

Tetrakis(1,6-hexanediammonium) tetra-kis(hydrogenarsenate) heptahydrate

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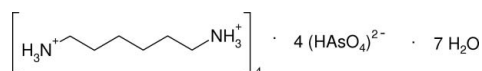
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; H-atom completeness 85%; disorder in main residue; R factor = 0.047; wR factor = 0.126; data-to-parameter ratio = 21.1.

The title compound, $4\text{C}_6\text{H}_{18}\text{N}_2^{2+} \cdot 4\text{HAsO}_4^{2-} \cdot 7\text{H}_2\text{O}$, contains a complex network of organic cations, hydrogenarsenate anions and water molecules. One of the two distinct $(\text{HAsO}_4)^{2-}$ anions shows the expected distinction between protonated and unprotonated As—O bond lengths. The component species interact by way of $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds, with the latter leading to distinct centrosymmetric $(\text{HAsO}_4)_4$ tetramers, in which the central linking $\text{O}-\text{H} \cdots \text{O}$ bond is assumed to be disordered about the inversion centre. Together, the cations and anions form an unusual three-dimensional framework encapsulating [100] channels occupied by the water molecules.

Related literature

For related structures and background literature, see: Wilkinson & Harrison (2007*a,b*); Wilson (2001).



Experimental

Crystal data

 $4\text{C}_6\text{H}_{18}\text{N}_2^{2+} \cdot 4\text{HAsO}_4^{2-} \cdot 7\text{H}_2\text{O}$ $M_r = 1158.72$ Triclinic, $P\bar{1}$ $a = 10.2206$ (2) Å $b = 11.2773$ (4) Å $c = 12.7211$ (4) Å $\alpha = 109.6648$ (12)° $\beta = 108.0176$ (18)° $\gamma = 97.7941$ (18)° $V = 1265.11$ (6) Å³ $Z = 1$ Mo $K\alpha$ radiation $\mu = 2.70$ mm⁻¹ $T = 120$ (2) K $0.42 \times 0.22 \times 0.06$ mm

Data collection

Bruker-Nonius KappaCCD diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2003)

 $T_{\min} = 0.397$, $T_{\max} = 0.855$

25594 measured reflections

5804 independent reflections

4788 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.053$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.047$ $wR(F^2) = 0.126$ $S = 1.07$

5804 reflections

275 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 3.79$ e Å⁻³ $\Delta\rho_{\text{min}} = -1.87$ e Å⁻³

Table 1

Selected bond lengths (Å).

As1—O1	1.667 (3)	As2—O8	1.652 (3)
As1—O2	1.671 (2)	As2—O7	1.668 (3)
As1—O3	1.674 (2)	As2—O6	1.689 (3)
As1—O4	1.734 (2)	As2—O5	1.721 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
O4—H1 ⁱ ⋯O8	0.89	1.72	2.584 (4)	163
O5—H2 ⁱ ⋯O5 ⁱ	0.90	1.76	2.657 (6)	180
O6—H3 ⁱ ⋯O12	0.90	2.00	2.894 (8)	180
N1—H1A ⁱ ⋯O2 ⁱⁱ	0.91	1.93	2.814 (4)	164
N1—H1B ⁱ ⋯O2	0.91	1.81	2.717 (4)	171
N1—H1C ⁱ ⋯O7 ⁱⁱⁱ	0.91	1.90	2.810 (4)	174
N2—H2D ⁱ ⋯O8 ^{iv}	0.91	1.82	2.716 (4)	169
N2—H2E ⁱ ⋯O1 ^v	0.91	1.95	2.836 (4)	165
N2—H2F ⁱ ⋯O7 ^v	0.91	2.06	2.965 (4)	171
N3—H3C ⁱ ⋯O7 ⁱⁱⁱ	0.91	1.94	2.826 (4)	166
N3—H3D ⁱ ⋯O3 ^{vi}	0.91	1.83	2.699 (4)	158
N3—H3E ⁱ ⋯O5 ^{vi}	0.91	1.89	2.779 (5)	166
N4—H4C ⁱ ⋯O3 ^{vii}	0.91	1.84	2.735 (4)	167
N4—H4D ⁱ ⋯O2 ^{iv}	0.91	2.06	2.941 (4)	162
N4—H4E ⁱ ⋯O6 ^{viii}	0.91	1.90	2.801 (4)	169

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

Data collection: COLLECT (Nonius, 1998); cell refinement: HKL SCALEPACK (Otwinowski & Minor, 1997); data reduction: HKL DENZO and SCALEPACK (Otwinowski & Minor, 1997) and SORTAV (Blessing, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2394).

