Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Propane-1,3-diaminium hydrogenarsenate monohydrate

Malcolm J. Todd and William T. A. Harrison*

Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland

Correspondence e-mail:
w.harrison@abdn.ac.uk

Received 6 June 2005
Accepted 29 June 2005
Online 6 July 2005

The title compound, $(C_3H_{12}N_2)[HAsO_4]\cdot H_2O$, contains a network of propane-1,3-diaminium cations, hydrogenarsenate anions [mean As—O = 1.687 (2) Å] and water molecules. The crystal packing involves anion-to-anion and water-to-anion O—H···O hydrogen bonds, resulting in infinite chains containing the unusual $R_3^3(10)$ graph-set motif. Cation-to-anion and cation-to-water N—H···O hydrogen bonds generate a three-dimensional overall structure.

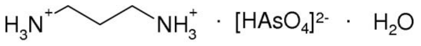
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.022
wR factor = 0.055
Data-to-parameter ratio = 19.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, $(C_3H_{12}N_2)[HAsO_4]\cdot H_2O$, (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), $(C_3H_{12}N_2)[H_2AsO_4]_2$ (Wilkinson & Harrison, 2005), prepared under different pH conditions.



(I)

The $[HAsO_4]^{2-}$ hydrogenarsenate group in (I) has normal tetrahedral geometry [mean As—O = 1.687 (2) Å], with the protonated As1—O4 vertex showing its usual lengthening relative to the unprotonated As—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 O—H···O and N—H···O hydrogen bonds (Table 2). The $[HAsO_4]^{2-}$ units and water molecules are linked into polymeric chains (Fig. 2) propagating along [010] by way of anion-to-anion O4—H1···O2^{i} and water-to-anion O5—H14···O1 and O5—

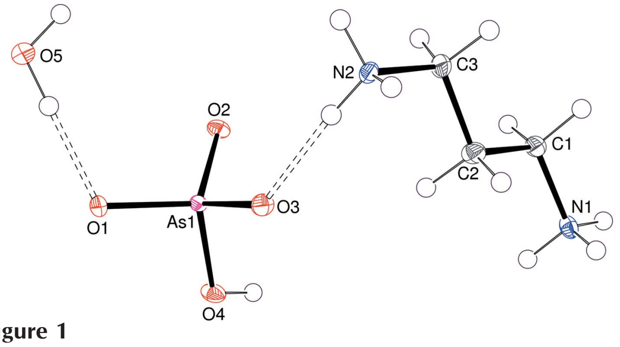


Figure 1
A view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are indicated by dashed lines.

H15$\cdots$O2^{ii} bonds (Table 2). This arrangement results in an unusual $R_3^3(10)$ graph-set (Bernstein *et al.*, 1995) motif. The As1$\cdots$As1^{i} separation is 4.7991 (3) Å.

The organic species interacts with the hydrogenarsenate/water chains by way of six N—H$\cdots$O hydrogen bonds [mean H$\cdots$O = 1.89 Å, mean N—H$\cdots$O = 171° and mean N$\cdots$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, 2003*c*), hydrogen-bonded dimers of $[HAsO_4]^{2-}$ units occur, with the dimers bridged into double chains by intervening water molecules. In the unhydrated piperidinium dihydrogenarsenate (Lee & Harrison, 2003*b*) and *t*-butylammonium dihydrogenarsenate (Wilkinson & Harrison, 2004), single chains of $[H_2AsO_4]^-$ 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 $[H_2AsO_4]^-$ 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 H_3AsO_4 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 H_3AsO_4 (*i.e.* the predominant species is $[HAsO_4]^{2-}$). Platy crystals of (I) grew as the water evaporated over the course of a few days.

