# Crystal structure of tetraguanidinium [hexahydrogen hexaarsenato(V)tetra-vanadate(V)] tetrahydrate

## William T. A. Harrison

Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland. *Correspondence e-mail: w.harrison@abdn.ac.uk

The complete polyoxidometallate anion in the title compound, $(CH_6N_3)_4[H_6V_4As_6O_{30}]\cdot4H_2O$, 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.

## 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 $mmm$.
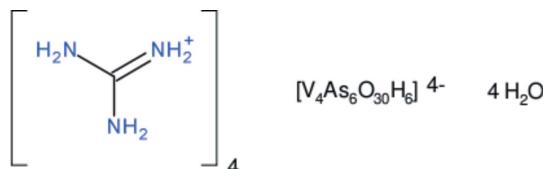


## 2. Experimental

### 2.1. Crystal data

| | |
|---|---|
| $(CH_6N_3)_4[H_6V_4As_6O_{30}]\cdot4H_2O$ | $\gamma = 111.751\ (1)°$ |
| $M_r = 1447.71$ | $V = 1071.39\ (10)$ Å³ |
| Triclinic, $P\bar{1}$ | $Z = 1$ |
| $a = 10.0403\ (5)$ Å | Mo $K\alpha$ radiation |
| $b = 11.0199\ (6)$ Å | $\mu = 5.56$ mm$^{-1}$ |
| $c = 11.9806\ (6)$ Å | $T = 293$ K |
| $\alpha = 114.892\ (1)°$ | $0.30 \times 0.20 \times 0.20$ mm |
| $\beta = 94.696\ (1)°$ | |

### 2.2. Data collection

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

### 2.3. Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | 271 parameters |
| $wR(F^2) = 0.129$ | H-atom parameters constrained |
| $S = 0.99$ | $\Delta\rho_{max} = 1.79$ e Å$^{-3}$ |
| 4909 reflections | $\Delta\rho_{min} = -1.25$ 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$B$···O1W | 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$A$···O2WA$^v$ | 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$A$···O2WB$^v$ | 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 reports

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).

## References

Bremner, C. A. & Harrison, W. T. A. (2002). *Acta Cryst.* E**58**, m254–m256.

Bruker (1999). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Dowty, E. (1999). *ATOMS*. Shape Software, Kingsport, Tennessee, USA.

Durif, A. & Averbuch-Pouchot, M. T. (1979). *Acta Cryst.* B**35**, 1441–1444.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Nenoff, T. M., Stucky, G. D. & Harrison, W. T. A. (1994). *Z. Kristallogr.* **209**, 892–898.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# 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.
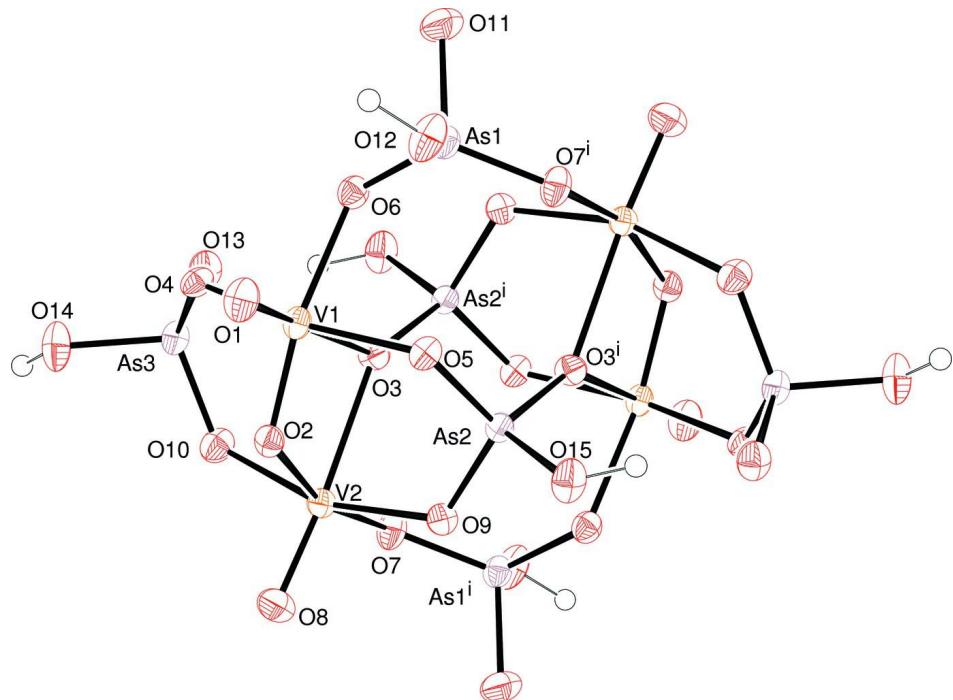


**Figure 1**

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.]
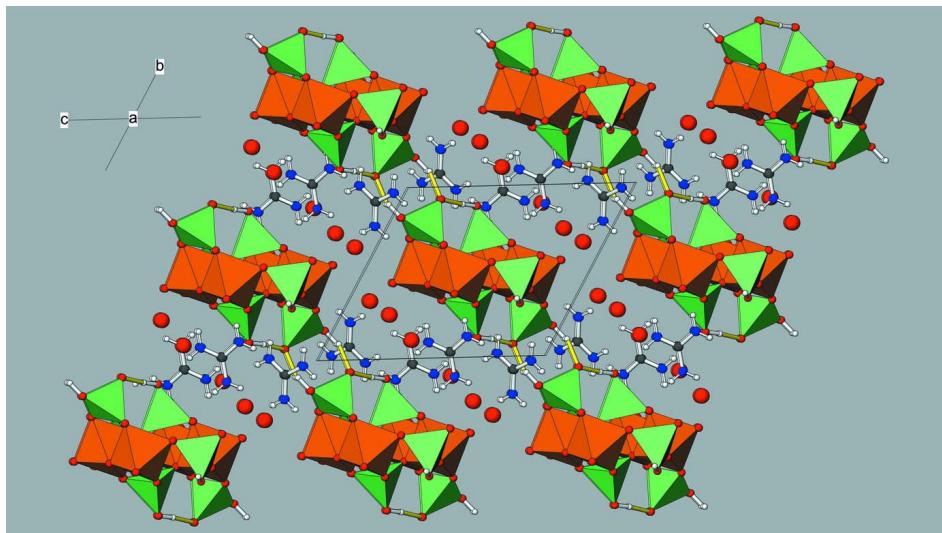
**Figure 2**

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*

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

*Data collection*

| | |
|---|---|
| Bruker SMART CCD diffractometer | 8711 measured reflections |
| Radiation source: fine-focus sealed tube | 4909 independent reflections |
| Graphite monochromator | 3655 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.035$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 1999) | $\theta_{max} = 27.2°$, $\theta_{min} = 1.9°$ |
| $T_{min} = 0.287$, $T_{max} = 0.403$ | $h = -13\rightarrow13$ |
| | $k = -14\rightarrow12$ |
| | $l = -11\rightarrow15$ |

*Refinement*

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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.