STRUCTURAL CHEMISTRY

ISSN 2053-2296

The coordination complex structures and hydrogen bonding in the three-dimensional alkaline earth metal salts (Mg, Ca, Sr and Ba) of (4-aminophenyl)arsonic acid

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Received 26 September 2016
Accepted 5 December 2016

Edited by A. G. Oliver, University of Notre Dame, USA

Keywords: *p*-arsanilic acid salts; alkaline earth metals; coordination polymers; hydrogen bonding; crystal structure; one-dimensional coordination polymer; barium; strontium; calcium; magnesium.

CCDC references: 1520680; 1520679; 1520678; 1520677

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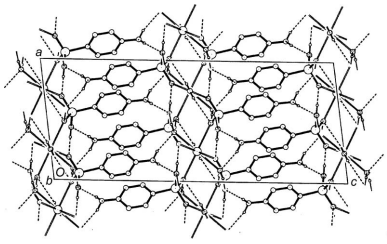
(4-Aminophenyl)arsonic acid (*p*-arsanilic acid) is used as an antihelminth in veterinary applications and was earlier used in the monosodium salt dihydrate form as the antisyphilitic drug atoxyl. Examples of complexes with this acid are rare. The structures of the alkaline earth metal (Mg, Ca, Sr and Ba) complexes with (4-aminophenyl)arsonic acid (*p*-arsanilic acid) have been determined, *viz.* hexaaquamagnesium bis[hydrogen (4-aminophenyl)arsonate] tetrahydrate, $[Mg(H_2O)_6](C_6H_7AsNO_3)\cdot 4H_2O$, (I), *catena*-poly[[[diaquacalcium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O']-[diaquacalcium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O]] dihydrate], $\{[Ca(C_6H_7AsNO_3)_2(H_2O)_2]\cdot 2H_2O\}_n$, (II), *catena*-poly[[triaquastrontium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O']], $[Sr(C_6H_7AsNO_3)_2(H_2O)_3]_n$, (III), and *catena*-poly[[triaquabarium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O']], $[Ba(C_6H_7AsNO_3)_2(H_2O)_3]_n$, (IV). In the structure of magnesium salt (I), the centrosymmetric octahedral $[Mg(H_2O)_6]^{2+}$ cation, the two hydrogen *p*-arsanilate anions and the four water molecules of solvation form a three-dimensional network structure through inter-species O—H and N—H hydrogen-bonding interactions with water and arsonate O-atom and amine N-atom acceptors. In one-dimensional coordination polymer (II), the distorted octahedral CaO_6 coordination polyhedron comprises two *trans*-related water molecules and four arsonate O-atom donors from bridging hydrogen arsanilate ligands. One bridging extension is four-membered *via* a single O atom and the other is eight-membered *via* O:O'-bridging, both across inversion centres, giving a chain coordination polymer extending along the [100] direction. Extensive hydrogen-bonding involving O—H···O, O—H···N and N—H···O interactions gives an overall three-dimensional structure. The structures of the polymeric Sr and Ba complexes (III) and (IV), respectively, are isotypic and are based on irregular MO_7 coordination polyhedra about the M^{2+} centres, which lie on twofold rotation axes along with one of the coordinated water molecules. The coordination centres are linked through inversion-related arsonate O:O'-bridges, giving eight-membered ring motifs and forming coordination polymeric chains extending along the [100] direction. Inter-chain N—H···O and O—H···O hydrogen-bonding interactions extend the structures into three dimensions and the crystal packing includes π–π ring interactions [minimum ring centroid separations = 3.4666 (17) Å for (III) and 3.4855 (8) Å for (IV)].



1. Introduction

The compound (4-aminophenyl)arsonic acid (or *p*-arsanilic acid) has a continuing usage as an antihelminth in veterinary applications (Steverding, 2010) and was earlier used in the monosodium salt dihydrate form as the antisyphilitic drug atoxyl (Ehrlich & Bertheim, 1907; O'Neil, 2001; Bosch & Rosich, 2008). We reported the crystal structure of atoxyl,

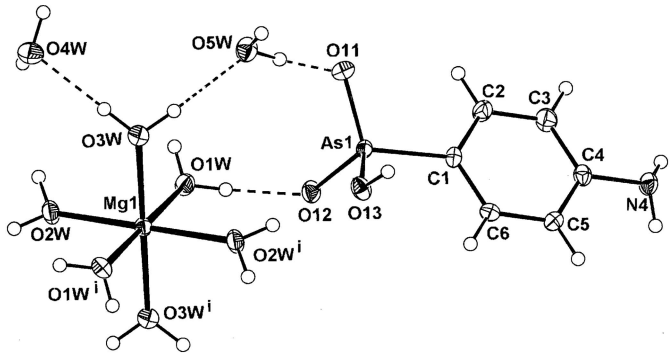
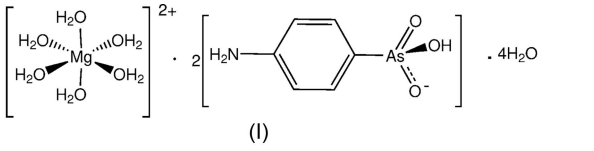
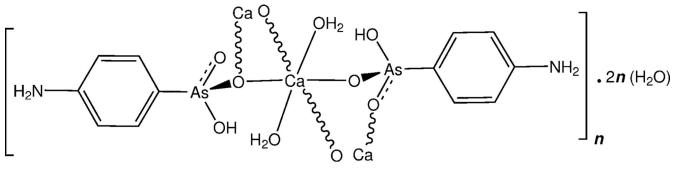


Figure 1
The molecular configuration and atom-naming scheme for the hexaaquamagnesium cation, the *p*-arsanilate anion and the two water molecules of solvation in the the asymmetric unit of (I), with displacement ellipsoids drawn at the 40% probability level. Inter-species hydrogen bonds are shown as dashed lines. [Inversion-related atoms are indicated by the symmetry code (i) $-x+1, -y+1, -z+1$.]

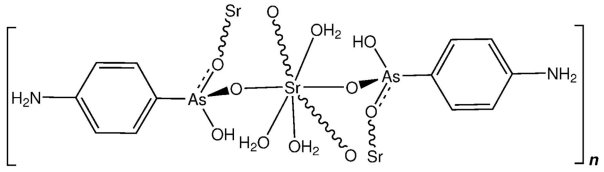
together with that of the monoammonium salt (Smith & Wermuth, 2014) but the number of structures of monometal (as distinct from mixed metal) complexes with *p*-arsanilic acid are few in the crystallographic literature, examples being with silver(I), zinc, cadmium and lead (Lesikar-Parrish *et al.*, 2013) and uranium (as UO_2) (Adelani *et al.*, 2012). An example of a mixed-metal compound is the sodium salt of a hybrid organic–inorganic polyoxovanadate cluster formed with *p*-arsanilate anions (Breen & Schmitt, 2008).



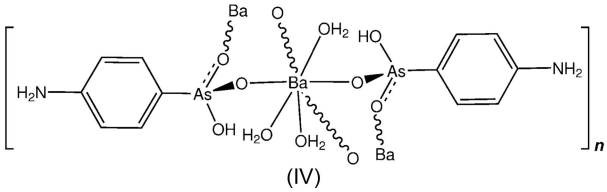
(I)



(II)



(III)



(IV)

Our 2:1 stoichiometric reactions of *p*-arsanilic acid with magnesium, calcium, strontium and barium carbonates in aqueous ethanol gave the title compounds $[Mg(H_2O)_6](C_6H_7AsNO_3)_2.4H_2O$, (I), $\{[Ca(C_6H_7AsNO_3)_2(H_2O)_2]\cdot 2H_2O\}_n$, (II), $[Sr(C_6H_7AsNO_3)_2(H_2O)_3]_n$, (III), and $[Ba(C_6H_7AsNO_3)_2(H_2O)_3]_n$, (IV), respectively. The structures and hydrogen-bonded packing modes for (I)–(IV) are reported herein.

