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Bis(cycloheptylaminium) hydrogenarsenate monohydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.028
wR factor = 0.068
Data-to-parameter ratio = 21.3

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

The title compound, $2C_7H_{16}N^+\cdot HAsO_4^{2-}\cdot H_2O$, contains a network of cycloheptylaminium cations, hydrogenarsenate anions and water molecules. The crystal packing involves N—H···O [average H···O = 1.86 Å, N—H···O = 172° and N···O = 2.756 (2) Å] and O—H···O [average H···O = 1.91 Å, O—H···O = 168° and O···O = 2.756 (2) Å] hydrogen bonds, resulting in a layered structure.

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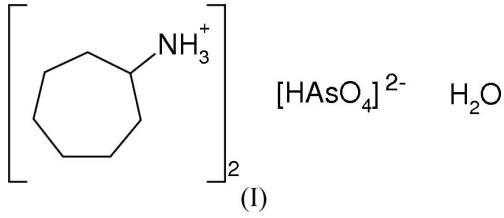
Comment

The title compound, (I) (Fig. 1), was prepared as part of our ongoing studies of hydrogen-bonding interactions in the crystal structures of (protonated) amine phosphates (Demir *et al.*, 2003), phosphites (Harrison, 2003), selenites (Ritchie & Harrison, 2003) and arsenates (Lee & Harrison, 2003*a*,*b*,*c*; Wilkinson & Harrison, 2004).



(I)

The crystal structure of (I) contains two unique $C_7H_{15}N^+$ cycloheptylaminium cations, one unique $HAsO_4^{2-}$ hydrogen-

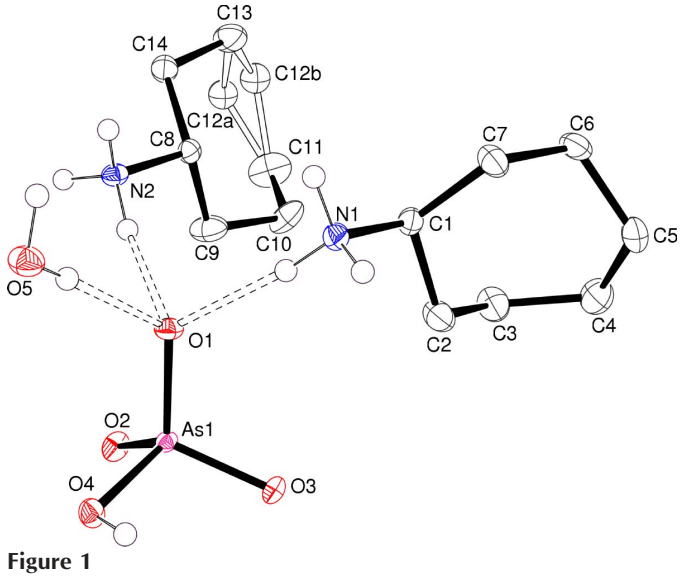


Figure 1
Asymmetric unit of (I), showing 50% displacement ellipsoids (arbitrary spheres for H atoms; C-bound H atoms have been omitted for clarity). Hydrogen bonds are indicated by dashed lines. Both disorder components are shown.

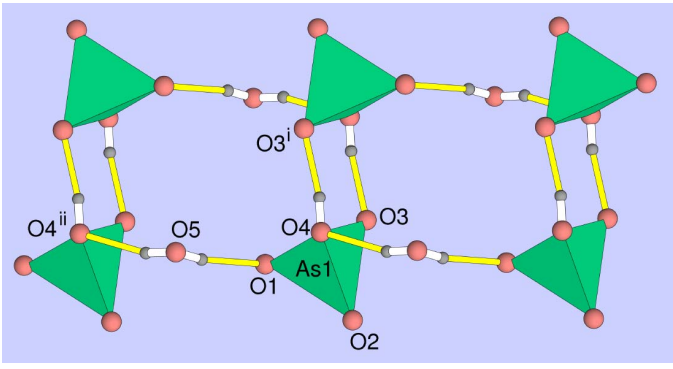


Figure 2
Detail of a hydrogen-bonded hydrogenarsenate/water chain in (I). Colour key: $[HAsO_4]^{2-}$ tetrahedra: green; O atoms: pink; H atoms: grey. The H···O portions of the hydrogen bonds are highlighted in yellow. Symmetry labels as in Table 2.

