v$ $-O3$ $-Te1$	131.3 (2)	$V1 - O7 - V2^{v}$	149.9 (4)

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

mean = 1.711 Å) in a distorted tetrahedral arrangement (see Table 1 for symmetry codes) The mean O-V1-O bond angle is  $109.2^{\circ}$ , although the  $O7-V1-O3^{i}$  [124.1 (7)°] and  $O3^{i}-V1-O4$  [97.0 (7)°] bond angles diverge considerably from the ideal tetrahedral value. The bond-valence-sum (BVS) values (in valence units) for V1, as calculated by the Brown & Altermatt (1985) formalism, using parameters appropriate for  $V^{IV}$  and  $V^{V}$ , are 4.96 and 5.22, respectively. Both clearly indicate a pentavalent state for this atom.

The coordination polyhedron about atom V2 is a distorted octahedron. O5 is bonded to V2 by a short 'vanadyl' V=O double bond [1.612 (5) Å], whilst O1, O4, O7 and O2 occupy the equatorial positions with V—O bond lengths between 1.93 and 2.06 Å. O6 is located *trans* to O5  $[O5-V2-O6=176.1\ (11)^\circ]$  and is consequently much farther away from the metal ion [2.311 (5) Å] than the other O atoms. This octahedral distortion mode is characteristic of both vanadium(IV) and vanadium(V) and may be theoretically analysed in terms of a second-order Jahn-Teller distortion (Kunz & Brown, 1995). The O-V2-O bond angles also show a broad spread [*cis*: 73.8 (5) to  $104.2\ (8)^\circ$ , *trans*:  $157.0\ (6)$  to  $176.1\ (11)^\circ$ ]. BVS calculations for V2 yield values of  $4.20\ (V^{IV}$  parameters) and  $4.42\ (V^{V}$  parameters), which both indicate vanadium(IV).

Te1 is three-coordinated by oxygen atoms (O1, O2 and O3) in a distorted trigonal–pyramidal arrangement [mean Te-O = 1.867 Å; BVS(Te1) = 3.98]. The O—Te—O bond angles are all less than 95°, suggesting that a treatment of the bonding about Te involving  $sp^3$  hybrid orbitals and a lone pair (as in ammonia) may be too simple (Wells, 1962). As is typical (Feger  $et\ al.$ , 1999) of the crystal chemistry of tellurium(IV), its environment includes further O atoms much closer than the Bondi (1964) van der Waals radius sum of 3.65 Å for Te and O. In particular, there is a fourth O atom within 2.70 Å [Te1—O7 $^{\text{vii}}$  = 2.695 (7) Å (vii) =  $\frac{1}{2}$  – x,  $\frac{1}{2}$  + y,  $\frac{1}{2}$  + z], which results in an overall distorted folded-square arrangement about Te1.

Assuming the presence of  $V^V$  and  $V^{IV}$  in equal amounts in the structure, the charge-balancing criterion indicates that N1 must be part of an ammonium ion (which is obviously consistent with the use of significant quantities of ammonia in the synthesis), although no H atoms could be located from the present diffraction data. However, short  $N\cdots O$  contacts in the crystal structure (*vide infra*) are indicative of hydrogen bonding. The presence of  $NH_4^+$  ions is also supported by the

Table 2 Hydrogen-bond geometry (Å).

$D-H\cdots A$ $D\cdots A$		D $ H···A$	$D \cdot \cdot \cdot A$	
$N1 \cdot \cdot \cdot O5^{vii}$	2.820 (7)	$N1 \cdots O5^{viii}$	3.15 (3)	
$N1 \cdot \cdot \cdot O1^{iii}$	2.89(2)	$N1 \cdots O1^{viii}$	3.20(2)	
$N1 \cdot \cdot \cdot O2$	2.95(2)	$N1 \cdot \cdot \cdot O5^{ix}$	3.20(3)	
$N1 \cdot \cdot \cdot O2^{viii}$	2.96(2)	N1···O3 <sup>iii</sup>	3.39 (3)	

Symmetry codes: (iii) x, y, z - 1; (vii) x, y - 1, z; (viii)  $-x, -y + 1, z - \frac{1}{2}$ ; (ix)  $-x, -y + 1, z + \frac{1}{2}$ .