2. Experimental

2.1. Synthesis and crystallization

The title compounds (I)–(IV) were synthesized by heating together under reflux for 5 min, 1.0 mmol (216 mg) of (4-aminophenyl)arsonic acid and 0.5 mmol of the appropriate carbonate, *i.e.* 42 mg of $MgCO_3$ for (I), 50 mg of $CaCO_3$ for (II), 74 mg of $SrCO_3$ for (III) or 98 mg of $BaCO_3$ for (IV), in 20 ml of 50% (*v*/*v*) ethanol/water. Partial room-temperature evaporation of the solutions gave colourless needles of all compounds from which specimens suitable for the X-ray analyses were cleaved. Crystals were mounted on conventional glass fibres using epoxy resin.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms involved in hydrogen bonds were located by difference Fourier methods and their isotropic displacement parameters were allowed to ride. N—H bond lengths were restrained to 0.88 (2) Å for (I) and (II), and to 0.90 (2) Å for (III) and (IV). O—H bond lengths were restrained to 0.90 (2) Å in all cases. H atoms bonded to C atoms were included in the refinement in calculated positions (C—H = 0.95 Å) and treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

3. Results and discussion

In the structure of magnesium compound (I), the cations exist as the commonly found centrosymmetric octahedral $[Mg(H_2O)_6]^{2+}$ species with the hydrogen *p*-arsanilate acting as counter-anions. The coordinated aqua ligands are related in pairs by $(-x+1, -y+1, -z+1)$, denoted symmetry code (i) (Fig. 1). The Mg—O bond lengths [2.066 (2)–2.0734 (18) Å]

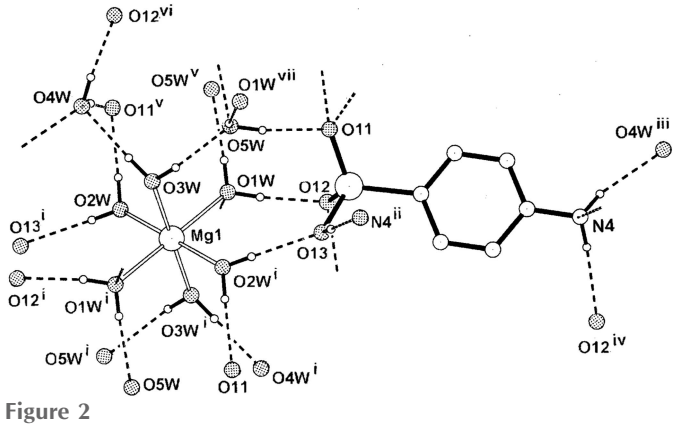


Figure 2
The hydrogen-bonding extension in the structure of (I), showing the symmetry operations (see Table 2). H atoms not involved in hydrogen bonding have been omitted.

Table 1
Experimental details.

	(I)	(II)	(III)	(IV)
Crystal data				
Chemical formula	$[Mg(H_2O)_6](C_6H_7AsNO_3)\cdot 4H_2O$	$[Ca(C_6H_7AsNO_3)_2(H_2O)_2]\cdot 2H_2O$	$[Sr(C_6H_7AsNO_3)_2(H_2O)_3]$	$[Ba(C_6H_7AsNO_3)_2(H_2O)_3]$
M_r	636.56	544.24	573.76	623.92
Crystal system, space group	Monoclinic, $P2_1/c$	Triclinic, $P\overline{1}$	Monoclinic, $I2/a$	Monoclinic, $I2/a$
Temperature (K)	200	200	200	200
a, b, c (Å)	15.1693 (6), 6.7367 (2), 12.9532 (4)	9.001 (5), 9.672 (5), 11.756 (5)	9.8935 (3), 7.5844 (3), 23.6669 (9)	9.9997 (8), 7.7305 (6), 23.979 (2)
α, β, γ (°)	90, 108.033 (4), 90	77.096 (5), 74.096 (5), 82.236 (5)	90, 97.866 (3), 90	90, 98.214 (7), 90
V (Å^3)	1258.68 (7)	956.4 (8)	1759.17 (11)	1834.7 (3)
Z	2	2	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.75	3.82	6.85	5.79
Crystal size (mm)	0.35 × 0.18 × 0.10	0.35 × 0.11 × 0.10	0.25 × 0.12 × 0.12	0.36 × 0.22 × 0.16
Data collection				
Diffractometer	Oxford Diffraction Gemini-S CCD detector	Oxford Diffraction Gemini-S CCD detector	Oxford Diffraction Gemini-S CCD detector	Oxford Diffraction Gemini-S CCD detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.700, 0.980	0.931, 0.980	0.791, 0.980	0.470, 0.970
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8901, 2466, 2149	6921, 3744, 3276	3326, 1737, 1606	3485, 1801, 1696
R_{int}	0.032	0.032	0.020	0.029
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.617	0.617	0.617	0.617
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.028, 0.072, 1.12	0.031, 0.068, 1.06	0.022, 0.051, 1.09	0.023, 0.055, 1.10
No. of reflections	2466	3744	1737	1801
No. of parameters	190	286	137	137
No. of restraints	13	14	7	7
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.40, −0.64	0.55, −0.47	0.47, −0.54	0.85, −0.95

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SIR92* (Altomare *et al.*, 1993), *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012), and *PLATON* (Spek, 2009).

are typical of those found in $[Mg(H_2O)_6]^{2+}$ complex cations with analogous phosphonate anions, *e.g.* with hydrogen diphenylmethylphosphonate (Lee *et al.*, 1988), hydrogen (2-aminoethyl)phosphonate (Schier *et al.*, 1990) and the carboxylate anion 4-nitrobenzoate (Arlin *et al.*, 2011).

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

D—H···A	D—H	H···A	D···A	D—H···A
O13—H13···N4^{ii}	0.85 (2)	1.89 (2)	2.746 (3)	177 (3)
N4—H41···O4W^{iii}	0.86 (3)	2.07 (3)	2.903 (3)	164 (3)
N4—H42···O12^{iv}	0.86 (2)	2.12 (2)	2.980 (3)	172 (2)
O1W—H11W···O12	0.86 (2)	1.78 (2)	2.636 (3)	175 (2)
O1W—H12W···O5W^{v}	0.84 (2)	1.91 (2)	2.745 (2)	172 (3)
O2W—H21W···O11^{v}	0.87 (2)	1.84 (2)	2.707 (2)	174 (2)
O2W—H22W···O13^{i}	0.84 (3)	2.00 (3)	2.832 (3)	177 (3)
O3W—H31W···O4W	0.89 (2)	1.90 (2)	2.781 (3)	175 (3)
O3W—H32W···O5W	0.87 (2)	1.94 (3)	2.796 (3)	165 (2)
O4W—H41W···O11^{v}	0.85 (2)	1.87 (2)	2.717 (2)	171 (3)
O4W—H42W···O12^{vi}	0.87 (2)	1.86 (2)	2.720 (2)	174 (3)
O5W—H51W···O11	0.87 (2)	1.89 (2)	2.754 (3)	170 (2)
O5W—H52W···O1W^{vii}	0.83 (2)	2.14 (2)	2.958 (3)	169 (3)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+2, -z+1$; (iii) $x-1, y, z$; (iv) $-x, -y+1, -z+1$; (v) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (vi) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (vii) $x, y+1, z$.

In the crystal of (I), extensive hydrogen-bonding involving all H-atom donors of the anions and those of both the coordinated and solvent water molecules (O4W and O5W) with arsanilate O- and N-atom acceptors and water O-atom acceptors (Table 2) generate a three-dimensional structure (Figs. 2 and 3). The coordinated water molecules interact with both arsanilate O11^{v}, O12 and O13^{i}, and water O4W, O5W and O5W^{v} atoms. These interactions include an $R_3^3(10)$ cyclic motif involving both the Mg and As atoms. The solvent water molecules involve O11, O11^{v}, O12^{vi} and O1W^{vii} acceptors. The arsanilate amine group acts as both a double donor to O12^{iv} and O4W^{iii} atoms and as an acceptor to the O13 acid H-atom donor of an inversion-related anion, linking these along [010]. Weak π–π associations are also present between inversion-related anions [ring centroid separation $Cg\cdots Cg^{ii}$ = 3.7187 (14) Å] [symmetry codes: (ii) $-x, -y+2, -z+1$; (iii) $x-1, y, z$; (iv) $-x, -y+1, -z+1$; (v) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (vi) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (vii) $x, y+1, z$].

