

Malcolm J. Todd and
William T. A. Harrison*Department of Chemistry, University of
Aberdeen, Meston Walk, Aberdeen AB24
3UE, ScotlandCorrespondence e-mail:
w.harrison@abdn.ac.uk

Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.022
 wR factor = 0.055
Data-to-parameter ratio = 19.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Propane-1,3-diaminium hydrogenarsenate
monohydrate

The title compound, $(\text{C}_3\text{H}_{12}\text{N}_2)[\text{HAsO}_4]\cdot\text{H}_2\text{O}$, contains a network of propane-1,3-diaminium cations, hydrogenarsenate anions [mean $\text{As}-\text{O} = 1.687$ (2) Å] and water molecules. The crystal packing involves anion-to-anion and water-to-anion $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, resulting in infinite chains containing the unusual $R_3^3(10)$ graph-set motif. Cation-to-anion and cation-to-water $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds generate a three-dimensional overall structure.

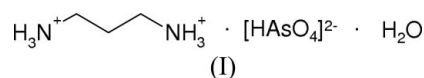
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Comment

The title compound, $(\text{C}_3\text{H}_{12}\text{N}_2)[\text{HAsO}_4]\cdot\text{H}_2\text{O}$, (I) (Fig. 1), was prepared as part of our ongoing structural studies of hydrogen-bonding interactions in protonated-amine (di)hydrogen arsenates (Lee & Harrison, 2003*a*; Wilkinson & Harrison, 2004; Todd & Harrison, 2005). In particular, (I) complements propane-1,3-diaminium bis(dihydrogenarsenate), $(\text{C}_3\text{H}_{12}\text{N}_2)[\text{H}_2\text{AsO}_4]_2$ (Wilkinson & Harrison, 2005), prepared under different pH conditions.



The $[\text{HAsO}_4]^{2-}$ hydrogenarsenate group in (I) has normal tetrahedral geometry [mean $\text{As}-\text{O} = 1.687$ (2) Å], with the protonated $\text{As1}-\text{O4}$ vertex showing its usual lengthening relative to the unprotonated $\text{As}-\text{O}$ bonds (Table 1). The propane-1,3-diaminium cation shows no unusual geometrical features.

As well as electrostatic attractions, the component species in (I) interact by means of a network of $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2). The $[\text{HAsO}_4]^{2-}$ units and water molecules are linked into polymeric chains (Fig. 2) propagating along [010] by way of anion-to-anion $\text{O4}-\text{H1}\cdots\text{O2}^i$ and water-to-anion $\text{O5}-\text{H14}\cdots\text{O1}$ and $\text{O5}-$

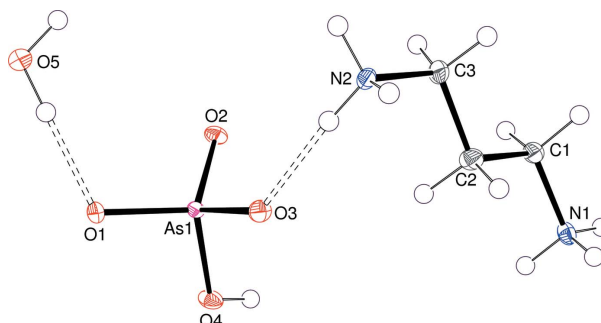


Figure 1

A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are indicated by dashed lines.

H15...O2ⁱⁱ bonds (Table 2). This arrangement results in an unusual $R_3^3(10)$ graph-set (Bernstein *et al.*, 1995) motif. The As1...As1ⁱ separation is 4.7991 (3) Å.

The organic species interacts with the hydrogenarsenate/water chains by way of six N—H...O hydrogen bonds [mean H...O = 1.89 Å, mean N—H...O = 171° and mean N...O = 2.793 (2) Å]. One of the acceptor O atoms is part of a water molecule, and the other five are parts of hydrogenarsenate groups. This hydrogen-bonding scheme results in a three-dimensional network (Fig. 3).

The hydrogen-bonded hydrogenarsenate/water chains in (I) are different from the motifs seen in related structures. In bis(cycloheptylaminium) hydrogenarsenate monohydrate (Todd & Harrison, 2005) and bis(benzylammonium) hydrogenarsenate monohydrate (Lee & Harrison, 2003c), hydrogen-bonded dimers of [HAsO₄]²⁻ units occur, with the dimers bridged into double chains by intervening water molecules. In the unhydrated piperidinium dihydrogenarsenate (Lee & Harrison, 2003b) and *t*-butylammonium dihydrogenarsenate (Wilkinson & Harrison, 2004), single chains of [H₂AsO₄]⁻ anions occur with each adjacent dihydrogenarsenate pair linked by a pair of hydrogen bonds. In propane-1,3-diaminium bis(dihydrogenarsenate) (Wilkinson & Harrison, 2005), the same organic cation as found in (I) is combined with dihydrogenarsenate [H₂AsO₄]⁻ groups, with the latter forming double chains.