IR spectrum of (I). The alternative possibilities of neutral ammonia molecules or water molecules and a different distribution of vanadium oxidation states seem far less likely to us.

### 3. Packing features

The connectivity of the VO<sub>4</sub>, VO<sub>6</sub> and TeO<sub>3</sub> polyhedra in (I) leads to a layered structure. The building blocks share vertices  $via\ V-O-V$  and V-O-Te bonds; conversely, there are no Te-O-Te links, which can occur in tellurium-rich compounds (Irvine *et al.*, 2003). Each anionic layer in (I) is constructed from two infinite (100) sheets of composition  $[(V^{IV}O_2)(V^VO_2)(TeO_3)]^-$ , built up from a network of cornersharing four- and six-membered rings (Fig. 2). The four-membered rings are built from one TeO<sub>3</sub>, one V1O<sub>4</sub> tetrahedron and two V2O<sub>6</sub> octahedra, whilst the six-membered rings are constructed from two of each different polyhedra. It is interesting to note the V-O-V inter-polyhedral angles (mean = 154.1°) are much more obtuse than the Te-O-V angles (mean = 124.0°).

The two sheets within each layer are linked through V2-O6-V1 bonds and are orientated so that the four-membered rings of one sheet are aligned with the six-membered rings of the other, and the lone-pair electrons of the  $Te^{IV}$  species point into the centre of the layer. These 'lone-pairs sandwiches' represent a novel way of accommodating the  $Te^{IV}$  lone-pairs, which may be compared to self-contained 'tubes' in  $BaTe_3O_7$ 

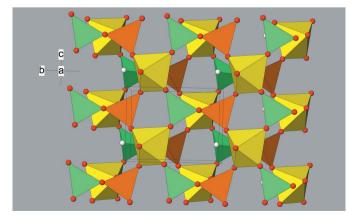


Figure 2 View approximately down [100] of part of a polyhedral layer in (I). Colour key:  $V1O_4$  tetrahedra orange,  $V2O_6$  octahedra yellow, O atoms red. The  $TeO_3$  pyramids are shown as green pseudo-tetrahedra with the presumed lone-pair of electrons shown as a white sphere.

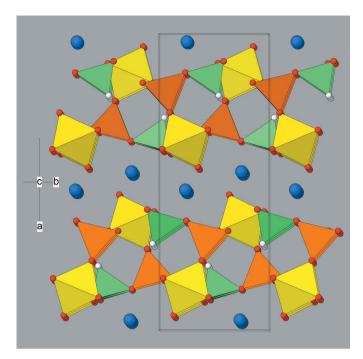


Figure 3 View approximately down [001] of the crystal structure of (I) showing the (100) polyhedral layers interspersed by ammonium ions. Colour key: N atoms blue, other atoms as in Fig. 2.

and  $BaTe_4O_9$  (Johnston & Harrison, 2002) or large 12-ring channels in  $Mg_{0.5}ZnFe(TeO_3)_3\cdot 4.5H_2O$  (Miletich, 1995).

The layers stack in the [100] direction, with the ammonium cations occupying the inter-layer regions (Fig. 3). Connectivity between the layers is presumably mediated by N-H $\cdots$ O hydrogen bonds, with N1 having eight O-atom neighbours within 3.4 Å (four in each layer). The N $\cdots$ O distances are listed in Table 2.

### 4. Database survey

A search of the Inorganic Crystal Structure Database (ICSD, 2014; web version 2.2.2) revealed three compounds containing ammonium ions, vanadium, tellurium and oxygen:  $(NH_4)_2(VO_2)[TeO_4(OH)] \cdot H_2O$  (Kim *et al.*, 2007) contains  $V^VO_4$  tetrahedra and  $Te^{VI}O_5(OH)$  octahedra, which link together into infinite chains.  $(NH_4)_2(VO_2)_2[TeO_4(OH_2)]$  (Yun *et al.*, 2010) is a layered structure containing unusual  $V^VO_5$  square pyramids and  $Te^{VI}O_4(OH_2)$  octahedra.  $(NH_4)_9K(Mo_{12}V_{12}TeO_{69})(TeO_3)_2 \cdot 27H_2O$  (Corella-Ochoa *et al.*, 2011) is a complex polyoxidometallate containing  $V^V$ ,  $V^{IV}$  and  $Te^{IV}$  atoms.