arsenate anion and one unique water molecule. The geometric parameters for the organic species are unexceptional. One of the C atoms of the C8-containing cation is disordered over two adjacent sites (see *Experimental*). The conformation of the C atoms of the undisordered (C1-containing) ring is close to a twist–chair (the predicted lowest-energy conformation for a seven-membered ring; Hendrickson, 1967) with a pseudo-twofold axis passing through C4 and the C1—C7 bond mid-point. The $HAsO_4^{2-}$ group in (I) shows its standard (Lee & Harrison, 2003) tetrahedral geometry [average As—O = 1.691 (2) Å], with the protonated As—O4 vertex showing its expected lengthening relative to the other As—O bonds.

As well as electrostatic attractions, the component species in (I) interact by means of a network of N—H···O and O—H···O hydrogen bonds (Table 2). The $HAsO_4^{2-}$ units and the water molecules (O5/H2/H3) are linked into a polymeric chain in the [010] direction by hydrogen bonds (Fig. 2). Inversion symmetry generates linked pairs of $HAsO_4^{2-}$ units (by way of two O4—H1···O3 bonds), which are in turn bridged by pairs of water molecules into a chain. The same chain motif occurs in bis(benzylaminium) hydrogenarsenate monohydrate (Lee & Harrison, 200*c*) but is different from that seen in propane-1,2-diaminium hydrogenarsenate monohydrate (Lee & Harrison, 2003*a*).

The organic species interact with the hydrogenarsenate/water chains by way of N—H···O hydrogen bonds (Table 2). All six of the $-NH_3^+$ H atoms are involved in these links [average H···O = 1.86 Å, N—H···O = 172° and N···O = 2.756 (2) Å]. Five of the acceptor O atoms are parts of $HAsO_4^{2-}$ species and one is part of a water molecule. This hydrogen-bonding scheme results in (101) hydrogenarsenate/water/ammonium layers sandwiched between the cycloheptyl moieties (Fig. 3), which interact in turn by way of van der Waals forces.

Experimental

A 0.5 *M* cycloheptylamine solution (10 ml) in cyclohexane was layered on top of a 0.5 *M* aqueous H_3AsO_4 solution (10 ml) and covered to prevent solvent evaporation. A mass of block-like crystals of (I) grew at the interface of the solvent layers over the course of a few days.

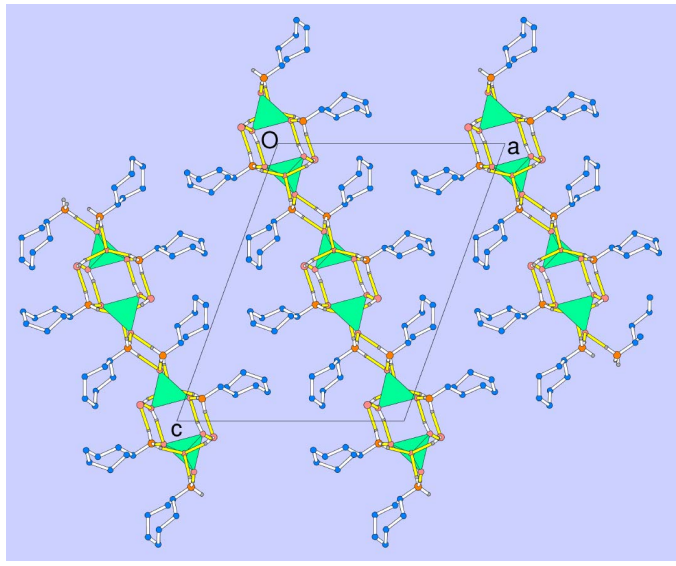


Figure 3
[010] projection of the unit cell packing for (I). Colour key as in Fig. 2; additionally, C atoms: blue; N atoms: orange. C-bound H atoms have been omitted for clarity.

