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Ethylenediammonium bis(dihydrogenarsenate): $R_4^3(14)$ loops built up from O—H···O hydrogen bonds

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The title compound, $C_2H_{10}N_2^{2+}\cdot 2H_2AsO_4^-$, contains a network of centrosymmetric doubly protonated ethylenediammonium cations and dihydogenarsenate anions. The component species interact by way of cation-to-anion N—H···O and anion-to-anion O—H···O hydrogen bonds, the latter leading to infinite (001) sheets of the $(H_2AsO_4)^-$ anions containing unusual $R_4^3(14)$ loops.

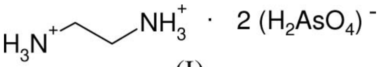
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.020
wR factor = 0.051
Data-to-parameter ratio = 27.0

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

Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing studies of hydrogen-bonding interactions in the molecular salts of arsenic oxo-anions (Wilkinson & Harrison, 2007).



(I)

The tetrahedral $H_2AsO_4^-$ anion in (I) [mean As—O = 1.677 (2) Å] shows the usual distinction (Table 1) between protonated and unprotonated As—O bond lengths, the latter showing formal partial double-bond character. The $C_2H_{10}N_2^{2+}$ dication is centrosymmetric.

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 $H_2AsO_4^-$ units are linked into infinite (001) sheets (Fig. 2) *via* the O—H···O bonds. The O2—H2···O3^{i} bond (see Table 2 for symmetry codes) results in [100] chains of translation-related tetrahedra. These [100] chains are cross-linked by the O1—H1···O3^{ii} bond, to result in a square grid of tetrahedra. The intertetrahedral As···As^{i} and As···As^{ii} separations are 4.8971 (4) and 4.8081 (4) Å, respectively.

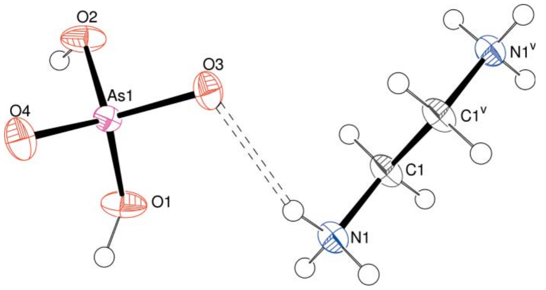


Figure 1
The structure of the ions of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radius. The hydrogen bond is indicated by a double-dashed line. [Symmetry code: (v) $2 - x, -y, 1 - z$.]

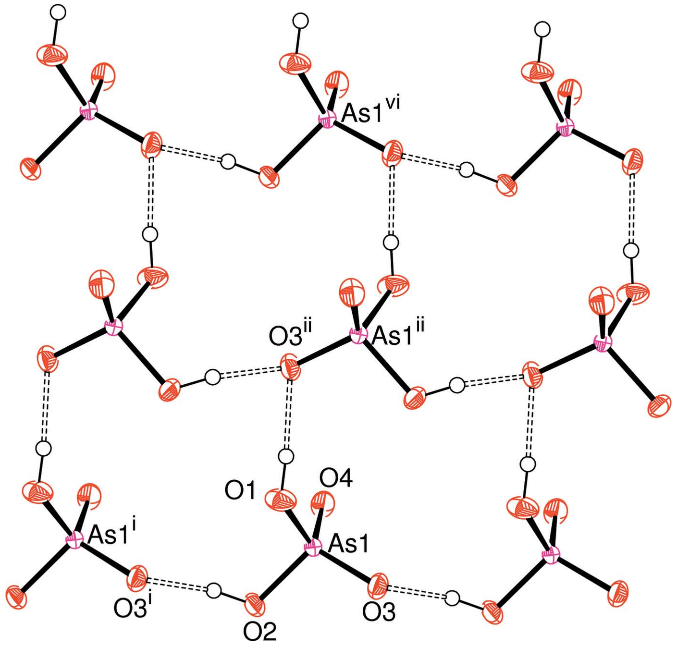


Figure 2
Detail of a part of a (100) hydrogen-bonded sheet of $H_2AsO_4^-$ groups in (I), with hydrogen bonds indicated by double-dashed lines. [Symmetry codes as in Table 2; additionally, (vi) x, $y + 1$, z.]

