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Crystal structure of tetraguanidinium [hexahydrogen hexaarsenato(V)tetravanadate(V)] tetrahydrate

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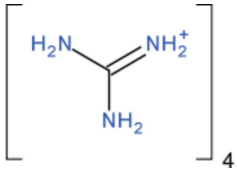
The complete polyoxidometallate anion in the title compound, $(CH_6N_3)_4[H_6V_4As_6O_{30}]\cdot 4H_2O$, is generated by crystallographic inversion symmetry. The polyhedral building units are distorted VO_6 octahedra and AsO_3OH tetrahedra. The VO_6 units feature a short formal V=O double bond and are linked by a common edge. Two such V_2O_6 double octahedral units are linked by four isolated AsO_3OH tetrahedra to complete the anion, which features two internal O—H···O hydrogen bonds. In the crystal, O—H···O hydrogen bonds between the polyoxidometallate anions generate $(01\bar{1})$ sheets. The sheets are connected by cation-to-cluster N—H···O hydrogen bonds, and cation-to-water N—H···O links also occur. The O atom of one of the water molecules is disordered over two sites in a 0.703 (17):0.297 (17) ratio.

Keywords: crystal structure; polyoxidometallate anion; vanadium; arsenic.

CCDC reference: 1004306

1. Related literature

For crystal structures containing the same type of anion accompanied by different counter-cations, see: Durif & Averbuch-Pouchot (1979); Nenoff *et al.* (1994); Bremner & Harrison (2002). The site symmetries of these anions include $\bar{1}$ (as seen for the title compound) as well as 2/*m* and *mmm*.



$[V_4As_6O_{30}H_6]^{4-}$ 4 H_2O

2. Experimental

2.1. Crystal data

$(CH_6N_3)_4[H_6V_4As_6O_{30}]\cdot 4H_2O$
M_r = 1447.71
Triclinic, $P\bar{1}$
a = 10.0403 (5) Å
b = 11.0199 (6) Å
c = 11.9806 (6) Å
α = 114.892 (1)°
β = 94.696 (1)°
γ = 111.751 (1)°
V = 1071.39 (10) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 5.56 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.20 mm

2.2. Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.287, T_{max} = 0.403
8711 measured reflections
4909 independent reflections
3655 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 0.99
4909 reflections
271 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.79 e Å^{-3}
$\Delta\rho_{min}$ = −1.25 e Å^{-3}

Table 1
Selected bond lengths (Å).

V1—O1	1.591 (4)	V2—O8	1.603 (5)
V1—O2	1.723 (4)	V2—O2	1.934 (4)
V1—O5	1.963 (4)	V2—O9	2.006 (4)
V1—O4	1.992 (4)	V2—O7	2.015 (4)
V1—O6	2.029 (4)	V2—O10	2.027 (4)
V1—O3	2.376 (4)	V2—O3	2.260 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O4^{i}	0.98	1.72	2.678 (5)	165
O14—H14···O13^{ii}	0.96	1.66	2.579 (6)	158
O15—H15···O13^{iii}	0.96	1.67	2.619 (6)	170
N1—H1*B*···O13^{iv}	0.86	2.26	3.056 (8)	154
N1—H1*A*···O6^{iv}	0.86	2.18	3.007 (7)	160
N2—H2*A*···O15^{v}	0.86	2.20	3.042 (8)	167
N2—H2*B*···O11^{iv}	0.86	2.44	3.210 (8)	150
N2—H2*B*···O1^{vi}	0.86	2.47	3.040 (8)	125
N3—H3*A*···O9^{v}	0.86	2.05	2.895 (7)	169
N3—H3*B*···O1*W*	0.86	2.20	2.984 (10)	152
N4—H4*A*···O12^{vi}	0.86	2.30	3.089 (9)	152
N4—H4*B*···O10^{vii}	0.86	2.48	3.230 (9)	146
N5—H5*A*···O2*WA*^{v}	0.86	2.14	2.959 (13)	160
N5—H5*B*···O7^{vii}	0.86	2.19	2.963 (8)	150
N6—H6*B*···O12^{vi}	0.86	2.37	3.140 (9)	150
N6—H6*B*···O5^{vi}	0.86	2.45	3.022 (7)	125
N6—H6*A*···O2*WB*^{v}	0.86	2.01	2.78 (5)	148

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM0004).