### 5. Synthesis and crystallization

0.7276~g~(4~mmol) of  $V_2O_5$  and 0.3249~g~(3~mmol) TeO $_2$  were placed in a 23 ml capacity Teflon-lined stainless steel autoclave. Added to this were 7 ml of a 1.3 M NH $_3$  solution and 8 ml of H $_2$ O (pre-oven pH = 8.5). The autoclave was sealed and heated in an oven at 438 K for three days, followed by cooling to room temperature over a few hours. The resulting

Table 3
Experimental details.

Crystal data	
Chemical formula	$(NH_4)(VO_2)(VO_2)(TeO_3)$
$M_{ m r}$	359.52
Crystal system, space group	Orthorhombic, Pna2 <sub>1</sub>
Temperature (K)	293
a, b, c (A)	18.945 (2), 7.0277 (8), 5.4402 (6)
$V(\mathring{A}^3)$	724.29 (14)
Z	4
Radiation type	Μο Κα
$\mu \text{ (mm}^{-1})$	6.52
Crystal size (mm)	$0.17 \times 0.02 \times 0.02$
Data collection	
Diffractometer	Bruker SMART1000 CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2000)
$T_{\min}$ , $T_{\max}$	0.404, 0.881
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	7528, 2368, 1595
$R_{\text{int}}$	0.047
$(\sin \theta/\lambda)_{\max} (\mathring{A}^{-1})$	0.756
(on ommax (11))	5.7.20
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.040, 0.082, 0.98
No. of reflections	2368
No. of parameters	101
No. of restraints	1
$\Delta \rho_{\rm max}$ , $\Delta \rho_{\rm min}$ (e Å <sup>-3</sup> )	0.99, -1.13
Absolute structure	Flack (1983), 1201 Friedel pairs
Absolute structure parameter	0.5 (1)
·	

Computer programs: SMART and SAINT (Bruker, 2000), SHELXS97 and SHELXL97 (Sheldrick, 2008), ORTEP-3 for Windows (Farrugia, 2012) and ATOMS (Dowty, 1999).

solid products, consisting of dark-red needles of (I), transparent chunks of  ${\rm TeO_2}$  and an unidentified yellow powder, were recovered by vacuum filtration and washing with water and acetone. IR data (KBr disk) were collected using a hand-picked sample of (I): broad bands at  $\sim 3400$  and  $3000~{\rm cm^{-1}}$  can be ascribed to the symmetric and asymmetric stretches of the tetrahedral ammonium ion (Balraj & Vidyasagar, 1998). The doublet at 1440 and 1411 cm<sup>-1</sup> is indicative of H-N-H bending modes; the presence of a doublet is in itself interesting, suggesting there may be some disorder associated with the H atoms of the ammonium cation. This phenomenon may also contribute to the difficulty in locating the H-atom positions from the X-ray data. The large number of overlapping bands in the  $1000-400~{\rm cm^{-1}}$  range can be attributed to framework V=O, V-O, Se-O and O-Se-O modes.

### 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms could not be located in difference maps, neither could they be geometrically placed. The crystal studied was found to be a racemic twin.