Crystal data

$2C_7H_{16}N^+\cdot HAsO_4^{2-}\cdot H_2O$
M_r = 386.36
Monoclinic, $P2_1/n$
a = 15.5003 (4) Å
b = 6.4005 (1) Å
c = 20.1552 (5) Å
β = 110.0396 (11)°
V = 1878.53 (7) Å^3
Z = 4
D_x = 1.366 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4402 reflections
θ = 2.9–27.5°
μ = 1.83 mm^{-1}
T = 120 (2) K
Block, colourless
0.48 × 0.14 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.473, T_{max} = 0.810
17 560 measured reflections
4294 independent reflections
3604 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.6°
$h = -15 \rightarrow 20$
$k = -8 \rightarrow 7$
$l = -26 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.068
S = 1.04
4294 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2 + 1.7751P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0027 (3)

Table 1
Selected interatomic distances (Å).

As1—O2	1.6644 (13)	As1—O1	1.6789 (13)
As1—O3	1.6732 (13)	As1—O4	1.7466 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1···O3^{i}	0.87	1.77	2.6250 (19)	169
O5—H2···O4^{ii}	0.89	2.00	2.865 (2)	165
O5—H3···O1	0.83	1.96	2.779 (2)	171
N1—H4···O3^{ii}	0.91	1.83	2.735 (2)	175
N1—H5···O5^{iii}	0.91	1.90	2.805 (2)	173
N1—H6···O1	0.91	1.87	2.762 (2)	166
N2—H20···O1	0.91	1.91	2.794 (2)	165
N2—H21···O2^{iv}	0.91	1.84	2.744 (2)	177
N2—H22···O2^{ii}	0.91	1.79	2.697 (2)	173

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

Atom C12 is disordered over two adjacent sites [C12*a*···C12*b* = 0.606 (5) Å]. The two components were refined isotropically, together with a population ratio of 0.662 (15):0.338 (15). The O-bound H atoms were found in difference maps and refined as riding in their as-found relative positions (Table 2). The H atoms bonded to C and N atoms were placed in idealized positions [C—H = 0.99 and 1.00 Å, and N—H = 0.91 Å] and refined as riding, allowing for free rotation of the rigid –NH_3 groups about the C—N bonds. The constraint U_{iso}(H) = 1.2U_{eq}(carrier) was applied in all cases.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *ATOMS* (Shape Software, 1999); software used to prepare material for publication: *SHELXL97*.

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supporting information

Acta Cryst. (2005). E**61**, m1024–m1026 [https://doi.org/10.1107/S1600536805012912]