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supplementary materials

Acta Cryst. (2007). E63, m1891 [doi:10.1107/S1600536807023719]

Tetrakis(1,6-hexanediammonium) tetrakis(hydrogenarsenate) heptahydrate

M. J. Todd and W. T. A. Harrison

Comment

The title compound, (I), was prepared as part of our ongoing studies of hydrogen bonding interactions in the molecular salts of arsenic oxo-anions (Wilkinson & Harrison, 2007a,b). Its asymmetric unit contains two organic cations, two anions and three-and-a-half water molecules (Fig. 1).

The tetrahedral (HAsO₄)²⁻ anion in (I) shows the expected distinction (Table 1) between its protonated and unprotonated As—O bond lengths. The situation for the (HAs₂O₄)²⁻ group is less clear cut, and seems to be correlated with disorder of its H atoms (see below). Both C₂H₁₀N₂²⁺ dications are in their extended conformations with all their backbone torsion angles close to 180°. Three fully occupied (O9, O10, O11) water molecules and one half occupied (O12) water molecule complete the structure. O12 cannot be more than half occupied due to a close O12^{xx} (xx = -x, 1 - y, -z) contact of 2.21 (1) Å.

As well as Coulombic forces, the component species in (I) interact by way of a network of N—H[⋯]O and O—H[⋯]O hydrogen bonds (Table 2). The (HAsO₄)²⁻ units are linked by way of O—H[⋯]O bonds into distinctive, isolated, tetramers (Fig. 2). The complete assembly is generated by inversion, thus the central O5—H2[⋯]O5ⁱ (see Table 2 for symmetry code) link must be disordered (*i.e.* O5—H2[⋯]O5ⁱ + O5ⁱ—H2[⋯]O5 bonds) or possibly a symmetric O5[⋯]H2[⋯]O5ⁱ bond (*i.e.* the H atom occupies the inversion centre (Wilson, 2001). These possibilities could not be distinguished in the present experiment and a disordered model was assumed. Either of these possibilities necessitates disorder of the H atoms of the As₂-hydrogenarsenate group, with one of the (HAsO₄)²⁻ moieties directing its H atom elsewhere. The extended As₂—O6 bond length suggests that O6 bears the H atom and if so, it makes an O—H[⋯]O link to the disordered O12 water molecule O atom (Fig. 2).

The cations interact with the anion tetramers by way of a large number of N—H[⋯]O bonds (Table 2), with each NH₃⁺ group making three such links, as is typically seen in these systems (Wilkinson & Harrison, 2007a). Unfortunately the H atoms of the water molecules in (I) could not be located in the present study. The disorder of the As₂ H atoms and O12 seems to require that at least some of the water H atoms are also disordered, and the situation is too uncertain to allow their geometrical placement with any confidence.

The packing for (I) results in a distinctive structure (Fig. 3) in which hydrogen-bonded (001) sheets of tetrahedral tetramers are bridged by the organic molecules to result in a framework encapsulating [100] channels, which are occupied by the water molecules.

The structure of 1,6-diamminiumhexane bis(dihydrogenarsenate), C₆H₁₈N₂·(H₂AsO₄)₂, (II), (Wilkinson & Harrison, 2007a), in which the arsenic-containing anion bears two protons, is entirely different to that of (I). In (II), infinite sheets of H-bonded tetrahedra arise and the centrosymmetric cation adopts a *gauche* conformation. Other types of supramolecular networks based on hydrogen bonded organic cations and arsenate tetrahedra are described by Wilkinson & Harrison (2007b).

supplementary materials

Experimental

0.5 M Aqueous solutions of 1,6-diaminohexane (10 ml) and arsenic acid (10 ml) were mixed, resulting in a colourless liquid. Aqueous ammonia was then added to the mixture to raise the pH to 11. Colourless slabs of (I) grew as the water slowly evaporated at 298 K.

Refinement

The highest difference peak is 0.82 Å from As2, and the deepest difference hole is 0.62 Å from As2 perhaps indicating disorder of the (HAs₂O₄)²⁻ group, but refinements attempting to model this were unstable. The next-highest feature in the final difference map (0.76 e Å³) is at the noise level.

