

Clare Lee 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  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.018  
 $wR$  factor = 0.042  
Data-to-parameter ratio = 31.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

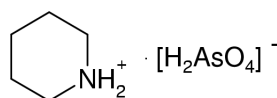
## Piperidinium dihydrogenarsenate

A combination of  $\text{O}-\text{H}\cdots\text{O}$  [ $d_{\text{av}}(\text{H}\cdots\text{O}) = 1.73\text{ \AA}$ ;  $\theta_{\text{av}}(\text{O}-\text{H}\cdots\text{O}) = 156^\circ$ ;  $d_{\text{av}}(\text{O}\cdots\text{O}) = 2.609(3)\text{ \AA}$ ] and  $\text{N}-\text{H}\cdots\text{O}$  [ $d_{\text{av}}(\text{H}\cdots\text{O}) = 1.85\text{ \AA}$ ;  $\theta_{\text{av}}(\text{N}-\text{H}\cdots\text{O}) = 169^\circ$ ;  $d_{\text{av}}(\text{N}\cdots\text{O}) = 2.740(2)\text{ \AA}$ ] hydrogen bonds defines a layered structure for the title compound,  $(\text{C}_5\text{H}_{12}\text{N})[\text{H}_2\text{AsO}_4]$ .

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## Comment

The title compound, (I) (Fig. 1), complements other simple ammonium arsenates, such as ethylenediammonium hydrogenarsenate,  $(\text{C}_2\text{H}_{10}\text{N}_2)[\text{HAsO}_4]$  (Averbuch-Pouchot & Durif, 1987) and propane-1,2-diammonium hydrogenarsenate hydrate,  $(\text{C}_3\text{H}_{12}\text{N}_2)[\text{HAsO}_4]\cdot\text{H}_2\text{O}$  (Lee & Harrison, 2003).



(I)

The structure of (I) consists of  $(\text{C}_5\text{H}_{12}\text{N})^+$  piperidinium cations and  $[\text{H}_2\text{AsO}_4]^-$  dihydrogenarsenate anions. The cation adopts a typical chair conformation with unexceptional geometrical parameters [ $d_{\text{av}}(\text{N}-\text{C}) = 1.484(3)\text{ \AA}$ ;  $d_{\text{av}}(\text{C}-\text{C}) = 1.515(3)\text{ \AA}$ ]. The anion shows its typical tetrahedral geometry [ $d_{\text{av}}(\text{As}-\text{O}) = 1.683(2)\text{ \AA}$ ], with the  $\text{As}-\text{O}$  bonds showing their expected lengthening relative to the  $\text{As}=\text{O}$  bonds.

As well as electrostatic attractions, the component species in (I) interact by means of  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  links (Table 2). The  $[\text{H}_2\text{AsO}_4]^-$  units are linked into a polymeric chain in the polar [010] direction by pairs of hydrogen bonds (Fig. 2) such that every  $[\text{H}_2\text{AsO}_4]^-$  tetrahedron makes an  $\text{O}-\text{H}\cdots\text{O}$  bond to each of its neighbours in the chain. This results in an  $\text{As}\cdots\text{As}^i$  (see Table 2 for symmetry code) separation of

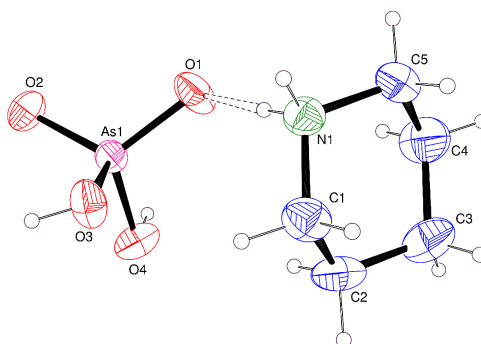


Figure 1

The asymmetric unit of (I) (30% displacement ellipsoids). H atoms are drawn as spheres of arbitrary radius and hydrogen bonds are indicated by dashed lines.

4.3424 (2) Å. The piperidinium cations crosslink the poly-(hydrogenarsenate) chains along [100] by way of N—H···O hydrogen bonds (Table 2). This results in (001) sheets (Fig. 3) that interact with each other by way of van der Waals forces. Overall, O1 accepts two N—H···O bonds [ $\theta(\text{H}\cdots\text{O}\cdots\text{H}) = 118^\circ$ ] and O2 accepts two O—H···O bonds [ $\theta(\text{H}\cdots\text{O}\cdots\text{H}) = 94^\circ$ ].

### Experimental

6 ml of an aqueous 1 M piperidine solution and 6 ml of an aqueous 0.5 M H<sub>3</sub>AsO<sub>4</sub> solution were mixed, resulting in a yellow solution which was sealed in a sample bottle. Block-like crystals of (I) grew over the course of a few weeks.