In the structure of the coordination polymeric calcium compound (II), the repeat unit is a distorted octahedral CaO_6 complex comprising four O-atom donors (with two bridging) from the two independent hydrogen *p*-arsanilate anions (*A*

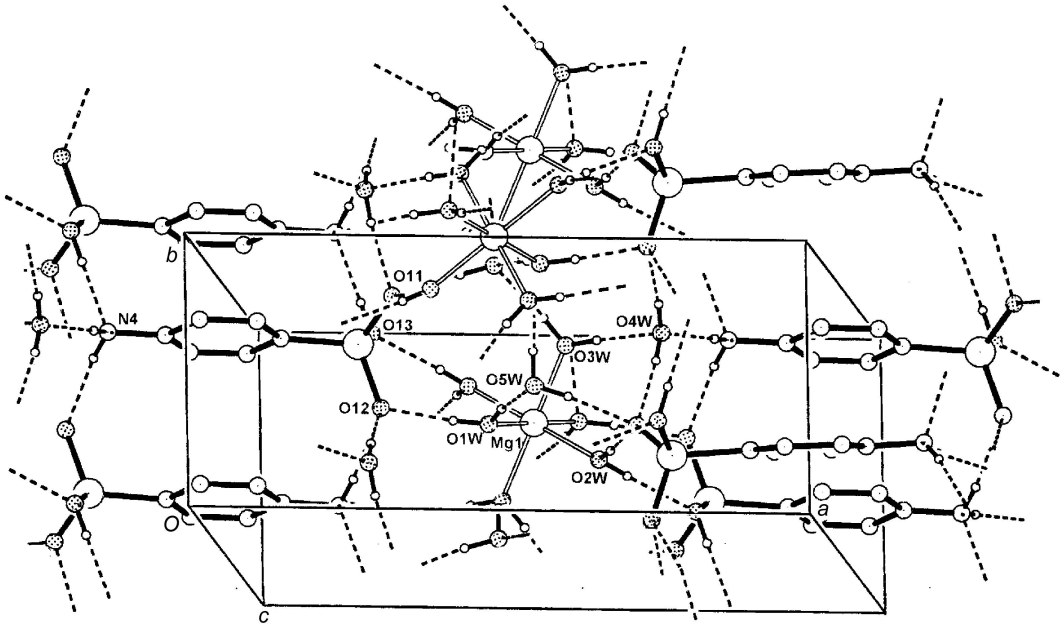


Figure 3
A perspective view of the three-dimensional hydrogen-bonded framework structure of (I) in the unit cell, showing the hydrogen-bonding associations as dashed lines. H atoms not involved in hydrogen bonding have been omitted. For symmetry codes, see Table 2.

Table 3
Selected bond lengths (Å) for (II).

Ca1—O1*W*	2.392 (3)	Ca1—O11*B*	2.361 (2)
Ca1—O2*W*	2.345 (3)	Ca1—O11*B*^{i}	2.420 (2)
Ca1—O11*A*	2.306 (2)	Ca1—O12*A*^{ii}	2.291 (2)

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$.

and *B*), together with two independent monodentate water molecules (O1*W* and O2*W*). Present also in the structure are two water molecules of solvation (O3*W* and O4*W*) (Fig. 4). The Ca—O bond lengths are in the range 2.306 (2)–2.420 (2) Å (Table 3), typical values for Ca—O bond lengths in the six-coordinate diaquacalcium–carboxylate–L-valine complex (Lamberts & Englert, 2015) (2.278–2.372 Å). The bridging carboxylate O atoms in (II) generate a coordination polymer extending along [100] through centrosymmetric cyclic ring systems, *i.e.* four-membered through O11*B*^{i} and eight-membered through O11*A*^{ii} and O12*A*^{ii} [symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$] (Fig. 5). The Ca···Ca^{i} separation in the four-membered ring is 3.801 (2) Å. The crystal structure involves a number of inter-chain N—H···O and O—H···O hydrogen-bonding interactions to arsonate and water O-atom acceptors, as well as to the amine N-atom acceptors (Table 4 and Fig. 5). These generate an

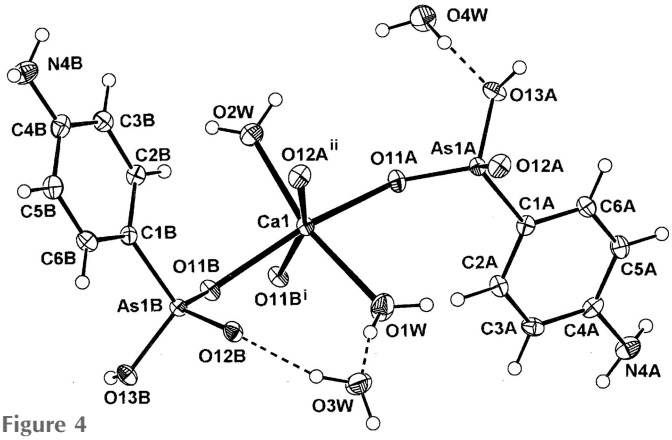


Figure 4
The molecular configuration and atom-naming scheme for the asymmetric unit and the water molecules of solvation (O3*W* and O4*W*) in (II). Displacement ellipsoids are drawn at the 40% probability level. For symmetry codes, see Table 3.

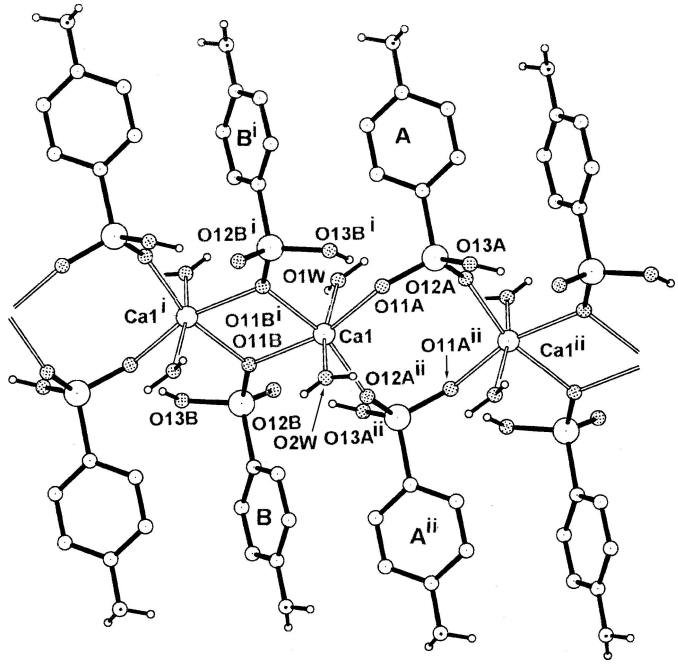


Figure 5
A portion of the one-dimensional coordination polymeric chains in the structure of (II) extending along *a*. For symmetry codes, see Table 3.

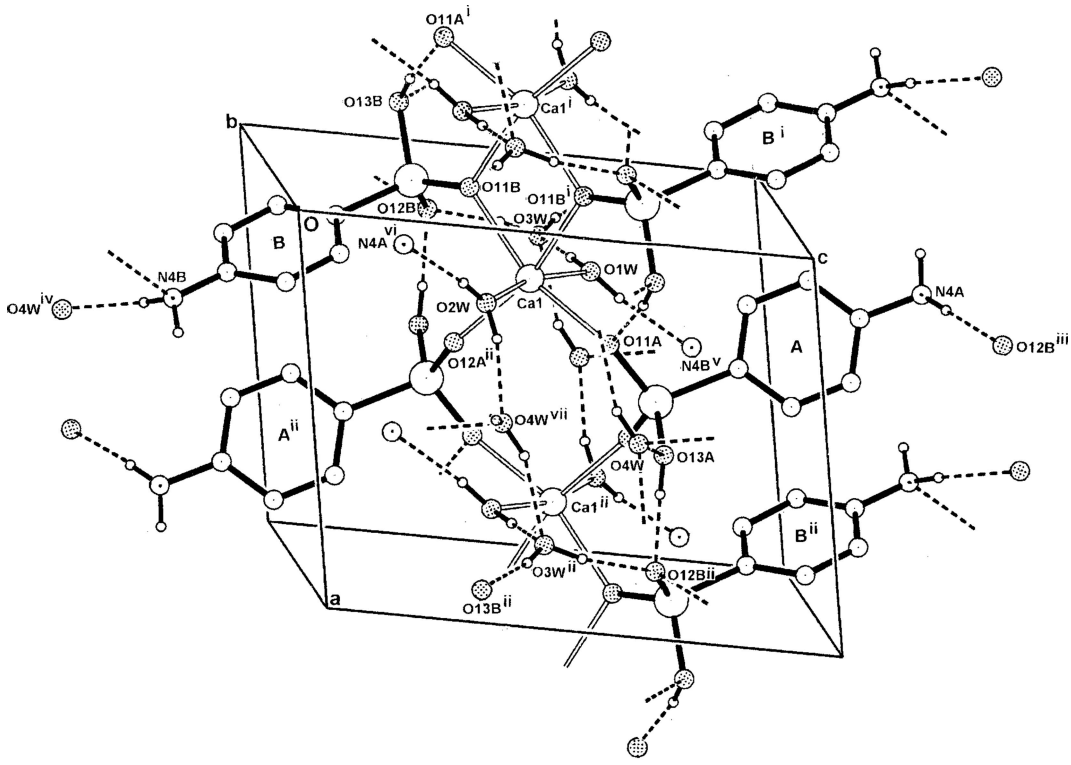


Figure 6
Hydrogen bonding in the three-dimensional structure of (II), viewed down *b*, showing hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted. For symmetry codes, see Table 4.