H1 was located in a difference map and refined as riding. H2, H3 and the C- and N-bonded hydrogen atoms were placed in idealized positions (O—H = 0.90 Å, C—H = 0.99 Å, N—H = 0.91 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$.

The H atoms of the water molecules could not be located.

Figures

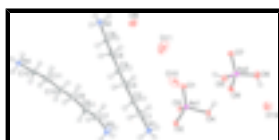


Fig. 1. The molecular structure of (I) (50% displacement ellipsoids; H atoms are drawn as spheres of arbitrary radius).

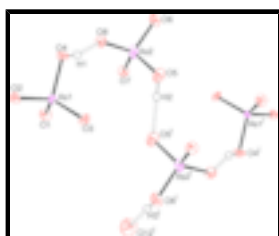


Fig. 2. A tetramer of (HAsO₄)²⁻ tetrahedra in (I) with with the H bonds indicated by double-dashed lines. Symmetry code as in Table 2. Only one disorder component for H2, H3 and O12 is shown.

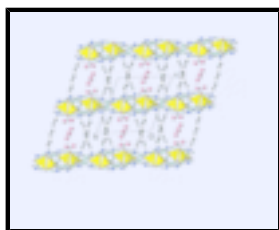
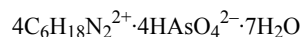


Fig. 3. Unit cell packing in (I) showing the (001) hydrogenarsenate layers bridged by the organic cations, with H bonds indicated by pale blue lines. The water O atoms (pink spheres) occupy [100] channels.

tetra(1,6-hexanediammonium) tetra(hydrogenarsenate) heptahydrate

Crystal data



$M_r = 1158.72$

Triclinic, $P\bar{1}$

$Z = 1$

$F_{000} = 606$

$D_x = 1.521 \text{ Mg m}^{-3}$

Hall symbol: -P 1
 $a = 10.2206$ (2) Å
 $b = 11.2773$ (4) Å
 $c = 12.7211$ (4) Å
 $\alpha = 109.6648$ (12)°
 $\beta = 108.0176$ (18)°
 $\gamma = 97.7941$ (18)°
 $V = 1265.11$ (6) Å³

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 5718 reflections
 $\theta = 2.9$ – 27.5 °
 $\mu = 2.70$ mm⁻¹
 $T = 120$ (2) K
 Slab, colourless
 $0.42 \times 0.22 \times 0.06$ mm

Data collection

Bruker-Nonius KappaCCD diffractometer	5804 independent reflections
Radiation source: fine-focus sealed tube	4788 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.053$
$T = 120$ (2) K	$\theta_{\text{max}} = 27.6$ °
ω and φ scans	$\theta_{\text{min}} = 3.3$ °
Absorption correction: multi-scan (SADABS; Bruker, 2003)	$h = -13 \rightarrow 13$
$T_{\text{min}} = 0.397$, $T_{\text{max}} = 0.855$	$k = -14 \rightarrow 14$
25594 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap and geom
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.126$	$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 3.4341P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
5804 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
275 parameters	$\Delta\rho_{\text{max}} = 3.79$ e Å ⁻³
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -1.87$ 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 $F^2 > 2\sigma(F^2)$ 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.