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Monoclinic, $P2_1/n$
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$a = 15.5003$ (4) Å
$b = 6.4005$ (1) Å
$c = 20.1552$ (5) Å
$\beta = 110.0396$ (11)°
$V = 1878.53$ (7) Å^3
$Z = 4$
$F(000) = 824$
$D_x = 1.366$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4402 reflections
$\theta = 2.9$–$27.5°$
$\mu = 1.83$ mm^{-1}
$T = 120$ K
Block, colourless
0.48 × 0.14 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
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Absorption correction: multi-scan (SADABS; Bruker, 2003)
$T_{min} = 0.473$, $T_{max} = 0.810$
17560 measured reflections
4294 independent reflections
3604 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.1°$
$h = -15\rightarrow20$
$k = -8\rightarrow7$
$l = -26\rightarrow25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.068$
$S = 1.04$
4294 reflections
202 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difmap (O-H) and geom (others)
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0232P)^2 + 1.7751P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0027 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
As1	0.414424 (12)	0.04918 (3)	0.390334 (10)	0.00982 (7)	
O1	0.41630 (9)	0.3104 (2)	0.38353 (7)	0.0144 (3)	
O2	0.34375 (9)	−0.0597 (2)	0.31679 (7)	0.0159 (3)	
O3	0.52048 (9)	−0.0513 (2)	0.41707 (7)	0.0137 (3)	
O4	0.36561 (9)	−0.0135 (2)	0.45409 (7)	0.0151 (3)	
H1	0.4078	−0.0045	0.4955	0.018*	
O5	0.31260 (10)	0.5555 (2)	0.44185 (8)	0.0207 (3)	
H2	0.3219	0.6907	0.4373	0.025*	
H3	0.3389	0.4858	0.4200	0.025*	
N1	0.57607 (11)	0.5423 (3)	0.41955 (9)	0.0154 (4)	
H4	0.5549	0.6759	0.4161	0.018*	
H5	0.6082	0.5124	0.4656	0.018*	
H6	0.5278	0.4528	0.4031	0.018*	
C1	0.63747 (13)	0.5189 (3)	0.37659 (11)	0.0152 (4)	
H7	0.6015	0.5565	0.3266	0.018*	
C2	0.66708 (15)	0.2909 (3)	0.37832 (13)	0.0236 (5)	
H8	0.6124	0.2002	0.3685	0.028*	
H9	0.7096	0.2571	0.4263	0.028*	
C3	0.71461 (15)	0.2430 (4)	0.32452 (13)	0.0250 (5)	
H10	0.7024	0.0954	0.3096	0.030*	
H11	0.6866	0.3313	0.2823	0.030*	
C4	0.81885 (15)	0.2788 (4)	0.35154 (14)	0.0291 (5)	
H12	0.8487	0.1568	0.3806	0.035*	
H13	0.8397	0.2834	0.3103	0.035*	
C5	0.85239 (15)	0.4751 (4)	0.39497 (14)	0.0276 (5)	
H14	0.9137	0.5109	0.3933	0.033*	
H15	0.8600	0.4437	0.4448	0.033*	
C6	0.79054 (15)	0.6666 (3)	0.37201 (12)	0.0220 (5)	
H16	0.8288	0.7941	0.3852	0.026*	
H17	0.7616	0.6653	0.3199	0.026*	
C7	0.71543 (14)	0.6770 (3)	0.40484 (12)	0.0211 (5)	
H18	0.6887	0.8192	0.3973	0.025*	
H19	0.7441	0.6561	0.4564	0.025*	
N2	0.33232 (11)	0.5513 (3)	0.26297 (8)	0.0134 (3)	