Experimental

0.5 M aqueous propane-1,3-diamine solution (10 ml) was added to 0.5 M aqueous H₃AsO₄ solution (10 ml) to result in a clear solution. Aqueous ammonia was added to this solution to raise the pH to about 12, which is beyond the second end-point for H₃AsO₄ (*i.e.* the predominant species is [HAsO₄]²⁻). Platy crystals of (I) grew as the water evaporated over the course of a few days.

Crystal data

(C ₃ H ₁₂ N ₂)[HAsO ₄]·H ₂ O	$D_x = 1.778 \text{ Mg m}^{-3}$
$M_r = 234.09$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2043 reflections
$a = 7.1327 (2) \text{ \AA}$	$\theta = 2.9\text{--}27.5^\circ$
$b = 16.8046 (6) \text{ \AA}$	$\mu = 3.87 \text{ mm}^{-1}$
$c = 7.9402 (2) \text{ \AA}$	$T = 120 (2) \text{ K}$
$\beta = 113.253 (2)^\circ$	Plate, colourless
$V = 874.42 (5) \text{ \AA}^3$	$0.32 \times 0.24 \times 0.03 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	1804 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{int} = 0.036$
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$\theta_{max} = 27.5^\circ$
$T_{min} = 0.370, T_{max} = 0.892$	$h = -9 \rightarrow 8$
11562 measured reflections	$k = -20 \rightarrow 21$
2002 independent reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2 + 0.7899P]$
$R[F^2 > 2\sigma(F^2)] = 0.022$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.055$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.49 \text{ e \AA}^{-3}$
2002 reflections	$\Delta\rho_{min} = -0.53 \text{ e \AA}^{-3}$
103 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.0032 (6)

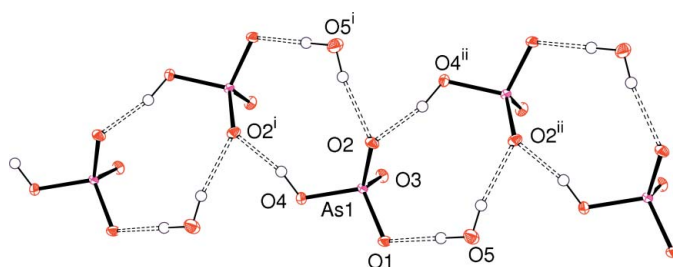


Figure 2
Detail of a hydrogen-bonded (dashed lines) hydrogenarsenate/water chain in (I).

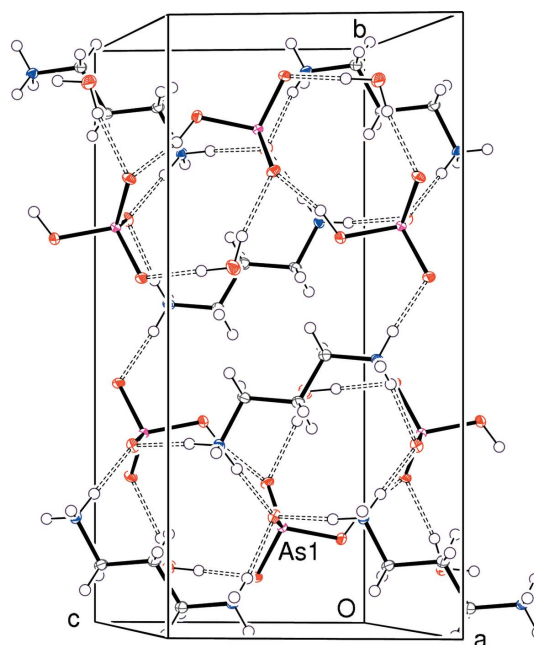


Figure 3
The crystal packing of (I). Dashed lines indicate hydrogen bonds.

Table 1
Selected geometric parameters (Å, °).