supplementary materials

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
As1	0.26080 (3)	0.19642 (3)	0.50711 (3)	0.01311 (11)	
O1	0.3494 (3)	0.3195 (3)	0.6417 (2)	0.0214 (6)	
O2	0.3187 (3)	0.0621 (2)	0.4901 (2)	0.0183 (5)	
O3	0.0842 (3)	0.1579 (3)	0.4714 (2)	0.0204 (5)	
O4	0.2927 (3)	0.2388 (3)	0.3965 (2)	0.0208 (6)	
H1	0.2858	0.3203	0.4126	0.025*	
As2	0.21274 (4)	0.58973 (4)	0.44105 (4)	0.02178 (12)	
O5	0.0387 (3)	0.5234 (3)	0.4168 (3)	0.0289 (6)	
H2	0.0125	0.5075	0.4732	0.035*	0.50
O6	0.1964 (3)	0.6605 (3)	0.3415 (3)	0.0309 (7)	
H3	0.1460	0.6094	0.2632	0.037*	0.50
O7	0.2942 (3)	0.7031 (2)	0.5818 (2)	0.0206 (5)	
O8	0.2976 (3)	0.4749 (2)	0.4154 (2)	0.0219 (6)	
N1	0.4702 (3)	-0.0659 (3)	0.6085 (3)	0.0160 (6)	
H1A	0.5363	-0.0810	0.5750	0.019*	
H1B	0.4155	-0.0210	0.5747	0.019*	
H1C	0.4137	-0.1434	0.5949	0.019*	
C1	0.5434 (4)	0.0118 (4)	0.7400 (3)	0.0170 (7)	
H1D	0.6166	0.0891	0.7549	0.020*	
H1E	0.5925	-0.0417	0.7782	0.020*	
C2	0.4383 (4)	0.0566 (4)	0.7969 (3)	0.0185 (7)	
H2A	0.3662	-0.0206	0.7836	0.022*	
H2B	0.3879	0.1088	0.7577	0.022*	
C3	0.5151 (4)	0.1388 (4)	0.9319 (3)	0.0194 (7)	
H3A	0.5918	0.2120	0.9444	0.023*	
H3B	0.5608	0.0843	0.9707	0.023*	
C4	0.4182 (4)	0.1948 (4)	0.9946 (3)	0.0203 (8)	
H4A	0.3691	0.2469	0.9545	0.024*	
H4B	0.3443	0.1222	0.9867	0.024*	
C5	0.5023 (4)	0.2805 (4)	1.1273 (3)	0.0186 (7)	
H5A	0.5784	0.3509	1.1348	0.022*	
H5B	0.5488	0.2273	1.1674	0.022*	
C6	0.4099 (4)	0.3416 (4)	1.1915 (3)	0.0174 (7)	
H6A	0.3597	0.3918	1.1497	0.021*	
H6B	0.3371	0.2717	1.1886	0.021*	
N2	0.4985 (3)	0.4299 (3)	1.3197 (3)	0.0164 (6)	
H2C	0.4405	0.4539	1.3601	0.020*	
H2D	0.5534	0.5024	1.3225	0.020*	
H2E	0.5558	0.3875	1.3546	0.020*	
N3	0.0774 (3)	-0.2552 (3)	0.6731 (3)	0.0237 (7)	
H3C	0.1528	-0.2535	0.6494	0.028*	
H3D	0.0206	-0.2099	0.6421	0.028*	
H3E	0.0262	-0.3395	0.6458	0.028*	
C7	0.1305 (4)	-0.1947 (5)	0.8063 (4)	0.0283 (9)	
H7A	0.1786	-0.1011	0.8348	0.034*	

H7B	0.2024	-0.2368	0.8395	0.034*	
C8	0.0135 (4)	-0.2063 (4)	0.8551 (4)	0.0236 (8)	
H8A	-0.0452	-0.2980	0.8176	0.028*	
H8B	-0.0495	-0.1511	0.8353	0.028*	
C9	0.0814 (4)	-0.1622 (4)	0.9913 (3)	0.0239 (8)	
H9A	0.1458	-0.2171	1.0090	0.029*	
H9B	0.1409	-0.0710	1.0266	0.029*	
C10	-0.0225 (4)	-0.1685 (4)	1.0540 (3)	0.0225 (8)	
H10A	-0.0775	-0.1036	1.0483	0.027*	
H10B	-0.0907	-0.2564	1.0133	0.027*	
C11	0.0575 (4)	-0.1407 (4)	1.1859 (3)	0.0204 (8)	
H11A	0.1177	-0.2020	1.1902	0.025*	
H11B	0.1220	-0.0511	1.2262	0.025*	
C12	-0.0375 (4)	-0.1525 (4)	1.2549 (3)	0.0200 (7)	
H12A	-0.0848	-0.0808	1.2648	0.024*	
H12B	-0.1125	-0.2366	1.2091	0.024*	
N4	0.0499 (3)	-0.1459 (3)	1.3753 (3)	0.0177 (6)	
H4C	-0.0065	-0.1489	1.4180	0.021*	
H4D	0.1209	-0.0700	1.4159	0.021*	
H4E	0.0888	-0.2149	1.3658	0.021*	
O9	0.8047 (3)	0.5471 (3)	1.2428 (3)	0.0365 (7)	
O10	0.6206 (4)	0.3245 (3)	0.7704 (4)	0.0487 (10)	
O11	0.7583 (5)	0.5024 (5)	1.0047 (4)	0.0727 (14)	
O12	0.0334 (8)	0.4948 (8)	0.0893 (7)	0.0505 (19)	0.50