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13*A*—H13*A*···O12*B*^{ii}	0.88 (2)	1.67 (2)	2.534 (3)	169 (4)
O13*B*—H13*B*···O11*A*^{i}	0.88 (3)	1.70 (3)	2.578 (3)	170 (3)
N4*A*—H41*A*···O12*B*^{iii}	0.88 (2)	2.13 (2)	3.014 (4)	176 (4)
N4*B*—H42*B*···O4*W*^{iv}	0.86 (3)	2.05 (3)	2.901 (5)	172 (4)
O1*W*—H11*W*···N4*B*^{v}	0.90 (4)	2.17 (4)	3.048 (5)	166 (4)
O1*W*—H12*W*···O3*W*	0.89 (2)	1.83 (2)	2.712 (4)	171 (3)
O2*W*—H21*W*···N4*A*^{vi}	0.86 (4)	2.04 (4)	2.893 (4)	173 (4)
O2*W*—H22*W*···O4*W*^{vii}	0.88 (3)	1.92 (3)	2.785 (4)	168 (4)
O3*W*—H31*W*···O13*B*^{viii}	0.88 (4)	1.96 (4)	2.827 (4)	169 (4)
O3*W*—H32*W*···O12*B*	0.89 (3)	1.92 (3)	2.758 (4)	157 (4)
O4*W*—H41*W*···O3*W*^{ix}	0.87 (4)	2.08 (4)	2.915 (4)	160 (3)
O4*W*—H42*W*···O13*A*	0.89 (4)	1.86 (3)	2.748 (4)	176 (2)

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$; (iii) $x, y-1, z+1$; (iv) $x, y+1, z-1$; (v) $x, y, z+1$; (vi) $x, y, z-1$; (vii) $-x+1, -y, -z+1$; (viii) $-x, -y+2, -z+1$; (ix) $x, y-1, z$.

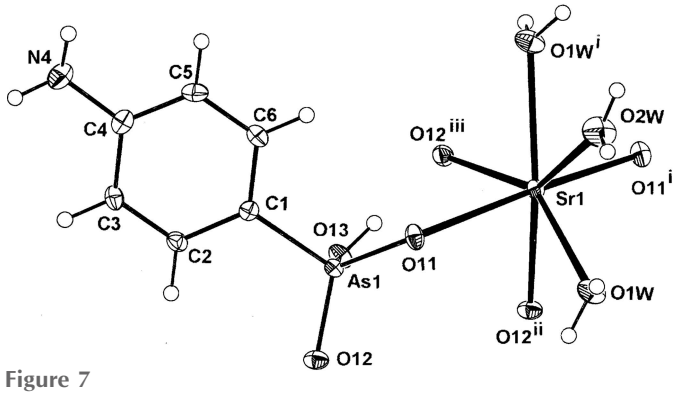


Figure 7
The molecular configuration and atom-naming scheme for the complex unit in (III). Displacement ellipsoids are drawn at the 40% probability level. [Symmetry codes: (i) $-x+\frac{3}{2}, y, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x+\frac{1}{2}, -y, z$.]

Table 5
Selected bond lengths (Å) for (III).

Sr1—O1*W*	2.628 (2)	Sr1—O11^{i}	2.5527 (18)
Sr1—O2*W*	2.628 (3)	Sr1—O12^{ii}	2.549 (2)
Sr1—O11	2.5527 (18)	Sr1—O12^{iii}	2.549 (2)
Sr1—O1*W*^{i}	2.628 (2)		

Symmetry codes: (i) $-x+\frac{3}{2}, y, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x+\frac{1}{2}, -y, z$.

overall three-dimensional framework structure (Fig. 6) in which no π–π ring interactions are present.

The structures of the polymeric Sr and Ba complexes (III) and (IV), respectively, are isotypic and are based on irregular MO_7 coordination polyhedra about the M^{2+} centres, which lie on twofold rotation axes. The basic SrO_7 or BaO_7 complex repeat units (Figs. 7 and 8) comprise three monodentate water molecules, with one (O2*W*) lying on a twofold rotation axis and the others (O1*W* and O1*W*^{i}) related by the rotation axis [symmetry code: (i) $-x+\frac{3}{2}, y, -z+1$]. The coordination sphere is completed by four *O*:*O*′-bridging arsonate O-atom donors, *i.e.* O11, O11^{i}, O12^{ii} and O12^{iii} [symmetry codes: (ii) $-x+1, -y, -z+1$; (iii) $x+\frac{1}{2}, -y, z$]. The *M*—O bond-length ranges are 2.549 (2)–2.628 (2) (for Sr) and 2.694 (2)–2.779 (2) Å (for Ba) (Tables 5 and 6).

Both *O*:*O*′-bridging groups provide eight-membered cyclic extensions of the structure, one of which is centrosymmetric, giving a one-dimensional chain polymer structure extending

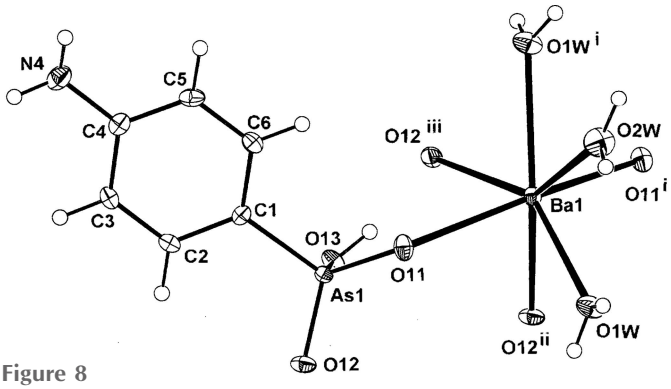


Figure 8
The molecular configuration and atom-naming scheme for the complex unit in (IV). Displacement ellipsoids are drawn at the 40% probability level. [Symmetry codes: (i) $-x+\frac{3}{2}, y, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x+\frac{1}{2}, -y, z$.]

along [100] (Fig. 9). In the crystal, there are inter-polymer N—H···O and O—H···O hydrogen-bonding associations to arsonate and water O-atom acceptors (Tables 7 and 8), generating a three-dimensional framework structure (Fig. 10). Present also in the crystal packing of (III) and (IV) are relatively strong π–π aromatic ring interactions with ring centroid separations ($Cg\cdots Cg^{vii}$) of 3.4666 (17) Å in (III) and 3.4855 (8) Å in (IV) between inversion-related hydrogen arsanilate ligands [symmetry code: (vii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{1}{2}$].

The monoanionic arsonate groups in all four structures are similar in having delocalized As—O11 and As—O12 bonds, which are essentially equal: 1.6617 (15) and 1.6581 (14) Å in (I); 1.673 (2) and 1.645 (2) Å in (II*A*), and 1.667 (2) and 1.668 (2) Å in (II*B*); 1.6650 (16) and 1.6705 (19) Å in (III); 1.667 (2) and 1.668 (2) Å in (IV). These compare with

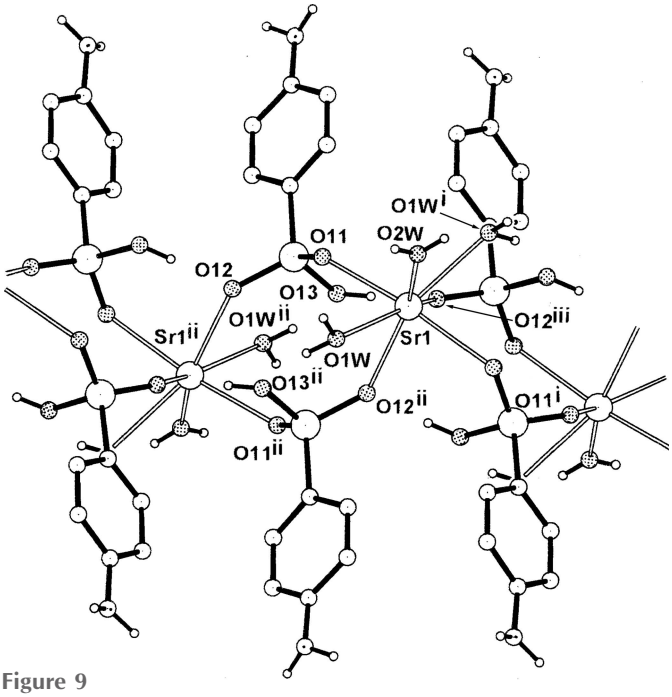


Figure 9
The one-dimensional coordination polymeric chains in the structure of (III), extending along *a*. The isotypic structure of (IV) is similar. For symmetry codes, see Tables 7 and 8.