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supporting information

Acta Cryst. (2014). E**70**, m305–m306 [doi:10.1107/S1600536814011349]

Crystal structure of tetraguanidinium [hexahydrogen hexaarsenato(V)tetravanadate(V)] tetrahydrate

William T. A. Harrison

S1. Synthesis and crystallization

0.91 g of V_2O_5 and 0.90 g of $(CN_3H_6)_2CO_3$ were added to 10 ml of a 0.1 M H_3AsO_4 solution and placed in a Teflon-lined hydrothermal vessel, which was heated to 423 K for 24 hours. After cooling to room temperature over several hours, solids were recovered by vacuum filtration to yield a few orange blocks of the title compound accompanied by an unidentified brown powder.

S2. Refinement

The H atoms were located in different maps (O—H) or geometrically placed (N—H) and refined as riding atoms with U_{iso}(H) = 1.2U_{eq}(carrier). The water-molecule H atoms could not be located in the present experiment. One of the water molecule O atoms is disordered over two adjacent sites in a 0.703 (17):0.297 (17) ratio.

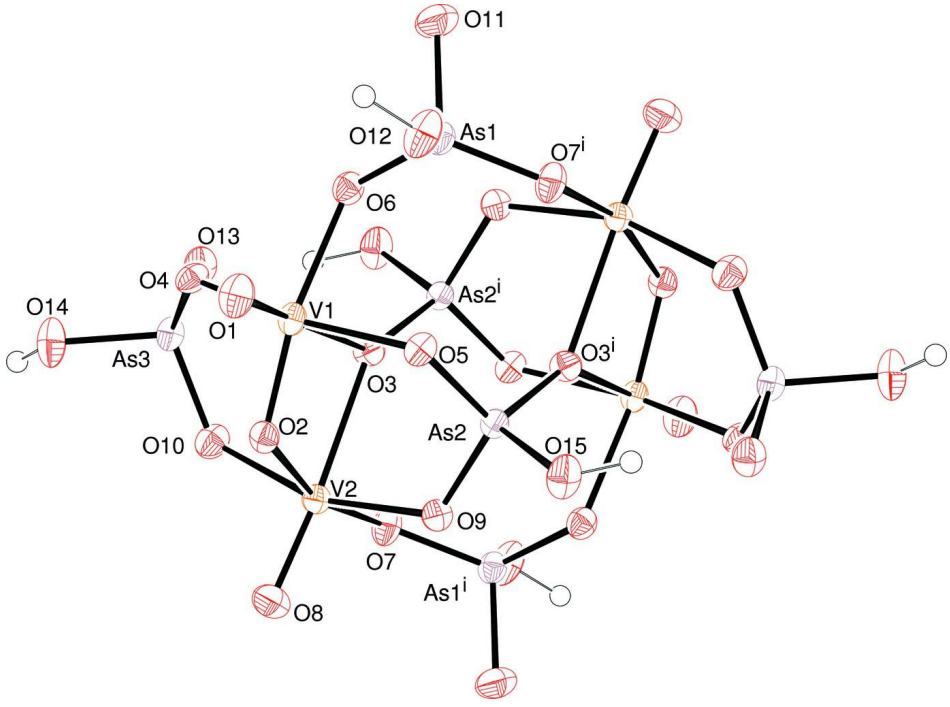

Figure 1

The molecular structure of the $(V_4As_6O_{30}H_6)^{4-}$ anion in the title compound showing 50% displacement ellipsoids. [Symmetry code: (i) –x, 1–y, 1–z.]