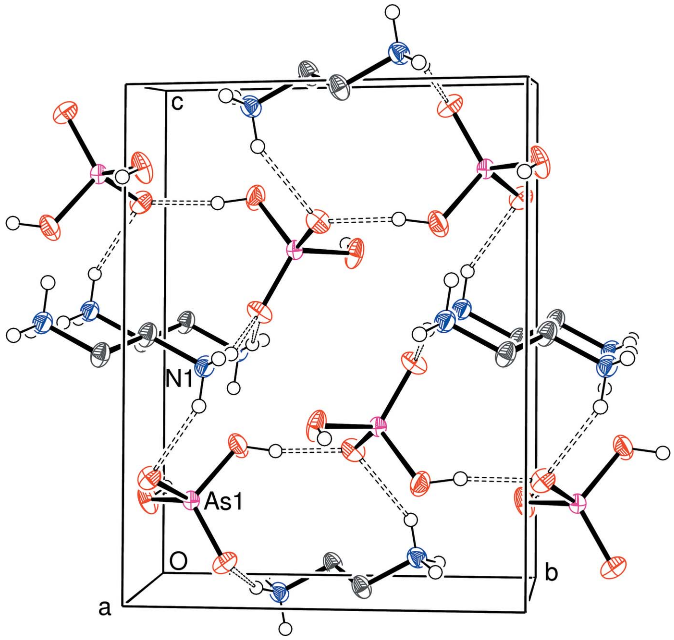


Figure 3
The packing of (I), showing the (001) dihydrogenarsenate layers mediated by the organic cations, with hydrogen bonds indicated by double-dashed lines.

An unusual $R_4^3(14)$ supramolecular loop (Bernstein *et al.*, 1995) arises from this hydrogen-bonding network. As the graph-set symbol implies, this arrangement is 'unbalanced', with atom O3 accepting two hydrogen bonds and atom O4 none. However, atom O4 accepts two N—H···O bonds from the organic cation, compared with one such interaction for atom O3.

The anionic sheets are bridged by the ethylenediammonium cations, which participate in three N—H···O interactions from each of their $–NH_3^+$ groups to nearby dihydrogenarsenate tetrahedra. This results (Fig. 3) in alternating organic and inorganic layers with respect to the *c* unit-cell direction.

These simple molecular salts show remarkable diversity in their inter-anion O—H···O bonding arrangements. Other ammonium (di)hydrogenarsenates contain isolated pairs of tetrahedra (Todd & Harrison, 2005), single chains of anions (Wilkinson & Harrison, 2005*a*), double chains of anions (Wilkinson & Harrison, 2005*b*) or three-dimensional arrays (Wilkinson & Harrison, 2006). The sheet structure seen here is different from other layered arrangements seen previously (*e.g.* Wilkinson & Harrison, 2007).

Experimental

Aqueous ethylenediamine solution (0.5 *M*, 10 ml) was added to aqueous H_3AsO_4 solution (0.5 *M*, 10 ml), resulting in a clear solution. Colourless blades and plates of (I) grew as the water evaporated over the course of a few days.

Crystal data

$C_2H_{10}N_2^{2+}\cdot 2AsH_2O_4^-$
$M_r = 343.98$
Monoclinic, $P2_1/c$
$a = 4.8971$ (3) Å
$b = 8.8054$ (5) Å
$c = 11.4894$ (6) Å
$\beta = 96.381$ (1)°

$V = 492.36$ (5) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 6.81$ mm^{-1}
$T = 293$ (2) K
0.41 × 0.16 × 0.05 mm

Data collection

Bruker SMART1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.166$, $T_{max} = 0.726$

5015 measured reflections
1783 independent reflections
1525 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.051$
$S = 1.06$
1783 reflections

66 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ e Å^{-3}

Table 1
Selected bond lengths (Å).

As1—O4	1.6372 (12)	As1—O2	1.7026 (13)
As1—O3	1.6532 (12)	As1—O1	1.7136 (13)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3^{i}	0.91	1.68	2.5810 (18)	168
O1—H1···O3^{ii}	0.89	1.81	2.689 (2)	171
N1—H5···O4^{iii}	0.89	1.91	2.7554 (19)	158
N1—H6···O3	0.89	2.18	2.978 (2)	149
N1—H7···O4^{iv}	0.89	2.02	2.8783 (19)	162

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

O-bound H atoms were found in a difference map and refined as riding on their carrier O atoms in their as-found relative positions. C- and N-bonded H atoms were placed in idealized positions, with C—H = 0.97 Å and N—H = 0.89 Å, and refined as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(carrier)$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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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