As1—O1	1.6612 (14)	As1—O3	1.6814 (14)
As1—O2	1.6746 (13)	As1—O4	1.7302 (13)
N1—C1—C2—C3	175.33 (16)	C1—C2—C3—N2	175.49 (16)

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

D—H...A	D—H	H...A	D...A	D—H...A
O4—H1...O2 ⁱ	0.93	1.71	2.6207 (19)	166
O5—H14...O1	0.92	1.79	2.709 (2)	177
O5—H15...O2 ⁱⁱ	0.89	1.98	2.858 (2)	169
N1—H2...O1 ⁱⁱⁱ	0.91	1.81	2.711 (2)	173
N1—H3...O3 ⁱ	0.91	1.96	2.855 (2)	166
N1—H4...O5 ^{iv}	0.91	1.90	2.798 (2)	168
N2—H11...O3 ⁱⁱ	0.91	1.90	2.802 (2)	170
N2—H12...O2 ^v	0.91	1.95	2.851 (2)	172
N2—H13...O3	0.91	1.84	2.743 (2)	175

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

The O-bound H atoms were found in difference maps and refined as riding on their carrier O atoms in their as-found relative positions. H atoms bonded to C and N atoms were placed in idealized positions (C–H = 0.99 Å and N–H = 0.91 Å) and refined as riding, allowing for free rotation of the –NH₃ groups. The constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ was applied in all cases.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*, *DENZO* (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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supporting information

Acta Cryst. (2005). E61, m1459–m1461 [https://doi.org/10.1107/S1600536805020702]

Propane-1,3-diaminium hydrogenarsenate monohydrate

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Propane-1,3-diaminium hydrogenarsenate monohydrate

Crystal data

(C₃H₁₂N₂)[HAsO₄]·H₂O

M_r = 234.09

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 7.1327 (2) Å

b = 16.8046 (6) Å

c = 7.9402 (2) Å

β = 113.253 (2)°

V = 874.42 (5) Å³

Z = 4

F(000) = 480

D_x = 1.778 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 2043 reflections

θ = 2.9–27.5°

μ = 3.87 mm⁻¹

T = 120 K

Plate, colourless

0.32 × 0.24 × 0.03 mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scans

Absorption correction: multi-scan
(SADABS; Bruker, 1999)

T_{min} = 0.370, *T_{max}* = 0.892

11562 measured reflections

2002 independent reflections

1804 reflections with *I* > 2σ(*I*)

R_{int} = 0.036

θ_{max} = 27.5°, θ_{min} = 3.0°

h = -9→8

k = -20→21

l = -10→10

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.022

wR(*F*²) = 0.055

S = 1.05

2002 reflections

103 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difmap (O-H) and geom
(others)

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0218*P*)² + 0.7899*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

Δρ_{max} = 0.49 e Å⁻³

Δρ_{min} = -0.53 e Å⁻³

Extinction correction: SHELXL97,

*F_c** = *kF_c*[1 + 0.001*xF_c*²λ³/sin(2θ)]^{-1/4}

Extinction coefficient: 0.0032 (6)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
As1	0.33625 (3)	0.169777 (11)	0.40666 (2)	0.00857 (9)
O1	0.2831 (2)	0.08464 (8)	0.48468 (19)	0.0146 (3)
O2	0.1852 (2)	0.24466 (8)	0.41487 (19)	0.0128 (3)
O3	0.5862 (2)	0.19099 (9)	0.51186 (19)	0.0128 (3)
O4	0.2905 (2)	0.15016 (8)	0.17988 (18)	0.0147 (3)
O5	0.1693 (2)	0.09842 (9)	0.7709 (2)	0.0209 (3)
N1	0.7723 (3)	0.44871 (10)	0.2130 (2)	0.0125 (3)
N2	0.7725 (3)	0.30904 (10)	0.7604 (2)	0.0108 (3)
C1	0.7251 (3)	0.45673 (12)	0.3780 (3)	0.0128 (4)
C2	0.7718 (3)	0.37949 (12)	0.4861 (3)	0.0140 (4)
C3	0.7418 (3)	0.38739 (12)	0.6644 (3)	0.0136 (4)
H1	0.2575	0.1935	0.1001	0.018*
H2	0.7457	0.4955	0.1504	0.015*
H3	0.6938	0.4095	0.1398	0.015*
H4	0.9065	0.4361	0.2476	0.015*
H5	0.5792	0.4703	0.3407	0.015*
H6	0.8075	0.5004	0.4562	0.015*
H7	0.9146	0.3637	0.5135	0.017*
H8	0.6814	0.3370	0.4107	0.017*
H9	0.8402	0.4266	0.7447	0.016*
H10	0.6023	0.4071	0.6384	0.016*
H11	0.7252	0.3118	0.8512	0.013*
H12	0.9079	0.2969	0.8097	0.013*
H13	0.7033	0.2706	0.6788	0.013*
H14	0.2035	0.0940	0.6706	0.025*
H15	0.1674	0.1492	0.8009	0.025*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.00955 (13)	0.00763 (13)	0.00873 (12)	-0.00002 (7)	0.00380 (9)	-0.00067 (7)
O1	0.0223 (8)	0.0088 (7)	0.0165 (7)	-0.0030 (6)	0.0117 (6)	-0.0006 (6)
O2	0.0129 (7)	0.0109 (7)	0.0148 (7)	0.0027 (6)	0.0057 (6)	-0.0021 (5)
O3	0.0095 (7)	0.0155 (7)	0.0127 (7)	-0.0013 (6)	0.0039 (6)	-0.0021 (6)
O4	0.0223 (8)	0.0119 (7)	0.0089 (7)	0.0029 (6)	0.0052 (6)	-0.0001 (6)
O5	0.0312 (9)	0.0169 (8)	0.0229 (8)	-0.0060 (7)	0.0195 (7)	-0.0049 (6)