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.01328 (18)	0.01261 (18)	0.01532 (19)	0.00403 (13)	0.00671 (14)	0.00644 (14)
O1	0.0217 (13)	0.0167 (13)	0.0159 (13)	0.0013 (10)	0.0032 (10)	0.0001 (10)
O2	0.0250 (13)	0.0156 (12)	0.0212 (13)	0.0100 (10)	0.0114 (11)	0.0112 (11)
O3	0.0103 (11)	0.0293 (14)	0.0210 (14)	0.0025 (10)	0.0065 (10)	0.0099 (12)
O4	0.0324 (15)	0.0173 (13)	0.0246 (14)	0.0104 (11)	0.0193 (12)	0.0134 (11)
As2	0.0211 (2)	0.0165 (2)	0.0222 (2)	0.00862 (15)	0.00423 (16)	0.00361 (16)
O5	0.0213 (14)	0.0269 (15)	0.0310 (16)	0.0043 (12)	0.0083 (12)	0.0051 (13)
O6	0.0370 (17)	0.0348 (17)	0.0321 (17)	0.0191 (13)	0.0139 (13)	0.0221 (14)
O7	0.0204 (13)	0.0176 (13)	0.0183 (13)	0.0024 (10)	0.0059 (11)	0.0031 (11)
O8	0.0278 (14)	0.0156 (12)	0.0282 (15)	0.0102 (11)	0.0147 (12)	0.0103 (11)
N1	0.0163 (14)	0.0159 (14)	0.0191 (16)	0.0055 (11)	0.0085 (12)	0.0087 (12)
C1	0.0150 (16)	0.0198 (17)	0.0175 (18)	0.0056 (14)	0.0073 (14)	0.0076 (15)
C2	0.0154 (16)	0.0204 (18)	0.0162 (18)	0.0033 (14)	0.0065 (14)	0.0031 (15)
C3	0.0173 (17)	0.0229 (18)	0.0160 (18)	0.0061 (14)	0.0068 (14)	0.0050 (15)
C4	0.0144 (17)	0.0219 (18)	0.0178 (18)	0.0029 (14)	0.0042 (14)	0.0022 (15)
C5	0.0154 (17)	0.0233 (18)	0.0154 (18)	0.0042 (14)	0.0066 (14)	0.0054 (15)
C6	0.0135 (16)	0.0193 (17)	0.0154 (17)	0.0019 (13)	0.0041 (13)	0.0044 (14)
N2	0.0174 (14)	0.0169 (14)	0.0149 (15)	0.0053 (12)	0.0061 (12)	0.0061 (12)
N3	0.0174 (15)	0.0352 (19)	0.0227 (17)	0.0067 (14)	0.0088 (13)	0.0155 (15)
C7	0.0175 (18)	0.048 (3)	0.022 (2)	0.0078 (17)	0.0079 (16)	0.0177 (19)

supplementary materials

C8	0.0168 (18)	0.036 (2)	0.022 (2)	0.0053 (16)	0.0078 (15)	0.0164 (18)
C9	0.0164 (18)	0.036 (2)	0.021 (2)	0.0052 (16)	0.0066 (15)	0.0158 (18)
C10	0.0153 (17)	0.031 (2)	0.0212 (19)	0.0030 (15)	0.0062 (15)	0.0127 (17)
C11	0.0160 (17)	0.028 (2)	0.0203 (19)	0.0048 (15)	0.0079 (14)	0.0127 (16)
C12	0.0170 (17)	0.0246 (19)	0.0168 (18)	0.0036 (14)	0.0045 (14)	0.0088 (15)
N4	0.0191 (15)	0.0153 (14)	0.0185 (16)	0.0018 (12)	0.0076 (12)	0.0072 (12)
O9	0.0394 (18)	0.0360 (18)	0.0305 (17)	0.0069 (14)	0.0184 (14)	0.0056 (14)
O10	0.0305 (18)	0.0350 (19)	0.064 (3)	0.0062 (14)	-0.0029 (16)	0.0200 (18)
O11	0.062 (3)	0.105 (4)	0.048 (3)	0.011 (3)	0.015 (2)	0.036 (3)
O12	0.040 (4)	0.054 (5)	0.040 (4)	0.002 (3)	0.006 (3)	0.010 (4)