H20	0.3537	0.4533	0.2974	0.016*	
H21	0.2736	0.5194	0.2357	0.016*	
H22	0.3335	0.6791	0.2831	0.016*	
C8	0.39174 (13)	0.5549 (3)	0.21807 (10)	0.0137 (4)	
H23	0.4534	0.6088	0.2478	0.016*	
C9	0.40445 (17)	0.3331 (3)	0.19637 (13)	0.0275 (5)	
H24	0.3450	0.2814	0.1635	0.033*	
H25	0.4223	0.2428	0.2388	0.033*	
C10	0.47663 (16)	0.3127 (4)	0.16093 (12)	0.0284 (5)	
H26	0.5279	0.4092	0.1845	0.034*	
H27	0.5014	0.1687	0.1682	0.034*	
C11	0.4413 (2)	0.3594 (4)	0.08213 (13)	0.0374 (6)	
H28	0.4954	0.3698	0.0668	0.045*	
H29	0.4055	0.2364	0.0579	0.045*	
C12A	0.3841 (4)	0.5461 (7)	0.0554 (2)	0.0246 (12)*	0.662 (15)
H30A	0.3188	0.5066	0.0435	0.029*	0.662 (15)
H31A	0.3928	0.5919	0.0112	0.029*	0.662 (15)
C12B	0.4204 (9)	0.5906 (14)	0.0611 (4)	0.026 (3)*	0.338 (15)
H30B	0.4717	0.6443	0.0472	0.032*	0.338 (15)
H31B	0.3650	0.5922	0.0180	0.032*	0.338 (15)
C13	0.40528 (17)	0.7385 (4)	0.10873 (13)	0.0287 (5)	
H32	0.4718	0.7447	0.1362	0.034*	
H33	0.3872	0.8710	0.0822	0.034*	
C14	0.35107 (14)	0.7092 (3)	0.15809 (11)	0.0203 (5)	
H34	0.2884	0.6619	0.1299	0.024*	
H35	0.3452	0.8466	0.1787	0.024*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.00959 (10)	0.00863 (10)	0.00972 (11)	0.00033 (8)	0.00137 (7)	−0.00013 (8)
O1	0.0172 (7)	0.0093 (7)	0.0159 (7)	−0.0002 (6)	0.0046 (6)	0.0008 (6)
O2	0.0145 (7)	0.0156 (7)	0.0134 (7)	−0.0003 (6)	−0.0005 (6)	−0.0060 (6)
O3	0.0108 (6)	0.0147 (7)	0.0146 (7)	0.0037 (6)	0.0029 (5)	0.0001 (6)
O4	0.0121 (6)	0.0197 (8)	0.0121 (7)	−0.0018 (6)	0.0022 (5)	0.0014 (6)
O5	0.0266 (8)	0.0158 (7)	0.0227 (8)	−0.0007 (6)	0.0124 (7)	−0.0009 (6)
N1	0.0133 (8)	0.0136 (8)	0.0186 (9)	−0.0003 (7)	0.0047 (7)	−0.0008 (7)
C1	0.0134 (9)	0.0166 (10)	0.0157 (10)	0.0008 (8)	0.0051 (8)	0.0005 (8)
C2	0.0241 (11)	0.0181 (11)	0.0335 (13)	−0.0022 (9)	0.0159 (10)	−0.0024 (10)
C3	0.0254 (11)	0.0218 (12)	0.0314 (13)	−0.0009 (10)	0.0142 (10)	−0.0076 (10)
C4	0.0254 (12)	0.0258 (13)	0.0388 (15)	0.0053 (10)	0.0147 (11)	−0.0021 (11)
C5	0.0150 (10)	0.0273 (12)	0.0416 (15)	−0.0014 (9)	0.0110 (10)	0.0024 (11)
C6	0.0231 (11)	0.0164 (11)	0.0305 (13)	−0.0030 (9)	0.0145 (10)	0.0028 (9)
C7	0.0203 (10)	0.0196 (11)	0.0262 (12)	−0.0039 (9)	0.0116 (9)	−0.0021 (9)
N2	0.0149 (8)	0.0114 (8)	0.0118 (8)	0.0001 (7)	0.0018 (7)	0.0001 (7)
C8	0.0124 (9)	0.0143 (10)	0.0134 (10)	−0.0007 (8)	0.0030 (8)	−0.0008 (8)
C9	0.0410 (14)	0.0174 (12)	0.0298 (13)	0.0060 (10)	0.0194 (11)	0.0021 (10)
C10	0.0313 (13)	0.0297 (13)	0.0243 (13)	0.0158 (10)	0.0099 (10)	0.0021 (10)

C11	0.0540 (17)	0.0346 (14)	0.0255 (14)	0.0073 (13)	0.0161 (12)	−0.0008 (12)
C13	0.0346 (13)	0.0261 (12)	0.0317 (14)	0.0064 (11)	0.0195 (11)	0.0107 (11)
C14	0.0182 (10)	0.0222 (11)	0.0208 (11)	0.0013 (9)	0.0071 (9)	0.0058 (9)

Geometric parameters (Å, º)