N1	0.0148 (8)	0.0095 (8)	0.0116 (8)	-0.0023 (7)	0.0034 (7)	0.0008 (6)
N2	0.0112 (8)	0.0119 (8)	0.0105 (8)	-0.0013 (7)	0.0055 (7)	0.0005 (7)
C1	0.0143 (10)	0.0107 (10)	0.0139 (10)	0.0005 (8)	0.0061 (8)	0.0001 (8)
C2	0.0170 (10)	0.0114 (10)	0.0157 (10)	0.0038 (8)	0.0086 (9)	0.0018 (8)
C3	0.0172 (10)	0.0095 (10)	0.0160 (10)	-0.0001 (8)	0.0086 (9)	0.0017 (8)

Geometric parameters (Å, °)

As1—O1	1.6612 (14)	C2—C3	1.518 (3)
As1—O2	1.6746 (13)	C2—H7	0.9900
As1—O3	1.6814 (14)	C2—H8	0.9900
As1—O4	1.7302 (13)	C3—N2	1.493 (2)
O4—H1	0.9323	C3—H9	0.9900
N1—C1	1.482 (2)	C3—H10	0.9900
N1—H2	0.9100	N2—H11	0.9100
N1—H3	0.9100	N2—H12	0.9100
N1—H4	0.9100	N2—H13	0.9100
C1—C2	1.519 (3)	O5—H14	0.9237
C1—H5	0.9900	O5—H15	0.8880
C1—H6	0.9900		
O1—As1—O2	112.81 (7)	C1—C2—C3	111.85 (16)
O1—As1—O3	110.52 (7)	C1—C2—H7	109.2
O2—As1—O3	113.18 (7)	C3—C2—H7	109.2
O1—As1—O4	104.30 (7)	C1—C2—H8	109.2
O2—As1—O4	108.90 (7)	C3—C2—H8	109.2
O3—As1—O4	106.54 (7)	H7—C2—H8	107.9
As1—O4—H1	116.9	N2—C3—C2	110.64 (16)
C1—N1—H2	109.5	N2—C3—H9	109.5
C1—N1—H3	109.5	C2—C3—H9	109.5
H2—N1—H3	109.5	N2—C3—H10	109.5
C1—N1—H4	109.5	C2—C3—H10	109.5
H2—N1—H4	109.5	H9—C3—H10	108.1
H3—N1—H4	109.5	C3—N2—H11	109.5
N1—C1—C2	110.27 (16)	C3—N2—H12	109.5
N1—C1—H5	109.6	H11—N2—H12	109.5
C2—C1—H5	109.6	C3—N2—H13	109.5
N1—C1—H6	109.6	H11—N2—H13	109.5
C2—C1—H6	109.6	H12—N2—H13	109.5
H5—C1—H6	108.1	H14—O5—H15	110.2
N1—C1—C2—C3	175.33 (16)	C1—C2—C3—N2	175.49 (16)

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 O2 ⁱ	0.93	1.71	2.6207 (19)	166
O5—H14 \cdots O1	0.92	1.79	2.709 (2)	177

O5—H15···O2 ⁱⁱ	0.89	1.98	2.858 (2)	169
N1—H2···O1 ⁱⁱⁱ	0.91	1.81	2.711 (2)	173
N1—H3···O3 ⁱ	0.91	1.96	2.855 (2)	166
N1—H4···O5 ^{iv}	0.91	1.90	2.798 (2)	168
N2—H11···O3 ⁱⁱ	0.91	1.90	2.802 (2)	170
N2—H12···O2 ^v	0.91	1.95	2.851 (2)	172
N2—H13···O3	0.91	1.84	2.743 (2)	175

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