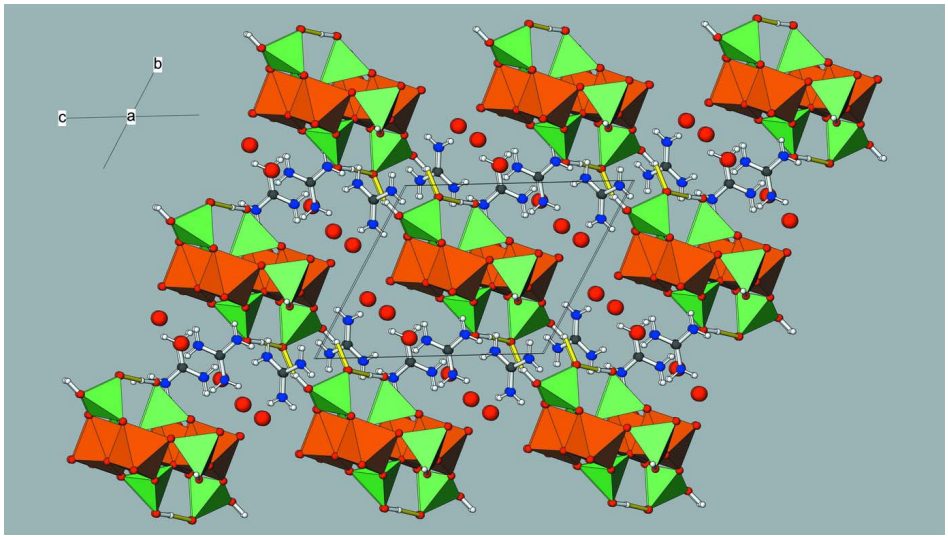

Figure 2

The packing of the title compound viewed down [100] with the anion shown in polyhedral representation (VO_6 octahedra orange, AsO_4 tetrahedra green). O—H···O hydrogen bonds within and between the anions are shown as yellow lines.

Tetraguanidinium [hexahydrogen hexaarsenato(V)tetravanadate(V)] tetrahydrate

Crystal data

$(CH_6N_3)_4[H_6V_4As_6O_{30}]\cdot 4H_2O$
M_r = 1447.71
Triclinic, $P\overline{1}$
a = 10.0403 (5) Å
b = 11.0199 (6) Å
c = 11.9806 (6) Å
α = 114.892 (1)°
β = 94.696 (1)°
γ = 111.751 (1)°
V = 1071.39 (10) Å^3
Z = 1
$F(000)$ = 704
D_x = 2.244 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.70173 Å
Cell parameters from 3266 reflections
θ = 2.3–27.5°
μ = 5.56 mm^{-1}
T = 293 K
Block, orange
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.287, T_{max} = 0.403
8711 measured reflections
4909 independent reflections
3655 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.2°, θ_{min} = 1.9°
h = −13→13
k = −14→12
l = −11→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 0.99
4909 reflections
271 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0766P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 1.79$ e Å^{-3}
$\Delta\rho_{min} = -1.25$ e Å^{-3}

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 *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
V1	0.26837 (10)	0.46285 (10)	0.57607 (9)	0.0176 (2)	
V2	0.05032 (10)	0.50770 (11)	0.74922 (9)	0.0193 (2)	
As1	0.26156 (6)	0.46227 (6)	0.30244 (6)	0.02044 (15)	
As2	−0.08161 (6)	0.30204 (6)	0.44111 (5)	0.01626 (14)	
As3	0.39072 (6)	0.77706 (6)	0.84192 (5)	0.01955 (15)	
O1	0.3661 (5)	0.3738 (5)	0.5506 (4)	0.0282 (10)	
O2	0.1882 (4)	0.4284 (4)	0.6880 (4)	0.0192 (8)	
O3	0.1265 (4)	0.6002 (4)	0.6171 (4)	0.0194 (8)	
O4	0.4243 (4)	0.6617 (4)	0.7138 (4)	0.0202 (8)	
O5	0.0880 (4)	0.3108 (4)	0.4329 (4)	0.0198 (8)	
O6	0.3215 (4)	0.5543 (4)	0.4605 (4)	0.0211 (8)	
O7	−0.0827 (4)	0.6107 (5)	0.7760 (4)	0.0254 (9)	
O8	0.0098 (5)	0.4435 (5)	0.8461 (4)	0.0328 (10)	
O9	−0.1059 (4)	0.3374 (4)	0.5863 (4)	0.0216 (8)	
O10	0.2301 (4)	0.7032 (4)	0.8748 (4)	0.0253 (9)	
O11	0.3741 (5)	0.5860 (6)	0.2582 (5)	0.0389 (12)	
O12	0.2976 (5)	0.3108 (5)	0.2403 (4)	0.0299 (10)	
H12	0.4043	0.3389	0.2643	0.036*	
O13	0.4138 (4)	0.9295 (4)	0.8315 (4)	0.0260 (9)	
O14	0.5335 (5)	0.8266 (5)	0.9655 (4)	0.0319 (10)	
H14	0.5560	0.9049	1.0518	0.038*	
O15	−0.2016 (5)	0.1152 (4)	0.3450 (4)	0.0290 (10)	
H15	−0.2710	0.1031	0.2766	0.035*	
C1	0.6359 (8)	−0.0343 (7)	0.5654 (7)	0.0336 (15)	
N1	0.5480 (7)	−0.1219 (6)	0.6055 (6)	0.0441 (16)	
H1A	0.5002	−0.2169	0.5551	0.053*	
H1B	0.5389	−0.0834	0.6818	0.053*	
N2	0.6494 (8)	−0.0929 (7)	0.4497 (7)	0.0530 (19)	
H2A	0.7044	−0.0363	0.4229	0.064*	
H2B	0.6033	−0.1882	0.4001	0.064*	
N3	0.7054 (8)	0.1105 (7)	0.6418 (7)	0.0510 (18)	
H3A	0.7607	0.1680	0.6159	0.061*	