Crystal data

$(C_3H_{12}N_2)[HAsO_4]\cdot H_2O$
$M_r = 234.09$
Monoclinic, $P2_1/c$
$a = 7.1327$ (2) Å
$b = 16.8046$ (6) Å
$c = 7.9402$ (2) Å
$\beta = 113.253$ (2)°
$V = 874.42$ (5) Å^3
$Z = 4$

$D_x = 1.778$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2043 reflections
$\theta = 2.9$–27.5°
$\mu = 3.87$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
0.32 × 0.24 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
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 $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 27.5°$
$h = -9 \rightarrow 8$
$k = -20 \rightarrow 21$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.055$
$S = 1.05$
2002 reflections
103 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2 + 0.7899P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.49$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0032 (6)

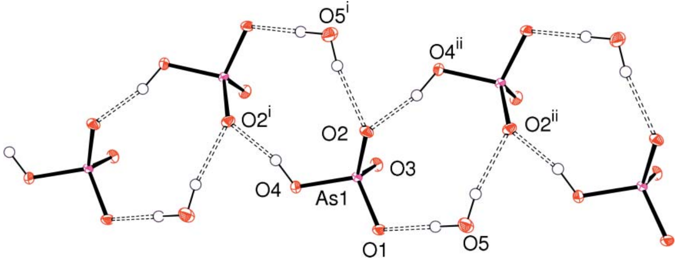


Figure 2
Detail of a hydrogen-bonded (dashed lines) hydrogenarsenate/water chain in (I).

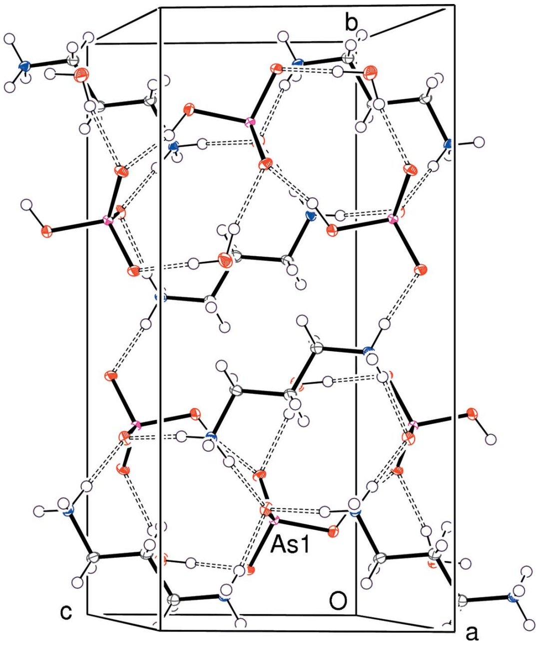


Figure 3
The crystal packing of (I). Dashed lines indicate hydrogen bonds.

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$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
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$\cdots$O2^{ii}	0.89	1.98	2.858 (2)	169
N1—H2$\cdots$O1^{iii}	0.91	1.81	2.711 (2)	173
N1—H3$\cdots$O3^{i}	0.91	1.96	2.855 (2)	166
N1—H4$\cdots$O5^{iv}	0.91	1.90	2.798 (2)	168
N2—H11$\cdots$O3^{ii}	0.91	1.90	2.802 (2)	170
N2—H12$\cdots$O2^{v}	0.91	1.95	2.851 (2)	172
N2—H13$\cdots$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 –NH_3 groups. The constraint $U_{iso}(H) = 1.2U_{eq}(carrier)$ 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*.

We thank the EPSRC National Crystallography Service (University of Southampton, England) for the data collection.

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.

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

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Lee, C. & Harrison, W. T. A. (2003*a*). *Acta Cryst.* E**59**, m739–m741.

Lee, C. & Harrison, W. T. A. (2003*b*). *Acta Cryst.* E**59**, m959–m960.

Lee, C. & Harrison, W. T. A. (2003*c*). *Acta Cryst.* E**59**, m1151–m1153.

Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Todd, M. J. & Harrison, W. T. A. (2005). *Acta Cryst.* E**61**, m1024–m1026.

Wilkinson, H. S. & Harrison, W. T. A. (2004). *Acta Cryst.* E**60**, m1359–m1361.

Wilkinson, H. S. & Harrison, W. T. A. (2005). *Acta Cryst.* E**61**, m1289–m1291.