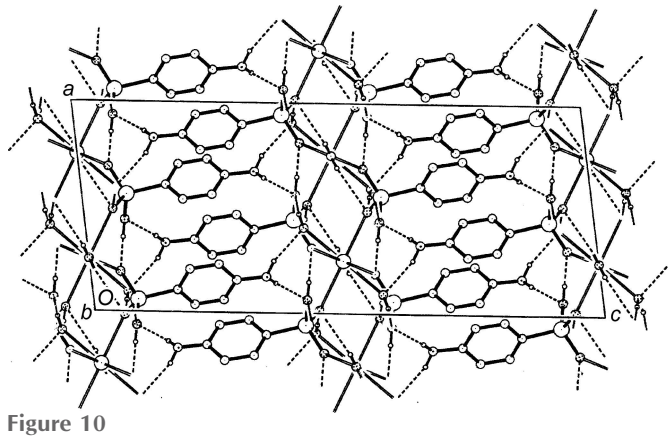


Figure 10
The hydrogen bonding in the three-dimensional complex structures of (III) and (IV). Non-associative H atoms have been omitted.

Table 6
Selected bond lengths (Å) for (IV).

Ba1—O1*W*	2.774 (2)	Ba1—O11^{i}	2.706 (2)
Ba1—O2*W*	2.779 (4)	Ba1—O12^{ii}	2.694 (2)
Ba1—O11	2.706 (2)	Ba1—O12^{iii}	2.694 (2)
Ba1—O1*W*^{i}	2.774 (2)		

Symmetry codes: (i) $-x+\frac{3}{2}, y, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x+\frac{1}{2}, -y, z$.

Table 7
Hydrogen-bond geometry (Å, °) for (III).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11*W*···O11^{iv}	0.88 (2)	1.92 (2)	2.791 (3)	173 (3)
O1*W*—H12*W*···O13^{ii}	0.88 (3)	2.02 (2)	2.845 (3)	156 (3)
O13—H13···O12^{iii}	0.88 (2)	1.73 (2)	2.606 (3)	176 (3)
O2*W*—H21*W*···O11^{iv}	0.83 (3)	2.52 (3)	3.267 (2)	149 (3)
N4—H41···O1*W*^{v}	0.89 (2)	2.24 (3)	3.115 (4)	165 (2)
N4—H42···O13^{vi}	0.89 (3)	2.33 (3)	3.210 (4)	170 (3)

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

Table 8
Hydrogen-bond geometry (Å, °) for (IV).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11*W*···O11^{iv}	0.88 (2)	1.87 (2)	2.738 (3)	168 (4)
O1*W*—H12*W*···O13^{ii}	0.86 (3)	2.13 (3)	2.902 (3)	149 (3)
O13—H13···O12^{iii}	0.87 (2)	1.77 (2)	2.626 (3)	167 (3)
O2*W*—H21*W*···O11^{iv}	0.87 (4)	2.46 (4)	3.229 (2)	149 (3)
N4—H41···O1*W*^{v}	0.87 (3)	2.23 (3)	3.092 (4)	171 (3)
N4—H42···O13^{vi}	0.88 (3)	2.37 (3)	3.241 (4)	170 (3)

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

1.672 (3) and 1.677 (3), and 1.670 (3) and 1.659 (3) Å in the ammonium and sodium salts, respectively (Smith & Wermuth, 2014), and 1.656 (6) and 1.669 (6) Å in the zwitterionic parent acid (Shimada, 1961; Nuttall & Hunter, 1996). The As—O13(H) bonds are 1.7412 (16) Å in (I), 1.721 (2) Å in (II*A*) and 1.727 (2) Å in (II*B*), 1.746 (2) Å in (III) and 1.749 (2) Å in (IV), compared with 1.737 (8) Å in the parent acid.

The work reported here provides a comparison of the coordination chemistry and hydrogen bonding in the three-

dimensional structures of the alkaline earth series of complexes with hydrogen (4-aminophenyl)arsonic acid. With the exception of Mg complex (I), all the compounds form basically similar supramolecular structures in which the primary core comprising the polymeric metal complex and water molecules form layers which are linked through the peripheral arsanilate ring systems by hydrogen bonding involving the *para*-related aniline amino group. This packing feature is also found in the sodium hydrogen *p*-arsanilate structure (Smith & Wermuth, 2014), although the primary layer differs from the alkaline-earth members (II)–(IV) in that it involves an NaO_5 complex core with one of the three coordinated water molecules bridging.

Acknowledgements

The authors acknowledge support from the Science and Engineering Faculty, Queensland University of Technology.

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supporting information

Acta Cryst. (2017). C**73**, 61-67 [https://doi.org/10.1107/S2053229616019434]

The coordination complex structures and hydrogen bonding in the three-dimensional alkaline earth metal salts (Mg, Ca, Sr and Ba) of (4-aminophenyl)-arsonic acid

Graham Smith and Urs D. Wermuth

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015 for (I); *CrysAlis PRO* (Rigaku OD, 2015) for (II), (III), (IV). For all compounds, cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 2012); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON* (Spek, 2009).

(I) Hexaaquamagnesium bis[hydrogen (4-aminophenyl)arsonate] tetrahydrate

Crystal data

$[Mg(H_2O)_6](C_6H_7AsNO_3)\cdot 4H_2O$
$M_r = 636.56$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.1693$ (6) Å
$b = 6.7367$ (2) Å
$c = 12.9532$ (4) Å
$\beta = 108.033$ (4)°
$V = 1258.68$ (7) Å^3
$Z = 2$
$F(000) = 652$
$D_x = 1.680$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3242 reflections
$\theta = 3.6$–$29.1°$
$\mu = 2.75$ mm^{-1}
$T = 200$ K
Prism, colourless
0.35 × 0.18 × 0.10 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
$T_{min} = 0.700$, $T_{max} = 0.980$
8901 measured reflections
2466 independent reflections
2149 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.3°$
$h = -18\rightarrow18$
$k = -8\rightarrow8$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.072$
$S = 1.12$
2466 reflections
190 parameters
13 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2 + 0.0883P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 0.40$ e Å^{-3}
	$\Delta\rho_{min} = -0.64$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
As1	0.23165 (2)	0.74327 (3)	0.60063 (2)	0.0166 (1)
O11	0.29867 (12)	0.8873 (2)	0.69756 (12)	0.0246 (5)
O12	0.26694 (12)	0.5094 (2)	0.60663 (12)	0.0236 (5)
O13	0.23879 (12)	0.8264 (2)	0.47619 (12)	0.0244 (5)
N4	−0.17769 (16)	0.7891 (3)	0.56343 (19)	0.0242 (7)
C1	0.10489 (18)	0.7641 (3)	0.59183 (19)	0.0186 (7)
C2	0.07751 (19)	0.7930 (4)	0.6839 (2)	0.0258 (8)
C3	−0.0154 (2)	0.7997 (4)	0.6747 (2)	0.0278 (8)
C4	−0.08290 (19)	0.7765 (3)	0.5742 (2)	0.0206 (7)
C5	−0.0548 (2)	0.7492 (3)	0.4821 (2)	0.0230 (8)
C6	0.0379 (2)	0.7422 (3)	0.4915 (2)	0.0218 (8)
Mg1	0.50000	0.50000	0.50000	0.0192 (3)
O1W	0.44254 (13)	0.4488 (3)	0.62346 (13)	0.0264 (5)
O2W	0.61417 (14)	0.3374 (3)	0.58760 (13)	0.0305 (6)
O3W	0.56172 (16)	0.7579 (2)	0.57476 (16)	0.0308 (7)
O4W	0.73459 (16)	0.7514 (2)	0.73245 (16)	0.0294 (7)
O5W	0.45949 (14)	1.0160 (3)	0.66270 (14)	0.0283 (6)
H2	0.12280	0.80820	0.75290	0.0310*
H3	−0.03370	0.82040	0.73770	0.0330*
H5	−0.10000	0.73540	0.41280	0.0280*
H6	0.05640	0.72220	0.42850	0.0260*
H13	0.2204 (19)	0.945 (3)	0.462 (2)	0.0370*
H41	−0.193 (2)	0.772 (3)	0.6215 (19)	0.0290*
H42	−0.2074 (19)	0.711 (3)	0.5111 (18)	0.0290*
H11W	0.3843 (13)	0.463 (4)	0.615 (2)	0.0400*
H12W	0.4704 (19)	0.481 (4)	0.6882 (15)	0.0400*
H21W	0.641 (2)	0.345 (4)	0.6574 (14)	0.0460*
H22W	0.6573 (19)	0.286 (4)	0.569 (3)	0.0460*
H31W	0.6181 (16)	0.761 (4)	0.623 (2)	0.0460*
H32W	0.5287 (19)	0.852 (3)	0.592 (2)	0.0460*