H3B	0.6959	0.1482	0.7180	0.061*	
C2	0.9422 (9)	0.0768 (8)	0.8896 (7)	0.0406 (17)	
N4	0.8450 (8)	0.0292 (9)	0.9471 (8)	0.069 (2)	
H4A	0.7854	−0.0646	0.9131	0.082*	
H4B	0.8411	0.0919	1.0186	0.082*	
N5	1.0309 (9)	0.2208 (7)	0.9432 (7)	0.061 (2)	
H5A	1.0945	0.2540	0.9067	0.073*	
H5B	1.0254	0.2818	1.0147	0.073*	
N6	0.9483 (9)	−0.0180 (8)	0.7813 (7)	0.064 (2)	
H6A	1.0112	0.0134	0.7435	0.076*	
H6B	0.8894	−0.1120	0.7475	0.076*	
O1W	0.7341 (7)	0.3408 (9)	0.9023 (6)	0.074 (2)	
O2WA	0.2417 (12)	0.2587 (12)	0.7880 (10)	0.082 (4)*	0.703 (17)
O2WB	0.063 (5)	0.108 (5)	0.631 (5)	0.18 (2)*	0.297 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.0164 (4)	0.0168 (5)	0.0200 (5)	0.0099 (3)	0.0038 (4)	0.0073 (4)
V2	0.0180 (4)	0.0218 (5)	0.0200 (5)	0.0095 (4)	0.0055 (4)	0.0113 (4)
As1	0.0179 (3)	0.0225 (3)	0.0212 (3)	0.0109 (2)	0.0055 (2)	0.0093 (2)
As2	0.0159 (3)	0.0134 (3)	0.0203 (3)	0.0069 (2)	0.0036 (2)	0.0089 (2)
As3	0.0180 (3)	0.0178 (3)	0.0190 (3)	0.0090 (2)	0.0025 (2)	0.0055 (2)
O1	0.026 (2)	0.029 (2)	0.034 (2)	0.0197 (18)	0.0072 (18)	0.0131 (19)
O2	0.0200 (18)	0.0169 (19)	0.022 (2)	0.0090 (15)	0.0038 (15)	0.0109 (16)
O3	0.0208 (18)	0.0184 (19)	0.025 (2)	0.0113 (15)	0.0075 (16)	0.0136 (17)
O4	0.0168 (18)	0.0189 (19)	0.0205 (19)	0.0072 (15)	0.0075 (15)	0.0062 (16)
O5	0.0183 (18)	0.0166 (19)	0.022 (2)	0.0088 (15)	0.0058 (15)	0.0067 (16)
O6	0.0197 (18)	0.0189 (19)	0.020 (2)	0.0072 (15)	0.0042 (15)	0.0069 (16)
O7	0.0196 (19)	0.027 (2)	0.027 (2)	0.0131 (16)	0.0031 (17)	0.0086 (18)
O8	0.032 (2)	0.044 (3)	0.033 (2)	0.018 (2)	0.013 (2)	0.027 (2)
O9	0.0207 (19)	0.023 (2)	0.022 (2)	0.0098 (16)	0.0057 (16)	0.0120 (17)
O10	0.022 (2)	0.025 (2)	0.024 (2)	0.0098 (16)	0.0073 (17)	0.0076 (18)
O11	0.027 (2)	0.046 (3)	0.052 (3)	0.010 (2)	0.014 (2)	0.035 (3)
O12	0.022 (2)	0.026 (2)	0.037 (2)	0.0171 (17)	0.0060 (18)	0.0053 (19)
O13	0.027 (2)	0.019 (2)	0.027 (2)	0.0086 (16)	0.0018 (17)	0.0084 (17)
O14	0.030 (2)	0.031 (2)	0.023 (2)	0.0181 (19)	−0.0040 (18)	0.0021 (19)
O15	0.029 (2)	0.0147 (19)	0.034 (2)	0.0065 (16)	−0.0027 (18)	0.0091 (18)
C1	0.039 (4)	0.026 (3)	0.049 (4)	0.018 (3)	0.018 (3)	0.026 (3)
N1	0.059 (4)	0.024 (3)	0.050 (4)	0.012 (3)	0.024 (3)	0.024 (3)
N2	0.082 (5)	0.036 (4)	0.052 (4)	0.026 (3)	0.042 (4)	0.028 (3)
N3	0.066 (4)	0.032 (3)	0.049 (4)	0.011 (3)	0.027 (3)	0.023 (3)
C2	0.048 (4)	0.030 (4)	0.037 (4)	0.019 (3)	0.007 (3)	0.011 (3)
N4	0.057 (5)	0.051 (5)	0.078 (6)	0.017 (4)	0.028 (4)	0.018 (4)
N5	0.090 (6)	0.031 (4)	0.045 (4)	0.016 (4)	0.032 (4)	0.012 (3)
N6	0.096 (6)	0.036 (4)	0.050 (4)	0.030 (4)	0.031 (4)	0.012 (3)
O1W	0.062 (4)	0.101 (5)	0.049 (4)	0.019 (4)	0.003 (3)	0.047 (4)