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# supporting information

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# Crystal structure of ammonium divanadium(IV,V) tellurium(IV) heptaoxide

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### **Computing details**

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *ATOMS* (Dowty, 1999); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

### Ammonium divanadium(IV,V) tellurium(IV) heptaoxide

Crystal data

 $(NH_4)(VO_2)_2(TeO_3)$   $M_r = 359.52$ Orthorhombic,  $Pna2_1$  a = 18.945 (2) Å b = 7.0277 (8) Å c = 5.4402 (6) Å V = 724.29 (14) Å<sup>3</sup> Z = 4F(000) = 660

Data collection

Bruker SMART1000 CCD diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2000)  $T_{min} = 0.404$ ,  $T_{max} = 0.881$ 

Refinement

Refinement on  $F^2$ Least-squares matrix: full  $R[F^2 > 2\sigma(F^2)] = 0.040$   $wR(F^2) = 0.082$  S = 0.982368 reflections 101 parameters 1 restraint Primary atom site location: structure-invariant

direct methods

Cell parameters from 5060 reflections  $\theta = 2.2-32.5^{\circ}$ 

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ Å}$ 

 $\mu = 6.52 \text{ mm}^{-1}$  T = 293 KRod, dark red  $0.17 \times 0.02 \times 0.02 \text{ mm}$ 

 $D_{\rm x} = 3.297 \; {\rm Mg \; m^{-3}}$ 

7528 measured reflections 2368 independent reflections 1595 reflections with  $I > 2\sigma(I)$ 

 $R_{\rm int}=0.047$ 

 $\theta_{\text{max}} = 32.5^{\circ}, \ \theta_{\text{min}} = 2.2^{\circ}$ 

 $h = -28 \rightarrow 26$   $k = -10 \rightarrow 10$   $l = -6 \rightarrow 8$ 

Secondary atom site location: difference Fourier

nap

Hydrogen site location: notdet  $w = 1/[\sigma^2(F_o^2) + (0.0318P)^2]$  where  $P = (F_o^2 + 2F_c^2)/3$ 

 $(\Delta/\sigma)_{\rm max} < 0.001$   $\Delta\rho_{\rm max} = 0.99 \text{ e Å}^{-3}$  $\Delta\rho_{\rm min} = -1.13 \text{ e Å}^{-3}$ 

Absolute structure: Flack (1983), 1201 Friedel

pairs

Absolute structure parameter: 0.5 (1)

Acta Cryst. (2014). E**70**, 27-30

### 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  $F^2 > \sigma(F^2)$  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 F-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters  $(\mathring{A}^2)$ 

	x	y	Z	$U_{ m iso}$ */ $U_{ m eq}$	
N1	0.0293 (3)	0.2583 (8)	0.225 (5)	0.031 (2)	
V1	0.31266 (5)	0.53486 (13)	0.2408 (12)	0.0245 (2)	
V2	0.12159 (5)	0.75777 (13)	0.2385 (9)	0.0238 (2)	
Te1	0.164135 (17)	0.50920 (5)	0.7434 (5)	0.02034 (10)	
O1	0.1064 (8)	0.561 (3)	0.985(2)	0.056 (5)	
O2	0.0970(8)	0.575 (3)	0.490(2)	0.048 (4)	
O3	0.1367 (2)	0.2463 (7)	0.728 (4)	0.053 (2)	
O4	0.3316 (4)	0.4539 (9)	-0.0638(13)	0.0355 (15)	
O5	0.0453 (3)	0.8594 (7)	0.231 (5)	0.055 (2)	
O6	0.2292 (2)	0.6033 (8)	0.224 (4)	0.043 (2)	
O7	0.3270(3)	0.3621 (9)	0.4347 (13)	0.0332 (14)	

### Atomic displacement parameters (Å<sup>2</sup>)

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.020(2)	0.021(3)	0.053 (6)	-0.004(2)	0.000(7)	0.005 (7)
V1	0.0150(4)	0.0096 (4)	0.0488 (7)	0.0017(3)	-0.003(2)	0.003(3)
V2	0.0200 (4)	0.0128 (4)	0.0386 (6)	-0.0008(3)	-0.0089 (16)	-0.003(2)
Te1	0.01691 (14)	0.01447 (16)	0.02964 (19)	0.00084 (13)	0.0000 (9)	-0.0019(7)
O1	0.017 (5)	0.099 (10)	0.052(8)	-0.022(5)	0.018 (4)	-0.054(7)
O2	0.018 (5)	0.088 (9)	0.040(8)	-0.011(5)	-0.006(4)	0.038(6)
O3	0.028(2)	0.018(2)	0.114 (7)	0.003(2)	-0.037 (7)	-0.004(8)
O4	0.043 (4)	0.029(3)	0.035(3)	0.012(3)	0.005(3)	-0.002(3)
O5	0.030(2)	0.020(2)	0.115 (6)	0.007(2)	-0.025(9)	-0.005(11)
O6	0.018(2)	0.040(3)	0.070(6)	0.0099 (19)	-0.008(6)	-0.021(7)
Ο7	0.036(3)	0.027(3)	0.037 (4)	0.003(3)	0.002(3)	0.010(3)