Geometric parameters (Å, °)

As1—O1	1.667 (3)	C6—H6A	0.9900
As1—O2	1.671 (2)	C6—H6B	0.9900
As1—O3	1.674 (2)	N2—H2C	0.9100
As1—O4	1.734 (2)	N2—H2D	0.9100
O4—H1	0.8895	N2—H2E	0.9100
As2—O8	1.652 (3)	N3—C7	1.478 (5)
As2—O7	1.668 (3)	N3—H3C	0.9100
As2—O6	1.689 (3)	N3—H3D	0.9100
As2—O5	1.721 (3)	N3—H3E	0.9100
O5—H2	0.8999	C7—C8	1.518 (5)
O6—H3	0.8975	C7—H7A	0.9900
N1—C1	1.479 (5)	C7—H7B	0.9900
N1—H1A	0.9100	C8—C9	1.521 (5)
N1—H1B	0.9100	C8—H8A	0.9900
N1—H1C	0.9100	C8—H8B	0.9900
C1—C2	1.522 (5)	C9—C10	1.519 (5)
C1—H1D	0.9900	C9—H9A	0.9900
C1—H1E	0.9900	C9—H9B	0.9900
C2—C3	1.524 (5)	C10—C11	1.519 (5)
C2—H2A	0.9900	C10—H10A	0.9900
C2—H2B	0.9900	C10—H10B	0.9900
C3—C4	1.528 (5)	C11—C12	1.517 (5)
C3—H3A	0.9900	C11—H11A	0.9900
C3—H3B	0.9900	C11—H11B	0.9900
C4—C5	1.517 (5)	C12—N4	1.486 (5)
C4—H4A	0.9900	C12—H12A	0.9900
C4—H4B	0.9900	C12—H12B	0.9900
C5—C6	1.519 (5)	N4—H4C	0.9100
C5—H5A	0.9900	N4—H4D	0.9100
C5—H5B	0.9900	N4—H4E	0.9100
C6—N2	1.489 (4)		
O1—As1—O2	114.20 (13)	H6A—C6—H6B	108.1
O1—As1—O3	112.72 (13)	C6—N2—H2C	109.5
O2—As1—O3	109.82 (13)	C6—N2—H2D	109.5
O1—As1—O4	109.54 (13)	H2C—N2—H2D	109.5
O2—As1—O4	101.96 (12)	C6—N2—H2E	109.5

O3—As1—O4	107.90 (13)	H2C—N2—H2E	109.5
As1—O4—H1	106.7	H2D—N2—H2E	109.5
O8—As2—O7	111.21 (13)	C7—N3—H3C	109.5
O8—As2—O6	111.64 (14)	C7—N3—H3D	109.5
O7—As2—O6	110.05 (14)	H3C—N3—H3D	109.5
O8—As2—O5	110.83 (14)	C7—N3—H3E	109.5
O7—As2—O5	109.28 (13)	H3C—N3—H3E	109.5
O6—As2—O5	103.56 (15)	H3D—N3—H3E	109.5
As2—O5—H2	123.3	N3—C7—C8	113.4 (3)
As2—O6—H3	117.5	N3—C7—H7A	108.9
C1—N1—H1A	109.5	C8—C7—H7A	108.9
C1—N1—H1B	109.5	N3—C7—H7B	108.9
H1A—N1—H1B	109.5	C8—C7—H7B	108.9
C1—N1—H1C	109.5	H7A—C7—H7B	107.7
H1A—N1—H1C	109.5	C7—C8—C9	109.0 (3)
H1B—N1—H1C	109.5	C7—C8—H8A	109.9
N1—C1—C2	111.2 (3)	C9—C8—H8A	109.9
N1—C1—H1D	109.4	C7—C8—H8B	109.9
C2—C1—H1D	109.4	C9—C8—H8B	109.9
N1—C1—H1E	109.4	H8A—C8—H8B	108.3
C2—C1—H1E	109.4	C10—C9—C8	115.5 (3)
H1D—C1—H1E	108.0	C10—C9—H9A	108.4
C1—C2—C3	110.9 (3)	C8—C9—H9A	108.4
C1—C2—H2A	109.5	C10—C9—H9B	108.4
C3—C2—H2A	109.5	C8—C9—H9B	108.4
C1—C2—H2B	109.5	H9A—C9—H9B	107.5
C3—C2—H2B	109.5	C11—C10—C9	110.4 (3)
H2A—C2—H2B	108.0	C11—C10—H10A	109.6
C2—C3—C4	114.2 (3)	C9—C10—H10A	109.6
C2—C3—H3A	108.7	C11—C10—H10B	109.6
C4—C3—H3A	108.7	C9—C10—H10B	109.6
C2—C3—H3B	108.7	H10A—C10—H10B	108.1
C4—C3—H3B	108.7	C12—C11—C10	114.5 (3)
H3A—C3—H3B	107.6	C12—C11—H11A	108.6
C5—C4—C3	111.5 (3)	C10—C11—H11A	108.6
C5—C4—H4A	109.3	C12—C11—H11B	108.6
C3—C4—H4A	109.3	C10—C11—H11B	108.6
C5—C4—H4B	109.3	H11A—C11—H11B	107.6
C3—C4—H4B	109.3	N4—C12—C11	109.6 (3)
H4A—C4—H4B	108.0	N4—C12—H12A	109.7
C4—C5—C6	112.9 (3)	C11—C12—H12A	109.7
C4—C5—H5A	109.0	N4—C12—H12B	109.7
C6—C5—H5A	109.0	C11—C12—H12B	109.7
C4—C5—H5B	109.0	H12A—C12—H12B	108.2
C6—C5—H5B	109.0	C12—N4—H4C	109.5
H5A—C5—H5B	107.8	C12—N4—H4D	109.5
N2—C6—C5	110.7 (3)	H4C—N4—H4D	109.5
N2—C6—H6A	109.5	C12—N4—H4E	109.5
C5—C6—H6A	109.5	H4C—N4—H4E	109.5