Geometric parameters (Å, º)

V1—O1	1.591 (4)	O3—As2^{i}	1.671 (4)
V1—O2	1.723 (4)	O7—As1^{i}	1.665 (4)
V1—O5	1.963 (4)	O12—H12	0.9769
V1—O4	1.992 (4)	O14—H14	0.9637
V1—O6	2.029 (4)	O15—H15	0.9600
V1—O3	2.376 (4)	C1—N2	1.304 (9)
V2—O8	1.603 (5)	C1—N3	1.309 (9)
V2—O2	1.934 (4)	C1—N1	1.330 (8)
V2—O9	2.006 (4)	N1—H1A	0.8600
V2—O7	2.015 (4)	N1—H1B	0.8600
V2—O10	2.027 (4)	N2—H2A	0.8600
V2—O3	2.260 (4)	N2—H2B	0.8600
As1—O6	1.649 (4)	N3—H3A	0.8600
As1—O7^{i}	1.665 (4)	N3—H3B	0.8600
As1—O12	1.708 (4)	C2—N6	1.304 (9)
As1—O11	1.726 (5)	C2—N5	1.313 (9)
As2—O3^{i}	1.671 (4)	C2—N4	1.318 (10)
As2—O9	1.675 (4)	N4—H4A	0.8600
As2—O5	1.683 (4)	N4—H4B	0.8600
As2—O15	1.719 (4)	N5—H5A	0.8600
As3—O13	1.669 (4)	N5—H5B	0.8600
As3—O10	1.679 (4)	N6—H6A	0.8600
As3—O4	1.687 (4)	N6—H6B	0.8600
As3—O14	1.714 (4)		
O1—V1—O2	102.7 (2)	O13—As3—O4	109.0 (2)
O1—V1—O5	99.0 (2)	O10—As3—O4	117.33 (19)
O2—V1—O5	93.43 (17)	O13—As3—O14	108.5 (2)
O1—V1—O4	98.6 (2)	O10—As3—O14	107.1 (2)
O2—V1—O4	90.70 (17)	O4—As3—O14	101.8 (2)
O5—V1—O4	160.49 (18)	V1—O2—V2	119.8 (2)
O1—V1—O6	99.5 (2)	As2^{i}—O3—V2	136.2 (2)
O2—V1—O6	157.65 (18)	As2^{i}—O3—V1	137.1 (2)
O5—V1—O6	84.99 (16)	V2—O3—V1	86.10 (14)
O4—V1—O6	83.98 (16)	As3—O4—V1	124.0 (2)
O1—V1—O3	178.9 (2)	As2—O5—V1	121.6 (2)
O2—V1—O3	77.34 (16)	As1—O6—V1	125.2 (2)
O5—V1—O3	82.06 (15)	As1^{i}—O7—V2	127.5 (2)
O4—V1—O3	80.26 (15)	As2—O9—V2	122.6 (2)
O6—V1—O3	80.37 (15)	As3—O10—V2	124.8 (2)
O8—V2—O2	99.5 (2)	As1—O12—H12	112.3
O8—V2—O9	100.3 (2)	As3—O14—H14	122.9
O2—V2—O9	87.28 (16)	As2—O15—H15	109.3
O8—V2—O7	96.9 (2)	N2—C1—N3	120.6 (6)
O2—V2—O7	163.55 (18)	N2—C1—N1	119.8 (6)
O9—V2—O7	88.82 (16)	N3—C1—N1	119.5 (7)