H41W	0.729 (2)	0.639 (3)	0.760 (2)	0.0440*
H42W	0.738 (2)	0.839 (3)	0.7823 (19)	0.0440*
H51W	0.4052 (14)	0.979 (4)	0.666 (2)	0.0420*
H52W	0.457 (2)	1.135 (3)	0.645 (2)	0.0420*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1	0.0139 (2)	0.0182 (2)	0.0174 (2)	0.0013 (1)	0.0043 (1)	0.0003 (1)
O11	0.0234 (11)	0.0262 (8)	0.0215 (8)	−0.0038 (7)	0.0030 (7)	−0.0036 (7)
O12	0.0209 (10)	0.0188 (8)	0.0305 (9)	0.0025 (7)	0.0069 (7)	0.0011 (7)
O13	0.0278 (11)	0.0250 (8)	0.0232 (9)	0.0068 (8)	0.0122 (8)	0.0044 (7)
N4	0.0171 (13)	0.0292 (10)	0.0275 (12)	−0.0008 (9)	0.0087 (10)	−0.0004 (9)
C1	0.0142 (14)	0.0191 (11)	0.0222 (12)	0.0018 (9)	0.0052 (10)	0.0021 (8)
C2	0.0208 (16)	0.0369 (13)	0.0180 (12)	0.0028 (11)	0.0037 (11)	−0.0006 (10)
C3	0.0264 (17)	0.0387 (13)	0.0203 (12)	0.0041 (12)	0.0103 (12)	0.0017 (11)
C4	0.0192 (15)	0.0168 (10)	0.0261 (13)	0.0017 (10)	0.0074 (11)	0.0024 (9)
C5	0.0189 (16)	0.0275 (13)	0.0204 (12)	0.0003 (10)	0.0031 (11)	−0.0028 (9)
C6	0.0224 (16)	0.0253 (12)	0.0183 (12)	0.0006 (10)	0.0070 (11)	−0.0045 (9)
Mg1	0.0172 (7)	0.0225 (5)	0.0180 (5)	0.0019 (5)	0.0056 (5)	0.0000 (4)
O1W	0.0199 (11)	0.0403 (9)	0.0197 (8)	0.0020 (9)	0.0073 (8)	−0.0018 (8)
O2W	0.0252 (12)	0.0448 (11)	0.0199 (9)	0.0172 (9)	0.0048 (8)	−0.0008 (8)
O3W	0.0246 (13)	0.0304 (10)	0.0352 (11)	0.0010 (8)	0.0060 (9)	−0.0106 (8)
O4W	0.0359 (14)	0.0277 (10)	0.0273 (10)	−0.0049 (8)	0.0136 (10)	−0.0021 (7)
O5W	0.0240 (12)	0.0257 (9)	0.0347 (10)	0.0002 (8)	0.0082 (8)	0.0030 (8)

Geometric parameters (Å, º)

Mg1—O1W	2.0734 (18)	O4W—H41W	0.85 (2)
Mg1—O2W	2.066 (2)	O4W—H42W	0.86 (2)
Mg1—O3W	2.0668 (16)	O5W—H51W	0.87 (2)
Mg1—O1W^{i}	2.0734 (18)	O5W—H52W	0.83 (2)
Mg1—O2W^{i}	2.066 (2)	N4—C4	1.404 (4)
Mg1—O3W^{i}	2.0668 (16)	N4—H41	0.86 (3)
As1—O11	1.6617 (15)	N4—H42	0.87 (2)
As1—O12	1.6581 (14)	C1—C6	1.388 (4)
As1—O13	1.7412 (15)	C1—C2	1.393 (4)
As1—C1	1.896 (3)	C2—C3	1.378 (4)
O13—H13	0.85 (2)	C3—C4	1.394 (4)
O1W—H12W	0.84 (2)	C4—C5	1.398 (4)
O1W—H11W	0.86 (2)	C5—C6	1.374 (4)
O2W—H21W	0.871 (18)	C2—H2	0.9500
O2W—H22W	0.84 (3)	C3—H3	0.9500
O3W—H32W	0.88 (2)	C5—H5	0.9500
O3W—H31W	0.89 (3)	C6—H6	0.9500
O11—As1—O12	113.97 (8)	H21W—O2W—H22W	100 (3)
O11—As1—O13	108.23 (7)	Mg1—O3W—H32W	121.0 (18)

O11—As1—C1	111.97 (9)	H31W—O3W—H32W	108 (2)
O12—As1—O13	103.73 (7)	Mg1—O3W—H31W	123.1 (17)
O12—As1—C1	112.33 (9)	H41W—O4W—H42W	107 (2)
O13—As1—C1	105.85 (9)	H51W—O5W—H52W	109 (3)
O1W^{i}—Mg1—O3W	89.08 (8)	C4—N4—H41	117 (2)
O2W^{i}—Mg1—O3W	89.59 (8)	H41—N4—H42	113 (2)
O3W—Mg1—O3W^{i}	180.00	C4—N4—H42	108 (2)
O1W^{i}—Mg1—O2W^{i}	88.24 (8)	C2—C1—C6	119.4 (3)
O1W^{i}—Mg1—O3W^{i}	90.92 (8)	As1—C1—C6	118.9 (2)
O2W^{i}—Mg1—O3W^{i}	90.41 (8)	As1—C1—C2	121.65 (19)
O1W^{i}—Mg1—O2W	91.76 (8)	C1—C2—C3	119.9 (2)
O1W—Mg1—O2W	88.24 (8)	C2—C3—C4	120.9 (2)
O1W—Mg1—O3W	90.92 (8)	N4—C4—C3	121.3 (2)
O1W—Mg1—O1W^{i}	180.00	C3—C4—C5	118.8 (3)
O1W—Mg1—O2W^{i}	91.76 (8)	N4—C4—C5	119.8 (2)
O1W—Mg1—O3W^{i}	89.08 (8)	C4—C5—C6	120.2 (2)
O2W—Mg1—O3W	90.41 (8)	C1—C6—C5	120.8 (2)
O2W—Mg1—O2W^{i}	180.00	C3—C2—H2	120.00
O2W—Mg1—O3W^{i}	89.59 (8)	C1—C2—H2	120.00
As1—O13—H13	113.2 (18)	C2—C3—H3	120.00
Mg1—O1W—H11W	122.9 (17)	C4—C3—H3	120.00
H11W—O1W—H12W	106 (3)	C6—C5—H5	120.00
Mg1—O1W—H12W	121.6 (19)	C4—C5—H5	120.00
Mg1—O2W—H21W	125.6 (19)	C1—C6—H6	120.00
Mg1—O2W—H22W	131 (2)	C5—C6—H6	120.00

O11—As1—C1—C2	−34.6 (2)	As1—C1—C6—C5	177.68 (16)
O11—As1—C1—C6	147.97 (16)	C2—C1—C6—C5	0.2 (3)
O12—As1—C1—C2	95.2 (2)	C1—C2—C3—C4	0.5 (4)
O12—As1—C1—C6	−82.29 (18)	C2—C3—C4—N4	−178.0 (2)
O13—As1—C1—C2	−152.29 (18)	C2—C3—C4—C5	−1.0 (4)
O13—As1—C1—C6	30.24 (18)	N4—C4—C5—C6	178.20 (19)
As1—C1—C2—C3	−177.5 (2)	C3—C4—C5—C6	1.1 (3)
C6—C1—C2—C3	0.0 (4)	C4—C5—C6—C1	−0.7 (3)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13—H13···N4^{ii}	0.85 (2)	1.90 (2)	2.745 (3)	177 (3)
N4—H41···O4*W*^{iii}	0.86 (3)	2.07 (3)	2.904 (3)	164 (3)
N4—H42···O12^{iv}	0.87 (2)	2.12 (2)	2.980 (3)	172 (2)
O1*W*—H11*W*···O12	0.86 (2)	1.78 (2)	2.636 (3)	175 (2)
O1*W*—H12*W*···O5*W*^{v}	0.84 (2)	1.91 (2)	2.745 (2)	172 (3)
O2*W*—H21*W*···O11^{v}	0.87 (2)	1.84 (2)	2.707 (2)	174 (2)
O2*W*—H22*W*···O13^{i}	0.84 (3)	1.99 (3)	2.831 (3)	178 (3)
O3*W*—H31*W*···O4*W*	0.89 (3)	1.89 (3)	2.781 (3)	175 (3)

O3*W*—H32*W*···O5*W*	0.88 (2)	1.94 (3)	2.796 (3)	165 (2)
O4*W*—H41*W*···O11^{v}	0.85 (2)	1.87 (2)	2.716 (2)	172 (3)
O4*W*—H42*W*···O12^{vi}	0.86 (2)	1.86 (2)	2.720 (2)	173 (3)
O5*W*—H51*W*···O11	0.87 (2)	1.89 (2)	2.755 (3)	170 (2)
O5*W*—H52*W*···O1*W*^{vii}	0.83 (2)	2.14 (2)	2.957 (3)	170 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*, −*y*+2, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) −*x*, −*y*+1, −*z*+1; (v) −*x*+1, *y*−1/2, −*z*+3/2; (vi) −*x*+1, *y*+1/2, −*z*+3/2; (vii) *x*, *y*+1, *z*.