supplementary materials

N2—C6—H6B	109.5	H4D—N4—H4E	109.5
C5—C6—H6B	109.5		
N1—C1—C2—C3	-178.9 (3)	N3—C7—C8—C9	170.5 (4)
C1—C2—C3—C4	176.4 (3)	C7—C8—C9—C10	-179.8 (4)
C2—C3—C4—C5	-177.4 (3)	C8—C9—C10—C11	172.1 (3)
C3—C4—C5—C6	178.1 (3)	C9—C10—C11—C12	-176.6 (3)
C4—C5—C6—N2	-177.1 (3)	C10—C11—C12—N4	170.3 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O4—H1 \cdots O8	0.89	1.72	2.584 (4)	163
O5—H2 \cdots O5 ⁱ	0.90	1.76	2.657 (6)	180
O6—H3 \cdots O12	0.90	2.00	2.894 (8)	180
N1—H1A \cdots O2 ⁱⁱ	0.91	1.93	2.814 (4)	164
N1—H1B \cdots O2	0.91	1.81	2.717 (4)	171
N1—H1C \cdots O7 ⁱⁱⁱ	0.91	1.90	2.810 (4)	174
N2—H2C \cdots O8 ^{iv}	0.91	1.82	2.716 (4)	169
N2—H2D \cdots O1 ^v	0.91	1.95	2.836 (4)	165
N2—H2E \cdots O7 ^v	0.91	2.06	2.965 (4)	171
N3—H3C \cdots O7 ⁱⁱⁱ	0.91	1.94	2.826 (4)	166
N3—H3D \cdots O3 ^{vi}	0.91	1.83	2.699 (4)	158
N3—H3E \cdots O5 ^{vi}	0.91	1.89	2.779 (5)	166
N4—H4C \cdots O3 ^{vii}	0.91	1.84	2.735 (4)	167
N4—H4D \cdots O2 ^{iv}	0.91	2.06	2.941 (4)	162
N4—H4E \cdots O6 ^{viii}	0.91	1.90	2.801 (4)	169