O8—V2—O10	97.8 (2)	C1—N1—H1A	120.0
O2—V2—O10	87.81 (16)	C1—N1—H1B	120.0
O9—V2—O10	161.83 (17)	H1A—N1—H1B	120.0
O7—V2—O10	90.97 (17)	C1—N2—H2A	120.0
O8—V2—O3	175.3 (2)	C1—N2—H2B	120.0
O2—V2—O3	76.60 (15)	H2A—N2—H2B	120.0
O9—V2—O3	82.29 (15)	C1—N3—H3A	120.0
O7—V2—O3	87.04 (16)	C1—N3—H3B	120.0
O10—V2—O3	79.55 (16)	H3A—N3—H3B	120.0
O6—As1—O7^{i}	122.0 (2)	N6—C2—N5	121.2 (8)
O6—As1—O12	111.2 (2)	N6—C2—N4	120.1 (7)
O7^{i}—As1—O12	102.3 (2)	N5—C2—N4	118.7 (7)
O6—As1—O11	103.8 (2)	C2—N4—H4A	120.0
O7^{i}—As1—O11	110.7 (2)	C2—N4—H4B	120.0
O12—As1—O11	106.1 (2)	H4A—N4—H4B	120.0
O3^{i}—As2—O9	114.15 (19)	C2—N5—H5A	120.0
O3^{i}—As2—O5	112.86 (19)	C2—N5—H5B	120.0
O9—As2—O5	112.62 (19)	H5A—N5—H5B	120.0
O3^{i}—As2—O15	108.9 (2)	C2—N6—H6A	120.0
O9—As2—O15	103.6 (2)	C2—N6—H6B	120.0
O5—As2—O15	103.63 (19)	H6A—N6—H6B	120.0
O13—As3—O10	112.3 (2)		