(II) *catena*-Poly[[[diaquacalcium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O']-[diaquacalcium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O]] dihydrate]

Crystal data

$[Ca(C_6H_7AsNO_3)_2(H_2O)_2]\cdot 2H_2O$
M_r = 544.24
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 9.001 (5) Å
b = 9.672 (5) Å
c = 11.756 (5) Å
α = 77.096 (5)°
β = 74.096 (5)°
γ = 82.236 (5)°
V = 956.4 (8) Å^3
Z = 2
F(000) = 548
D_x = 1.890 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 2319 reflections
θ = 3.7–29.1°
μ = 3.82 mm^{-1}
T = 200 K
Needle, colourless
0.35 × 0.11 × 0.10 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.931, T_{max} = 0.980
6921 measured reflections
3744 independent reflections
3276 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 26.0°, θ_{min} = 3.3°
h = −11→11
k = −11→11
l = −11→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.068
S = 1.06
3744 reflections
286 parameters
14 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0206P)^2 + 0.3783P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
As1A	0.46094 (3)	0.29369 (3)	0.70142 (3)	0.0151 (1)
As1B	0.05599 (3)	0.77612 (3)	0.30427 (3)	0.0150 (1)
Ca1	0.21559 (7)	0.47820 (7)	0.49139 (5)	0.0163 (2)
O1W	0.2142 (3)	0.6294 (3)	0.6273 (3)	0.0385 (10)
O2W	0.2504 (3)	0.2937 (3)	0.3844 (2)	0.0337 (9)
O11A	0.3299 (2)	0.3106 (2)	0.62105 (19)	0.0212 (7)
O11B	0.0283 (2)	0.6219 (2)	0.40158 (19)	0.0188 (6)
O12A	0.5836 (2)	0.4184 (2)	0.65510 (19)	0.0237 (7)
O12B	0.1482 (2)	0.8895 (2)	0.3433 (2)	0.0215 (7)
O13A	0.5597 (2)	0.1296 (2)	0.6925 (2)	0.0245 (7)
O13B	−0.1207 (2)	0.8695 (2)	0.3001 (2)	0.0210 (7)
N4A	0.1055 (3)	0.1983 (3)	1.2273 (3)	0.0280 (10)
N4B	0.3873 (3)	0.6567 (4)	−0.1903 (3)	0.0316 (11)
C1A	0.3520 (3)	0.2727 (3)	0.8657 (3)	0.0174 (9)
C1B	0.1555 (3)	0.7413 (3)	0.1487 (3)	0.0175 (9)
C2A	0.2011 (3)	0.3329 (4)	0.8981 (3)	0.0214 (10)
C2B	0.2238 (3)	0.6061 (3)	0.1366 (3)	0.0193 (10)
C3A	0.1191 (3)	0.3081 (4)	1.0179 (3)	0.0219 (10)
C3B	0.3031 (3)	0.5791 (4)	0.0248 (3)	0.0224 (10)
C4A	0.1858 (4)	0.2232 (4)	1.1056 (3)	0.0211 (10)
C4B	0.3143 (4)	0.6866 (4)	−0.0779 (3)	0.0234 (10)
C5A	0.3378 (4)	0.1650 (4)	1.0731 (3)	0.0237 (10)
C5B	0.2442 (4)	0.8224 (4)	−0.0651 (3)	0.0233 (10)
C6A	0.4189 (4)	0.1893 (4)	0.9545 (3)	0.0219 (10)
C6B	0.1668 (3)	0.8504 (4)	0.0465 (3)	0.0220 (10)
O3W	0.1947 (3)	0.9169 (3)	0.5593 (2)	0.0356 (9)
O4W	0.4898 (3)	−0.1005 (3)	0.6212 (3)	0.0354 (9)
H2A	0.15470	0.39070	0.83840	0.0260*
H2B	0.21590	0.53190	0.20580	0.0230*
H3A	0.01610	0.34950	1.04030	0.0260*
H3B	0.35060	0.48650	0.01740	0.0270*
H5A	0.38500	0.10860	1.13290	0.0280*
H5B	0.25020	0.89610	−0.13440	0.0280*
H6A	0.52240	0.14880	0.93250	0.0260*
H6B	0.12120	0.94340	0.05430	0.0260*
H11W	0.273 (4)	0.622 (5)	0.679 (3)	0.0580*
H12W	0.202 (5)	0.723 (2)	0.600 (4)	0.0580*
H13A	0.660 (2)	0.135 (4)	0.680 (3)	0.0370*
H13B	−0.194 (3)	0.810 (3)	0.319 (3)	0.0310*
H21W	0.200 (4)	0.270 (4)	0.340 (3)	0.0510*

H22W	0.325 (3)	0.227 (3)	0.393 (4)	0.0510*
H41A	0.121 (4)	0.109 (2)	1.263 (3)	0.0340*
H41B	0.459 (3)	0.587 (3)	−0.192 (3)	0.0380*
H42A	0.008 (2)	0.230 (4)	1.234 (3)	0.0340*
H42B	0.421 (4)	0.731 (3)	−0.241 (3)	0.0380*
H31W	0.159 (4)	0.984 (4)	0.602 (3)	0.0530*
H32W	0.172 (4)	0.934 (5)	0.488 (2)	0.0530*
H41W	0.415 (4)	−0.087 (5)	0.585 (3)	0.0530*
H42W	0.511 (5)	−0.023 (3)	0.641 (4)	0.0530*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
As1A	0.0135 (2)	0.0163 (2)	0.0145 (2)	−0.0032 (1)	−0.0032 (1)	−0.0001 (1)
As1B	0.0142 (2)	0.0138 (2)	0.0154 (2)	−0.0020 (1)	−0.0030 (1)	−0.0003 (1)
Ca1	0.0148 (3)	0.0169 (3)	0.0157 (3)	−0.0035 (3)	−0.0033 (3)	0.0008 (3)
O1W	0.0565 (18)	0.0258 (15)	0.0412 (17)	0.0004 (14)	−0.0250 (14)	−0.0096 (14)
O2W	0.0362 (15)	0.0327 (16)	0.0398 (16)	0.0034 (12)	−0.0159 (13)	−0.0184 (13)
O11A	0.0198 (11)	0.0229 (13)	0.0203 (12)	−0.0042 (10)	−0.0078 (9)	0.0016 (10)
O11B	0.0148 (10)	0.0176 (12)	0.0198 (11)	−0.0042 (9)	−0.0027 (9)	0.0043 (10)
O12A	0.0228 (11)	0.0252 (13)	0.0217 (12)	−0.0131 (10)	−0.0002 (10)	−0.0017 (11)
O12B	0.0172 (11)	0.0219 (13)	0.0276 (13)	−0.0032 (9)	−0.0079 (10)	−0.0055 (11)
O13A	0.0179 (11)	0.0204 (13)	0.0345 (14)	0.0014 (10)	−0.0064 (11)	−0.0061 (11)
O13B	0.0162 (11)	0.0176 (12)	0.0281 (13)	−0.0025 (9)	−0.0069 (10)	−0.0001 (10)
N4A	0.0315 (16)	0.0289 (18)	0.0184 (16)	−0.0068 (15)	0.0017 (13)	−0.0011 (14)
N4B	0.0362 (18)	0.032 (2)	0.0212 (17)	−0.0058 (15)	0.0028 (14)	−0.0046 (15)
C1A	0.0182 (15)	0.0179 (17)	0.0147 (16)	−0.0065 (13)	−0.0021 (13)	0.0002 (13)
C1B	0.0159 (15)	0.0203 (17)	0.0160 (16)	−0.0030 (13)	−0.0040 (13)	−0.0018 (14)
C2A	0.0198 (16)	0.0227 (18)	0.0219 (18)	−0.0018 (14)	−0.0082 (14)	−0.0010 (15)
C2B	0.0227 (16)	0.0168 (17)	0.0177 (17)	−0.0054 (14)	−0.0045 (14)	−0.0002 (14)
C3A	0.0128 (15)	0.0235 (19)	0.0276 (19)	−0.0033 (13)	−0.0004 (14)	−0.0061 (16)
C3B	0.0217 (16)	0.0190 (18)	0.0285 (19)	−0.0033 (14)	−0.0084 (15)	−0.0054 (15)
C4A	0.0257 (17)	0.0201 (18)	0.0164 (17)	−0.0094 (14)	0.0005 (14)	−0.0040 (14)
C4B	0.0197 (16)	0.031 (2)	0.0212 (18)	−0.0115 (15)	−0.0040 (14)	−0.0045 (16)
C5A	0.0262 (17)	0.0226 (19)	0.0219 (18)	−0.0019 (15)	−0.0100 (15)	0.0014 (15)
C5B	0.0292 (18)	0.0210 (18)	0.0160 (17)	−0.0047 (15)	−0.0057 (14)	0.0056 (15)
C6A	0.0183 (16)	0.0227 (18)	0.0243 (18)	−0.0020 (14)	−0.0064 (14)	−0.0021 (15)
C6B	0.0226 (16)	0.0167 (17)	0.0250 (18)	−0.0019 (14)	−0.0064 (14)	0.0002 (15)
O3W	0.0452 (16)	0.0310 (16)	0.0298 (16)	0.0053 (13)	−0.0086 (13)	−0.0104 (13)
O4W	0.0402 (15)	0.0256 (15)	0.0433 (17)	−0.0040 (13)	−0.0149 (13)	−0.0067 (13)