As1—O2	1.6644 (13)	C7—H19	0.9900
As1—O3	1.6732 (13)	N2—C8	1.496 (3)
As1—O1	1.6789 (13)	N2—H20	0.9100
As1—O4	1.7466 (14)	N2—H21	0.9100
O4—H1	0.8680	N2—H22	0.9100
O5—H2	0.8874	C8—C9	1.518 (3)
O5—H3	0.8255	C8—C14	1.521 (3)
N1—C1	1.498 (3)	C8—H23	1.0000
N1—H4	0.9100	C9—C10	1.525 (3)
N1—H5	0.9100	C9—H24	0.9900
N1—H6	0.9100	C9—H25	0.9900
C1—C2	1.526 (3)	C10—C11	1.522 (3)
C1—C7	1.529 (3)	C10—H26	0.9900
C1—H7	1.0000	C10—H27	0.9900
C2—C3	1.536 (3)	C11—C12A	1.476 (4)
C2—H8	0.9900	C11—C12B	1.543 (8)
C2—H9	0.9900	C11—H28	0.9900
C3—C4	1.535 (3)	C11—H29	0.9900
C3—H10	0.9900	C12A—C13	1.593 (5)
C3—H11	0.9900	C12A—H30A	0.9900
C4—C5	1.518 (3)	C12A—H31A	0.9900
C4—H12	0.9900	C12B—C13	1.424 (8)
C4—H13	0.9900	C12B—H30B	0.9900
C5—C6	1.527 (3)	C12B—H31B	0.9900
C5—H14	0.9900	C13—C14	1.518 (3)
C5—H15	0.9900	C13—H32	0.9900
C6—C7	1.525 (3)	C13—H33	0.9900
C6—H16	0.9900	C14—H34	0.9900
C6—H17	0.9900	C14—H35	0.9900
C7—H18	0.9900		

O2—As1—O3	113.58 (7)	C8—N2—H22	109.5
O2—As1—O1	111.64 (7)	H20—N2—H22	109.5
O3—As1—O1	111.51 (7)	H21—N2—H22	109.5
O2—As1—O4	103.90 (7)	N2—C8—C9	109.12 (16)
O3—As1—O4	107.47 (7)	N2—C8—C14	108.51 (15)
O1—As1—O4	108.23 (7)	C9—C8—C14	115.96 (18)
As1—O4—H1	108.6	N2—C8—H23	107.7
H2—O5—H3	110.2	C9—C8—H23	107.7
C1—N1—H4	109.5	C14—C8—H23	107.7
C1—N1—H5	109.5	C8—C9—C10	113.79 (19)
H4—N1—H5	109.5	C8—C9—H24	108.8