O1—V1—O2—V2	−178.0 (2)	O3^{i}—As2—O5—V1	92.3 (3)
O5—V1—O2—V2	−77.9 (2)	O9—As2—O5—V1	−38.8 (3)
O4—V1—O2—V2	83.0 (2)	O15—As2—O5—V1	−150.1 (3)
O6—V1—O2—V2	7.3 (6)	O1—V1—O5—As2	152.3 (3)
O3—V1—O2—V2	3.14 (19)	O2—V1—O5—As2	48.8 (3)
O8—V2—O2—V1	179.4 (2)	O4—V1—O5—As2	−53.1 (6)
O9—V2—O2—V1	79.4 (2)	O6—V1—O5—As2	−108.9 (3)
O7—V2—O2—V1	2.9 (7)	O3—V1—O5—As2	−27.9 (2)
O10—V2—O2—V1	−83.1 (2)	O7^{i}—As1—O6—V1	67.3 (3)
O3—V2—O2—V1	−3.3 (2)	O12—As1—O6—V1	−53.5 (3)
O8—V2—O3—As2^{i}	−152 (2)	O11—As1—O6—V1	−167.1 (3)
O2—V2—O3—As2^{i}	173.8 (3)	O1—V1—O6—As1	73.3 (3)
O9—V2—O3—As2^{i}	84.8 (3)	O2—V1—O6—As1	−111.9 (4)
O7—V2—O3—As2^{i}	−4.4 (3)	O5—V1—O6—As1	−25.0 (3)
O10—V2—O3—As2^{i}	−95.9 (3)	O4—V1—O6—As1	171.1 (3)
O8—V2—O3—V1	37 (3)	O3—V1—O6—As1	−107.8 (3)
O2—V2—O3—V1	2.09 (13)	O8—V2—O7—As1^{i}	−88.3 (3)
O9—V2—O3—V1	−86.94 (14)	O2—V2—O7—As1^{i}	88.2 (6)
O7—V2—O3—V1	−176.15 (14)	O9—V2—O7—As1^{i}	12.0 (3)
O10—V2—O3—V1	92.32 (15)	O10—V2—O7—As1^{i}	173.8 (3)
O1—V1—O3—As2^{i}	91 (11)	O3—V2—O7—As1^{i}	94.3 (3)
O2—V1—O3—As2^{i}	−174.0 (3)	O3^{i}—As2—O9—V2	−85.4 (3)
O5—V1—O3—As2^{i}	−78.6 (3)	O5—As2—O9—V2	45.0 (3)
O4—V1—O3—As2^{i}	93.1 (3)	O15—As2—O9—V2	156.3 (2)
O6—V1—O3—As2^{i}	7.6 (3)	O8—V2—O9—As2	−153.2 (3)

O1—V1—O3—V2	−97 (10)	O2—V2—O9—As2	−54.0 (3)
O2—V1—O3—V2	−2.33 (14)	O7—V2—O9—As2	110.0 (3)
O5—V1—O3—V2	93.02 (15)	O10—V2—O9—As2	20.5 (7)
O4—V1—O3—V2	−95.27 (15)	O3—V2—O9—As2	22.8 (2)
O6—V1—O3—V2	179.25 (15)	O13—As3—O10—V2	109.2 (3)
O13—As3—O4—V1	−112.7 (3)	O4—As3—O10—V2	−18.1 (4)
O10—As3—O4—V1	16.3 (4)	O14—As3—O10—V2	−131.8 (3)
O14—As3—O4—V1	132.8 (3)	O8—V2—O10—As3	137.6 (3)
O1—V1—O4—As3	−142.3 (3)	O2—V2—O10—As3	38.3 (3)
O2—V1—O4—As3	−39.3 (3)	O9—V2—O10—As3	−36.1 (7)
O5—V1—O4—As3	63.0 (6)	O7—V2—O10—As3	−125.3 (3)
O6—V1—O4—As3	118.9 (3)	O3—V2—O10—As3	−38.5 (3)
O3—V1—O4—As3	37.7 (3)		

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O4^{ii}	0.98	1.72	2.678 (5)	165
O14—H14···O13^{iii}	0.96	1.66	2.579 (6)	158
O15—H15···O13^{i}	0.96	1.67	2.619 (6)	170
N1—H1*B*···O13^{iv}	0.86	2.26	3.056 (8)	154
N1—H1*A*···O6^{iv}	0.86	2.18	3.007 (7)	160
N2—H2*A*···O15^{v}	0.86	2.20	3.042 (8)	167
N2—H2*B*···O11^{iv}	0.86	2.44	3.210 (8)	150
N2—H2*B*···O1^{vi}	0.86	2.47	3.040 (8)	125
N3—H3*A*···O9^{v}	0.86	2.05	2.895 (7)	169
N3—H3*B*···O1*W*	0.86	2.20	2.984 (10)	152
N4—H4*A*···O12^{vi}	0.86	2.30	3.089 (9)	152
N4—H4*B*···O10^{vii}	0.86	2.48	3.230 (9)	146
N5—H5*A*···O2*WA*^{v}	0.86	2.14	2.959 (13)	160
N5—H5*B*···O7^{vii}	0.86	2.19	2.963 (8)	150
N6—H6*B*···O12^{vi}	0.86	2.37	3.140 (9)	150
N6—H6*B*···O5^{vi}	0.86	2.45	3.022 (7)	125
N6—H6*A*···O2*WB*^{v}	0.86	2.01	2.78 (5)	148

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