### Geometric parameters (Å, °)

V1—O7	1.631 (7)	V2—O6	2.311 (5)
V1—O6	1.656 (5)	Te1—O1	1.748 (14)
$V1$ — $O3^i$	1.770 (5)	Te1—O3	1.921 (5)
V1—O4	1.788 (9)	Te1—O2	1.931 (14)
V2—O5	1.612 (5)	$O1$ — $V2^{iv}$	1.973 (16)
V2—O2	1.935 (15)	O3—V1 <sup>v</sup>	1.770 (5)
V2—O4 <sup>ii</sup>	1.961 (7)	$O4$ — $V2^{vi}$	1.961 (7)

Acta Cryst. (2014). E**70**, 27-30 Sup-2

# supporting information

V2—O1 <sup>iii</sup>	1.973 (16)	O7—V2 <sup>v</sup>	2.053 (7)
V2—O7 <sup>i</sup>	2.053 (7)		
O7—V1—O6	114.3 (6)	$O1^{iii}$ — $V2$ — $O7^{i}$	75.9 (6)
O7—V1—O3 <sup>i</sup>	124.1 (7)	O5—V2—O6	176.1 (11)
O6—V1—O3 <sup>i</sup>	105.7 (3)	O2—V2—O6	85.7 (6)
O7—V1—O4	109.2 (4)	O4 <sup>ii</sup> —V2—O6	87.1 (4)
O6—V1—O4	103.4 (8)	O1 <sup>iii</sup> —V2—O6	77.0 (6)
O3 <sup>i</sup> —V1—O4	97.0 (7)	O7 <sup>i</sup> —V2—O6	73.8 (5)
O5—V2—O2	95.6 (9)	O1—Te1—O3	93.7 (9)
O5—V2—O4 <sup>ii</sup>	96.2 (5)	O1—Te1—O2	94.2 (3)
O2—V2—O4 <sup>ii</sup>	100.8 (6)	O3—Te1—O2	91.2 (7)
O5—V2—O1 <sup>iii</sup>	99.2 (8)	Te1—O1—V2 <sup>iv</sup>	125.5 (9)
O2—V2—O1 <sup>iii</sup>	89.7 (3)	Te1—O2—V2	120.3 (8)
O4 <sup>ii</sup> —V2—O1 <sup>iii</sup>	160.3 (5)	V1 <sup>v</sup> —O3—Te1	131.3 (2)
O5—V2—O7 <sup>i</sup>	104.2 (8)	V1—O4—V2 <sup>vi</sup>	145.3 (4)
O2V2O7 <sup>i</sup>	157.0 (6)	V1—O6—V2	167.7 (6)
$O4^{ii}$ — $V2$ — $O7^{i}$	88.6 (3)	V1—O7—V2 <sup>v</sup>	149.9 (4)

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

### Hydrogen-bond geometry (Å)

<i>D</i> —H··· <i>A</i>	D···A	
N1···O5 <sup>vii</sup>	2.820 (7)	
N1···O1 <sup>iii</sup>	2.89 (2)	
N1···O2	2.95 (2)	
N1···O2viii	2.96 (2)	
N1···O5 <sup>viii</sup>	3.15 (3)	
N1···O1 <sup>viii</sup>	3.20 (2)	
N1···O5 <sup>ix</sup>	3.20 (3)	
N1···O3 <sup>iii</sup>	3.39 (3)	

Symmetry codes: (iii) x, y, z-1; (vii) x, y-1, z; (viii) -x, -y+1, z-1/2; (ix) -x, -y+1, z+1/2.

Acta Cryst. (2014). E**70**, 27-30 **sup-3**