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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.029
 wR factor = 0.059
Data-to-parameter ratio = 17.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

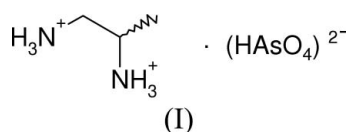
Propane-1,2-diaminium hydrogenarsenate

The title compound, $(\text{C}_3\text{H}_{12}\text{N}_2)[\text{AsHO}_4]$, is a molecular salt containing a network of propane-1,2-diaminium cations and hydrogenarsenate anions [mean As—O 1.686 (2) Å]. The crystal packing involves cation-to-anion N—H...O and anion-to-anion O—H...O hydrogen bonds, the latter resulting in dimeric associations of two adjacent hydrogenarsenate anions.

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Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing structural studies of hydrogen-bonding interactions in protonated-amine (di)hydrogenarsenates (Todd & Harrison, 2005).



The $[\text{HAsO}_4]^{2-}$ hydrogenarsenate group in (I) shows its normal tetrahedral geometry [mean As—O 1.686 (2) Å], with the protonated As1—O4 vertex showing its usual lengthening relative to the unprotonated As—O bonds (Table 1). The propane-1,2-diaminium cation is disordered over two overlapped positions (Fig. 1). This positional disorder manifests itself as a terminal methyl group (atoms C3 or C4) being attached to either C1 or C2, with 50% occupancy in each case. The N atoms and atoms C1 and C2 of the two orientations of the cation are not resolved. Allowing for the disorder, this ion is chiral, but crystal symmetry generates a 50:50 mix of enantiomers, which is consistent with the racemic starting material. Atoms N1 and N2 are close to being *trans* with respect to the C1—C2 backbone of the molecule (Table 1).

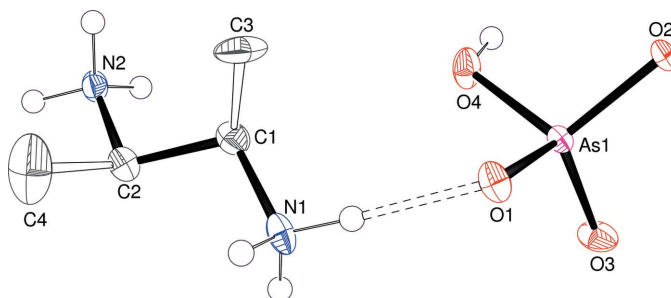


Figure 1

A view of (I), showing 50% probability displacement ellipsoids, with H atoms drawn as spheres of arbitrary radius. C-bound H atoms have been omitted for clarity and the hydrogen bond is indicated by a dashed line. Bonds to the disordered atoms C3 and C4 (see text) are shown as open lines.

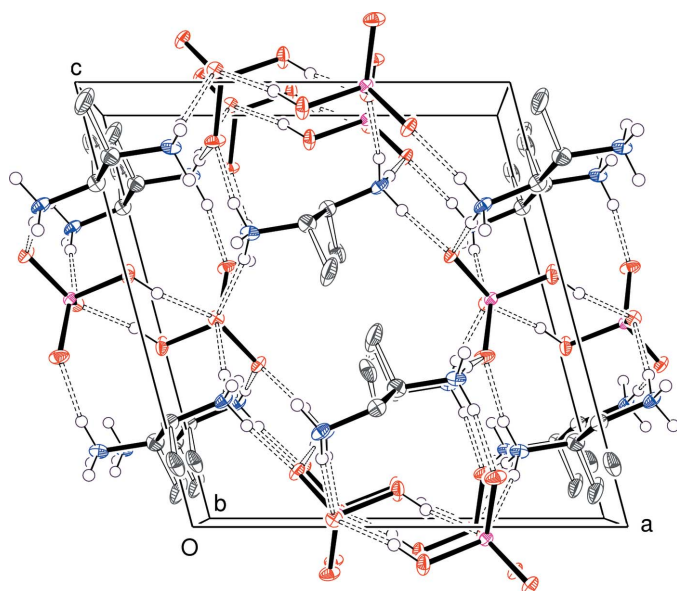


Figure 2
The packing for (I), with all C-bound H atoms omitted for clarity. Hydrogen bonds are indicated by dashed lines.