C1—N1—H6	109.5	C10—C9—H24	108.8
H4—N1—H6	109.5	C8—C9—H25	108.8
H5—N1—H6	109.5	C10—C9—H25	108.8
N1—C1—C2	109.17 (16)	H24—C9—H25	107.7
N1—C1—C7	107.23 (16)	C11—C10—C9	114.4 (2)
C2—C1—C7	115.55 (17)	C11—C10—H26	108.7
N1—C1—H7	108.2	C9—C10—H26	108.7
C2—C1—H7	108.2	C11—C10—H27	108.7
C7—C1—H7	108.2	C9—C10—H27	108.7
C1—C2—C3	112.84 (19)	H26—C10—H27	107.6
C1—C2—H8	109.0	C12A—C11—C10	119.8 (2)
C3—C2—H8	109.0	C12A—C11—C12B	23.0 (3)
C1—C2—H9	109.0	C10—C11—C12B	116.2 (3)
C3—C2—H9	109.0	C12A—C11—H28	107.4
H8—C2—H9	107.8	C10—C11—H28	107.4
C4—C3—C2	114.9 (2)	C12B—C11—H28	88.3
C4—C3—H10	108.5	C12A—C11—H29	107.4
C2—C3—H10	108.5	C10—C11—H29	107.4
C4—C3—H11	108.5	C12B—C11—H29	126.7
C2—C3—H11	108.5	H28—C11—H29	106.9
H10—C3—H11	107.5	C11—C12A—C13	114.9 (3)
C5—C4—C3	116.05 (19)	C11—C12A—H30A	108.6
C5—C4—H12	108.3	C13—C12A—H30A	108.6
C3—C4—H12	108.3	C11—C12A—H31A	108.6
C5—C4—H13	108.3	C13—C12A—H31A	108.6
C3—C4—H13	108.3	H30A—C12A—H31A	107.5
H12—C4—H13	107.4	C13—C12B—C11	121.3 (5)
C4—C5—C6	115.7 (2)	C13—C12B—H30B	107.1
C4—C5—H14	108.3	C11—C12B—H30B	107.3
C6—C5—H14	108.3	C13—C12B—H31B	106.8
C4—C5—H15	108.3	C11—C12B—H31B	106.8
C6—C5—H15	108.3	H30B—C12B—H31B	106.7
H14—C5—H15	107.4	C12B—C13—C14	128.0 (5)
C7—C6—C5	113.38 (19)	C12B—C13—C12A	22.3 (4)
C7—C6—H16	108.9	C14—C13—C12A	108.3 (3)
C5—C6—H16	108.9	C12B—C13—H32	91.3
C7—C6—H17	108.9	C14—C13—H32	110.0
C5—C6—H17	108.9	C12A—C13—H32	110.0
H16—C6—H17	107.7	C12B—C13—H33	106.6
C6—C7—C1	115.67 (18)	C14—C13—H33	110.0
C6—C7—H18	108.4	C12A—C13—H33	110.0
C1—C7—H18	108.4	H32—C13—H33	108.4
C6—C7—H19	108.4	C13—C14—C8	115.45 (18)
C1—C7—H19	108.4	C13—C14—H34	108.4
H18—C7—H19	107.4	C8—C14—H34	108.4
C8—N2—H20	109.5	C13—C14—H35	108.4
C8—N2—H21	109.5	C8—C14—H35	108.4
H20—N2—H21	109.5	H34—C14—H35	107.5

N1—C1—C2—C3	168.86 (17)	C9—C10—C11—C12B	71.9 (6)
C7—C1—C2—C3	−70.2 (2)	C10—C11—C12A—C13	30.6 (6)
C1—C2—C3—C4	88.2 (2)	C12B—C11—C12A—C13	−56.9 (9)
C2—C3—C4—C5	−42.1 (3)	C12A—C11—C12B—C13	84.6 (12)
C3—C4—C5—C6	−36.1 (3)	C10—C11—C12B—C13	−20.2 (12)
C4—C5—C6—C7	86.7 (3)	C11—C12B—C13—C14	−41.0 (12)
C5—C6—C7—C1	−71.9 (3)	C11—C12B—C13—C12A	−72.1 (12)
N1—C1—C7—C6	175.18 (17)	C11—C12A—C13—C12B	69.5 (9)
C2—C1—C7—C6	53.2 (3)	C11—C12A—C13—C14	−85.1 (4)
N2—C8—C9—C10	−170.24 (17)	C12B—C13—C14—C8	66.8 (6)
C14—C8—C9—C10	66.9 (3)	C12A—C13—C14—C8	78.8 (3)
C8—C9—C10—C11	−84.1 (3)	N2—C8—C14—C13	177.73 (18)
C9—C10—C11—C12A	46.1 (4)	C9—C8—C14—C13	−59.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H1···O3^{i}	0.87	1.77	2.6250 (19)	169
O5—H2···O4^{ii}	0.89	2.00	2.865 (2)	165
O5—H3···O1	0.83	1.96	2.779 (2)	171
N1—H4···O3^{ii}	0.91	1.83	2.735 (2)	175
N1—H5···O5^{iii}	0.91	1.90	2.805 (2)	173
N1—H6···O1	0.91	1.87	2.762 (2)	166
N2—H20···O1	0.91	1.91	2.794 (2)	165
N2—H21···O2^{iv}	0.91	1.84	2.744 (2)	177
N2—H22···O2^{ii}	0.91	1.79	2.697 (2)	173

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) *x*, *y*+1, *z*; (iii) −*x*+1, −*y*+1, −*z*+1; (iv) −*x*+1/2, *y*+1/2, −*z*+1/2.