#### Crystal data

(C <sub>5</sub> H <sub>12</sub> N)[H <sub>2</sub> AsO <sub>4</sub> ]	$D_x = 1.676 \text{ Mg m}^{-3}$
$M_r = 227.09$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 4144 reflections
$a = 8.6593$ (4) Å	$\theta = 2.5\text{--}32.5^\circ$
$b = 6.3233$ (3) Å	$\mu = 3.75 \text{ mm}^{-1}$
$c = 8.9090$ (4) Å	$T = 293$ (2) K
$\beta = 112.676$ (1)°	Chunk, colourless
$V = 450.11$ (4) Å <sup>3</sup>	$0.56 \times 0.53 \times 0.36 \text{ mm}$
$Z = 2$	

#### Data collection

Bruker SMART1000 CCD diffractometer	3187 independent reflections
$\omega$ scans	2936 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$R_{\text{int}} = 0.014$
$T_{\text{min}} = 0.171$ , $T_{\text{max}} = 0.259$	$\theta_{\text{max}} = 32.5^\circ$
5328 measured reflections	$h = -12 \rightarrow 13$
	$k = -9 \rightarrow 9$
	$l = -11 \rightarrow 13$

#### Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$R[F^2 > 2\sigma(F^2)] = 0.018$	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
$wR(F^2) = 0.042$	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
$S = 1.00$	Extinction correction: SHELXL97
3187 reflections	Extinction coefficient: 0.0311 (18)
101 parameters	Absolute structure: Flack (1983), 1504 Friedel pairs
H-atom parameters constrained	Flack parameter = 0.063 (8)
$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

**Table 1**

Selected geometric parameters (Å).

As1—O1	1.6431 (11)	As1—O3	1.7100 (11)
As1—O2	1.6622 (8)	As1—O4	1.7161 (13)

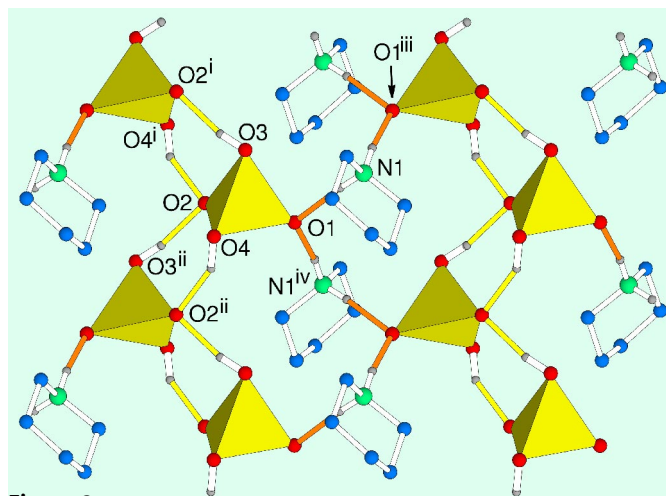
**Table 2**

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
O3—H1···O2 <sup>i</sup>	0.85	1.74	2.588 (2)	169
O4—H2···O2 <sup>ii</sup>	1.00	1.72	2.629 (3)	149
N1—H3···O1 <sup>iii</sup>	0.90	1.85	2.720 (2)	162
N1—H4···O1	0.90	1.86	2.759 (2)	176

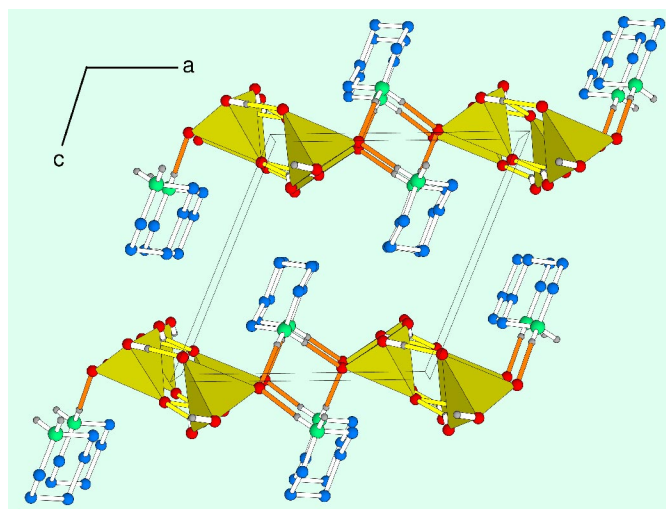
Symmetry codes: (i)  $2 - x, \frac{1}{2} + y, 2 - z$ ; (ii)  $2 - x, y - \frac{1}{2}, 2 - z$ ; (iii)  $1 - x, \frac{1}{2} + y, 2 - z$ .

The oxygen-bound H atoms were found in difference maps and were refined as riding in their as-found positions. H atoms bonded to C and N atoms were placed in calculated positions [ $d(\text{C—H}) = 0.97$  Å;  $d(\text{N—H}) = 0.90$  Å] and refined as riding. The constraint  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$  was applied in all cases.



**Figure 2**

Detail of a hydrogen-bonded piperidinium/dihydrogenarsenate (001) sheet in (I). Colour key: [H<sub>2</sub>AsO<sub>4</sub>]<sup>−</sup> tetrahedra yellow, O atoms red, C atoms blue, N atoms grey (all radii arbitrary). The H···O portions of the O—H···O and N—H···O hydrogen bonds are highlighted in yellow and orange, respectively. Symmetry codes are as in Table 2; additionally (iv)  $1 - x, y - \frac{1}{2}, 2 - z$ .



**Figure 3**

[010] projection of (I). Colour key as in Fig. 2.

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; molecular graphics: ORTEP-3 (Farrugia, 1997) and ATOMS (Shape Software, 1999); software used to prepare material for publication: SHELXL97.

### References

- Averbuch-Pouchot, M. T. & Durif, A. (1987). *Acta Cryst.* **C43**, 1894–1896.  
 Bruker (1999). SMART (Version 5.624), SAINT-Plus (Version 6.02A) and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Lee, C. & Harrison, W. T. A. (2003). *Acta Cryst.* **E59**, m739–m741.  
 Shape Software (1999). ATOMS. Shape Software, Kingsport, Tennessee, USA.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.