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 (HAsO₄)²⁻ units are linked into inversion-generated dimeric pairs by way of the O4—H1···O2ⁱⁱ bond (see Table 2 for symmetry code), with a resulting As1···As1ⁱ separation of 4.3963 (4) Å. This situation is distinct from that observed in related materials, where chains (Lee & Harrison, 2003) and sheets (Wilkinson & Harrison, 2005) of (di)hydrogenarsenate ions linked by O—H···O bonds are seen.

In (I), the organic species interacts with the hydrogenarsenate dimers by way of six N—H···O hydrogen bonds [mean H···O 1.85 Å, mean N—H···O 170° and mean N···O 2.744 (3) Å]. Atoms O1, O2 and O3 accept two N—H···O bonds each. This hydrogen-bonding scheme results in a three-dimensional network (Fig. 2).

Experimental

Aqueous propane-1,2-diamine solution (0.5 M, 10 ml) was added to aqueous H₃AsO₄ solution (0.5 M, 10 ml) to result in a clear mixture. 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 solution species is HAsO₄²⁻). Crystals of (I) grew as the water evaporated over the course of a few days.

Crystal data

(C₃H₁₂N₂)[AsHO₄]
M_r = 216.07
Monoclinic, P2₁/n
a = 10.9568 (4) Å
b = 6.4297 (3) Å
c = 11.5999 (5) Å
β = 104.816 (2)°
V = 790.03 (6) Å³
Z = 4
D_x = 1.817 Mg m⁻³

Mo Kα radiation
Cell parameters from 1952 reflections
θ = 2.9–27.5°
μ = 4.27 mm⁻¹
T = 120 (2) K
Shard (broken from plate), colourless
0.08 × 0.06 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 1999)
T_{min} = 0.726, T_{max} = 0.883
10517 measured reflections

1816 independent reflections
1533 reflections with I > 2σ(I)
R_{int} = 0.051
θ_{max} = 27.5°
h = -14 → 14
k = -8 → 8
l = -15 → 15

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.029
wR(F²) = 0.059
S = 1.11
1816 reflections
103 parameters
H-atom parameters constrained

w = 1/[σ²(F_o²) + (0.0164P)² + 0.583P]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} < 0.001
Δρ_{max} = 0.51 e Å⁻³
Δρ_{min} = -0.41 e Å⁻³
Extinction correction: SHELXL97 (Sheldrick, 1997)
Extinction coefficient: 0.0047 (7)

Table 1

Selected geometric parameters (Å, °).

As1—O1	1.6642 (17)	As1—O2	1.6817 (18)
As1—O3	1.6659 (18)	As1—O4	1.7336 (18)
N1—C1—C2—N2			
		-164.9 (2)	

Table 2

Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1···O2 ⁱ	0.93	1.76	2.679 (2)	170
N1—H2···O2 ⁱⁱ	0.91	1.87	2.765 (3)	168
N1—H3···O1 ⁱⁱⁱ	0.91	1.83	2.738 (3)	175
N1—H4···O1	0.91	1.81	2.716 (3)	177
N2—H5···O3 ^{iv}	0.91	1.82	2.713 (3)	168
N2—H6···O2 ^v	0.91	1.95	2.829 (3)	162
N2—H7···O3 ^{vi}	0.91	1.80	2.702 (3)	169

Symmetry codes: (i) -x + 1, -y, -z; (ii) x, y + 1, z; (iii) -x + ½, y + ½, -z + ½; (iv) -x + 1, -y + 1, -z; (v) x + ½, -y + ½, z + ½; (vi) x + ½, -y + ½, z + ½.

The organic cation is orientationally disordered, such that the two positions of atoms N1, N2, C1, and C2 overlap and cannot be resolved. The site-occupation factors of atoms C3 and C4 refined to 50% within experimental error and were both fixed at 0.50 for the final cycles of refinement. The O-bound H atom was found in a difference map and refined as riding in its as-found relative position. The H atoms bonded to C and N were located in idealized positions, with N—H = 0.91 Å and C—H = 0.98–0.99 Å, and refined as riding, allowing for free rotation of the -NH₃ groups. The constraint U_{iso}(H) = 1.2U_{eq}(carrier) or U_{iso}(H) = 1.5U_{eq}(methyl carrier) was applied.

Data collection: COLLECT (Nonius, 1998); cell refinement: HKL SCALEPACK (Otwinowski & Minor, 1997); data reduction: HKL DENZO (Otwinowski & Minor 1997), SCALEPACK and SORTAV (Blessing 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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