Acta Crystallographica Section E
**Structure Reports
Online**
ISSN 1600-5368

tert-Butylammonium dihydrogenarsenate

Hazel S. Wilkinson 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 28 July 2004
Accepted 25 August 2004
Online 4 September 2004

Key indicators

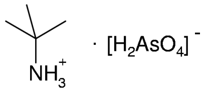
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.024
wR factor = 0.064
Data-to-parameter ratio = 33.3

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

The title compound, $(C_4H_{12}N)[H_2AsO_4]$, contains a network of *tert*-butylammonium cations and dihydrogenarsenate anions [d_{av}(As—O) = 1.682 (2) Å]. The crystal packing involves N—H···O [d_{av}(H···O) = 1.96 Å, θ_{av}(N—H···O) = 169° and d_{av}(N···O) = 2.837 (3) Å] and O—H···O [d_{av}(H···O) = 1.68 Å, θ_{av}(O—H···O) = 169° and d_{av}(O···O) Å] hydrogen bonds, resulting in a layered structure.

Comment

The title compound, (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*,*b*,*c*).


$$\mathrm{(CH_3)_3C{-}NH_3^+} \cdot [H_2AsO_4]^-$$

(I)

The $[H_2AsO_4]^-$ dihydrogenarsenate group in (I) shows its normal tetrahedral geometry [d_{av}(As—O) = 1.682 (2) Å], with the protonated As1—O3 and As1—O4 vertices showing their expected lengthening relative to the unprotonated As—O bonds, which have formal partial double-bond character (Table 1). The *tert*-butylammonium cation shows no unusual geometrical features.

As well as electrostatic attractions, the component species in (I) interact by means of a network of cation-to-anion N—H···O and anion-to-anion O—H···O hydrogen bonds (Table 2). The $[H_2AsO_4]^-$ units are linked into polymeric chains propagating along [010] by way of inversion-generated pairs of O—H···O bonds, alternately involving O3—H1···O1

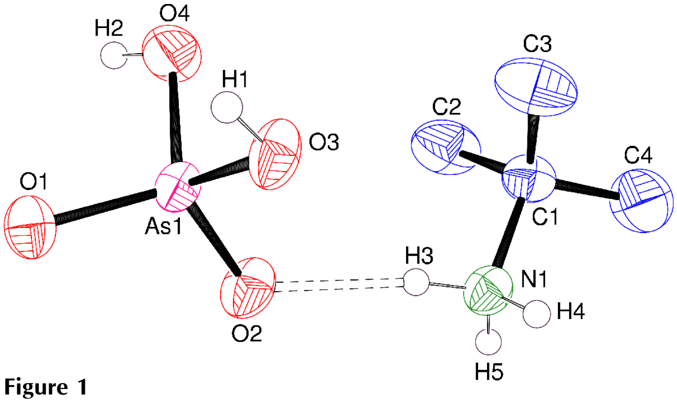


Figure 1
The asymmetric unit of (I) (50% displacement ellipsoids). H atoms are drawn as small spheres of arbitrary radius and the hydrogen bond is indicated by a dashed line. C—H H atoms have been omitted for clarity.

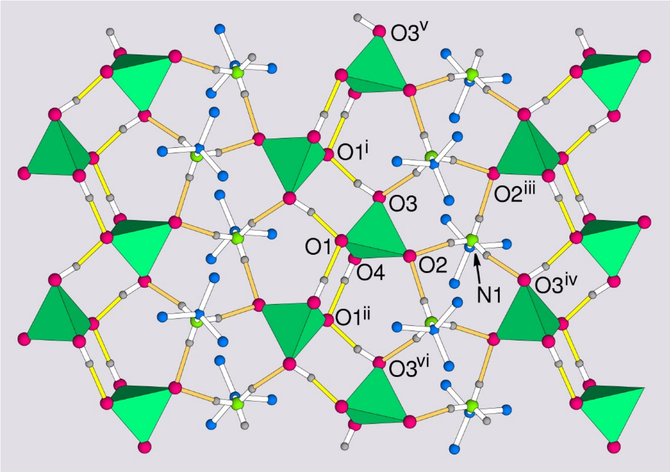


Figure 2
Detail of a hydrogen-bonded dihydrogenarsenate/*tert*-butylammonium sheet in (I). Colour key: $[H_2AsO_4]^-$ tetrahedra green, O atoms red, H atoms grey, C atoms blue, N atoms green (all radii arbitrary). The H···O portions of the O—H···O and N—H···O hydrogen bonds are highlighted in yellow and orange, respectively. Symmetry codes as in Table 2; additionally, (v) x,y − 1,z; (vi) *x*, 1 + *y*, *z*.

and O4—H2···O1 links (Fig. 2). This results in every $[H_2AsO_4]^-$ tetrahedron in the chain making one hydrogen bond to each of its neighbours and accepting one hydrogen bond from each neighbour. The As···As^i (*via* O3—H1···O1^i) and As···As^{ii} (*via* O4—H2···O1^{ii}) separations are 4.3002 (4) and 4.2662 (3) Å, respectively (see Table 2 for symmetry codes). Similar hydrogen-bonded chains of $[H_2AsO_4]^-$ anions have been seen in piperidinum dihydrogenarsenate, $(C_5H_{12}N)[H_2AsO_4]$ (Lee & Harrison, 2003*b*), although in this case they are generated by a 2_1 screw axis.