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

Fig. 1

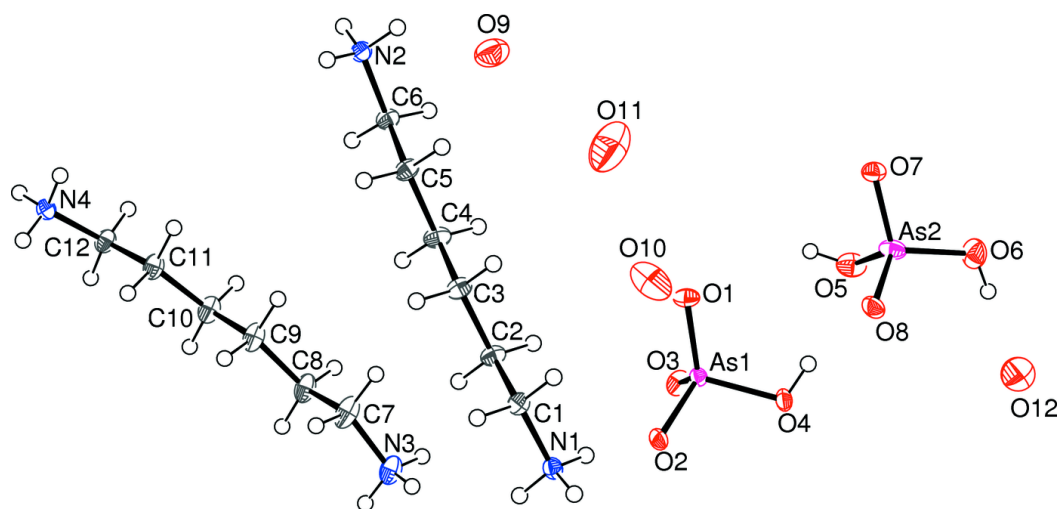


Fig. 2

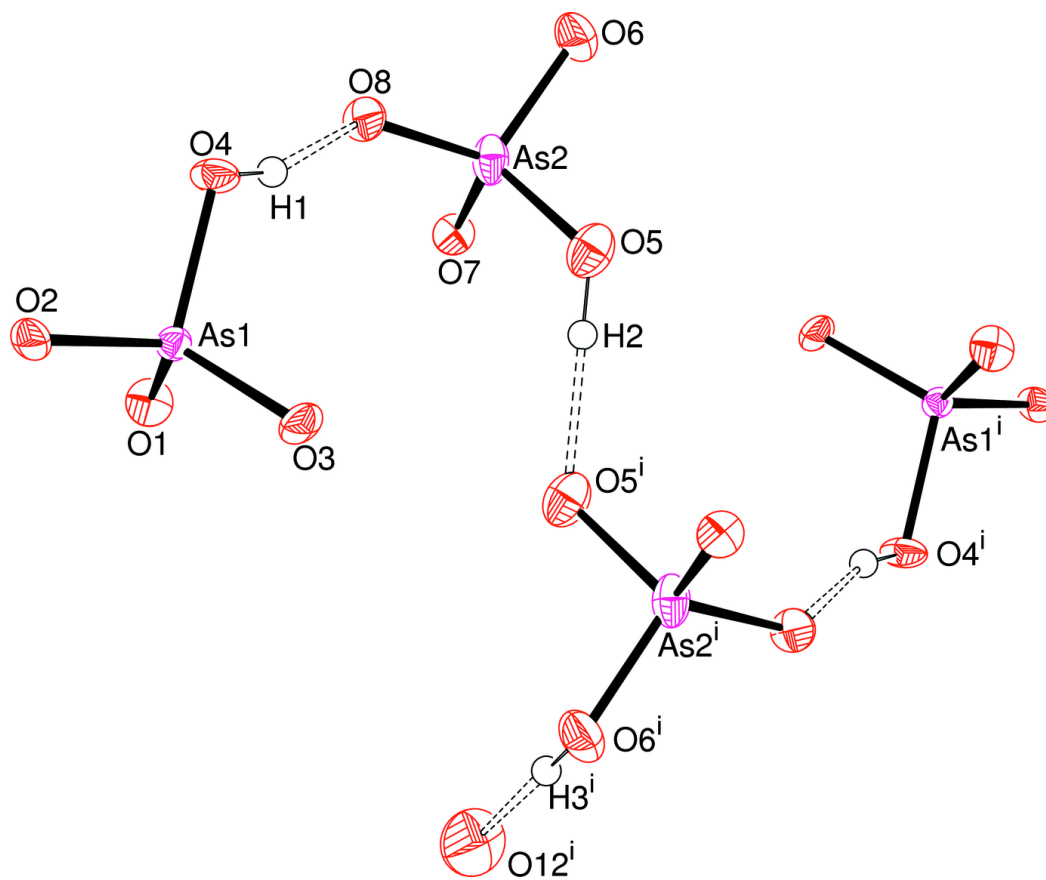


Fig. 3