As shown in Table 2, the organic species interacts with the dihydrogenarsenate chains by way of three N—H···O hydrogen bonds [d_{av}(H···O) = 1.96 Å, θ_{av}(N—H···O) = 169° and d_{av}(N···O) = 2.837 (3) Å], such that each *tert*-butylammonium cation cross-links a dihydrogenarsenate chain to its neighbour by forming two hydrogen bonds to one chain, and one to the other. This results in neutral (101) layers (Fig. 3) of stoichiometry $(C_4H_{12}N)[H_2AsO_4]$, which interact with each other by van der Waals forces.

Experimental

An aqueous *tert*-butylamine solution (10 ml of 0.5 *M*) was added to a H_3AsO_4 solution (10 ml of 0.5 *M*), resulting in a clear solution. A mass of plate-shaped and rod-like crystals of (I) grew as the water evaporated over the course of a few days.

Crystal data

$(C_4H_{12}N)[H_2AsO_4]$
$M_r = 215.08$
Monoclinic, $P2_1/n$
$a = 9.7364$ (5) Å
$b = 6.3254$ (3) Å
$c = 14.2606$ (8) Å
$\beta = 94.864$ (1)°
$V = 875.10$ (8) Å^3
$Z = 4$
$D_x = 1.633$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3025 reflections
$\theta = 2.4$–32.4°
$\mu = 3.85$ mm^{-1}
$T = 293$ (2) K
Bar, colourless
0.55 × 0.15 × 0.05 mm

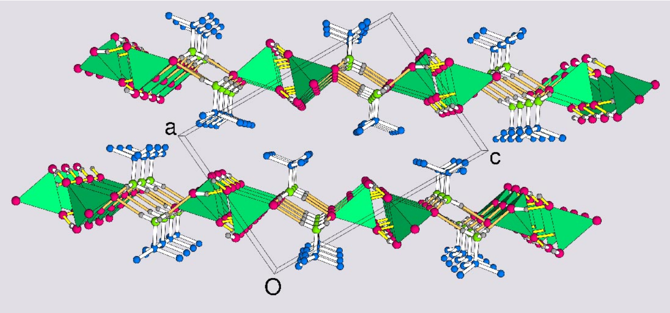


Figure 3
Projection of (I) onto (010). The colour key is as in Fig. 2.

Data collection

Bruker SMART1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.226$, $T_{max} = 0.831$
8742 measured reflections
3165 independent reflections
1807 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 32.5°$
$h = -10 \rightarrow 14$
$k = -7 \rightarrow 9$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.064$
$S = 0.88$
3165 reflections
95 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}

Table 1
Selected bond distances (Å).

As1—O2	1.6412 (13)	As1—O3	1.7061 (13)
As1—O1	1.6687 (13)	As1—O4	1.7101 (14)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O1^i	0.97	1.62	2.5752 (19)	166
O4—H2···O1^{ii}	0.94	1.74	2.6763 (19)	172
N1—H3···O2	0.89	1.92	2.801 (2)	168
N1—H4···O2^{iii}	0.89	1.88	2.765 (2)	173
N1—H5···O3^{iv}	0.89	2.07	2.944 (2)	166

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

The O—H H atoms were found in difference maps and refined by riding on their carrier O atoms in their as-found relative positions. H atoms bonded to C and N atoms were placed in calculated positions [d(C—H) = 0.96 Å and d(N—H) = 0.89 Å] and refined as riding, with the rigid NH_3 or CH_3 groups allowed to freely rotate about the bond joining the atoms in question to atom C1. The constraint U_{iso}(H) = $1.2U_{eq}$(O or N parent atom) or $1.5U_{eq}$(methyl C parent atom) was applied as appropriate.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *ATOMS* (Shape Software, 1999); software used to prepare material for publication: *SHELXL*97.

HSW thanks the Carnegie Trust for the Universities of Scotland for an undergraduate vacation studentship.

References

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.

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.

Shape Software (1999). *ATOMS*. Shape Software, 525 Hidden Valley Road, Kingsport, Tennessee, USA.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.