Geometric parameters (Å, º)

Ca1—O1W	2.392 (3)	N4B—C4B	1.380 (5)
Ca1—O2W	2.345 (3)	N4A—H42A	0.88 (2)
Ca1—O11A	2.306 (2)	N4A—H41A	0.88 (2)
Ca1—O11B	2.361 (2)	N4B—H41B	0.87 (3)
Ca1—O11B^{i}	2.420 (2)	N4B—H42B	0.86 (3)

Ca1—O12A^{ii}	2.291 (2)	C1A—C6A	1.393 (5)
As1A—O11A	1.673 (2)	C1A—C2A	1.390 (4)
As1A—O12A	1.645 (2)	C1B—C6B	1.401 (5)
As1A—O13A	1.721 (2)	C1B—C2B	1.388 (4)
As1A—C1A	1.890 (3)	C2A—C3A	1.383 (5)
As1B—O11B	1.667 (2)	C2B—C3B	1.376 (5)
As1B—O12B	1.668 (2)	C3A—C4A	1.388 (5)
As1B—O13B	1.727 (2)	C3B—C4B	1.396 (5)
As1B—C1B	1.886 (3)	C4A—C5A	1.392 (5)
O1W—H12W	0.89 (2)	C4B—C5B	1.399 (6)
O1W—H11W	0.90 (4)	C5A—C6A	1.369 (5)
O2W—H22W	0.88 (3)	C5B—C6B	1.374 (5)
O2W—H21W	0.86 (4)	C2A—H2A	0.9500
O13A—H13A	0.88 (2)	C2B—H2B	0.9500
O13B—H13B	0.88 (3)	C3A—H3A	0.9500
O3W—H31W	0.88 (4)	C3B—H3B	0.9500
O3W—H32W	0.89 (3)	C5A—H5A	0.9500
O4W—H42W	0.89 (4)	C5B—H5B	0.9500
O4W—H41W	0.87 (4)	C6A—H6A	0.9500
N4A—C4A	1.398 (5)	C6B—H6B	0.9500

O11A—As1A—O12A	114.52 (10)	C4A—N4A—H41A	112 (2)
O11A—As1A—O13A	106.16 (10)	H41A—N4A—H42A	115 (3)
O11A—As1A—C1A	107.43 (11)	C4A—N4A—H42A	109 (2)
O12A—As1A—O13A	109.97 (10)	C4B—N4B—H42B	112 (2)
O12A—As1A—C1A	114.58 (12)	H41B—N4B—H42B	111 (3)
O13A—As1A—C1A	103.28 (12)	C4B—N4B—H41B	116 (2)
O11B—As1B—O12B	114.80 (10)	C2A—C1A—C6A	119.5 (3)
O11B—As1B—O13B	109.59 (10)	As1A—C1A—C2A	120.2 (2)
O11B—As1B—C1B	109.58 (12)	As1A—C1A—C6A	120.2 (2)
O12B—As1B—O13B	102.37 (10)	As1B—C1B—C6B	121.2 (2)
O12B—As1B—C1B	111.56 (12)	C2B—C1B—C6B	119.8 (3)
O13B—As1B—C1B	108.58 (12)	As1B—C1B—C2B	119.0 (2)
O1W—Ca1—O2W	166.97 (10)	C1A—C2A—C3A	119.5 (3)
O1W—Ca1—O11A	83.52 (9)	C1B—C2B—C3B	120.3 (3)
O1W—Ca1—O11B	95.61 (9)	C2A—C3A—C4A	120.6 (3)
O1W—Ca1—O11B^{i}	97.05 (9)	C2B—C3B—C4B	120.6 (3)
O1W—Ca1—O12A^{ii}	93.82 (9)	N4A—C4A—C3A	121.5 (3)
O2W—Ca1—O11A	83.57 (8)	C3A—C4A—C5A	119.7 (3)
O2W—Ca1—O11B	97.20 (8)	N4A—C4A—C5A	118.8 (3)
O2W—Ca1—O11B^{i}	84.13 (8)	N4B—C4B—C3B	120.1 (4)
O2W—Ca1—O12A^{ii}	87.99 (8)	C3B—C4B—C5B	118.9 (3)
O11A—Ca1—O11B	162.13 (8)	N4B—C4B—C5B	121.0 (3)
O11A—Ca1—O11B^{i}	87.66 (7)	C4A—C5A—C6A	119.7 (3)
O11A—Ca1—O12A^{ii}	105.49 (7)	C4B—C5B—C6B	120.8 (3)
O11B—Ca1—O11B^{i}	74.70 (7)	C1A—C6A—C5A	121.0 (3)
O11B—Ca1—O12A^{ii}	92.38 (7)	C1B—C6B—C5B	119.7 (3)
O11B^{i}—Ca1—O12A^{ii}	163.83 (8)	C1A—C2A—H2A	120.00

As1A—O11A—Ca1	141.02 (11)	C3A—C2A—H2A	120.00
As1B—O11B—Ca1	126.08 (10)	C1B—C2B—H2B	120.00
As1B—O11B—Ca1^{i}	127.68 (10)	C3B—C2B—H2B	120.00
Ca1—O11B—Ca1^{i}	105.30 (8)	C4A—C3A—H3A	120.00
As1A—O12A—Ca1^{ii}	148.32 (12)	C2A—C3A—H3A	120.00
H11W—O1W—H12W	104 (4)	C2B—C3B—H3B	120.00
Ca1—O1W—H11W	130 (3)	C4B—C3B—H3B	120.00
Ca1—O1W—H12W	117 (3)	C4A—C5A—H5A	120.00
Ca1—O2W—H21W	134 (3)	C6A—C5A—H5A	120.00
Ca1—O2W—H22W	118 (2)	C6B—C5B—H5B	120.00
H21W—O2W—H22W	108 (3)	C4B—C5B—H5B	120.00
As1A—O13A—H13A	111 (3)	C1A—C6A—H6A	120.00
As1B—O13B—H13B	110 (2)	C5A—C6A—H6A	119.00
H31W—O3W—H32W	115 (4)	C5B—C6B—H6B	120.00
H41W—O4W—H42W	115 (4)	C1B—C6B—H6B	120.00

O12A—As1A—O11A—Ca1	−20.6 (2)	O12A^{ii}—Ca1—O11B—As1B	−20.34 (14)
O13A—As1A—O11A—Ca1	−142.16 (17)	O12A^{ii}—Ca1—O11B—Ca1^{i}	170.13 (8)
C1A—As1A—O11A—Ca1	107.86 (19)	O1W—Ca1—O11B^{i}—As1B^{i}	−96.79 (15)
O11A—As1A—O12A—Ca1^{ii}	−67.3 (2)	O1W—Ca1—O11B^{i}—Ca1^{i}	93.92 (10)
O13A—As1A—O12A—Ca1^{ii}	52.2 (2)	O2W—Ca1—O11B^{i}—As1B^{i}	70.16 (14)
C1A—As1A—O12A—Ca1^{ii}	167.9 (2)	O2W—Ca1—O11B^{i}—Ca1^{i}	−99.14 (9)
O11A—As1A—C1A—C2A	−28.5 (3)	O11A—Ca1—O11B^{i}—As1B^{i}	−13.62 (14)
O11A—As1A—C1A—C6A	147.5 (3)	O11A—Ca1—O11B^{i}—Ca1^{i}	177.09 (9)
O12A—As1A—C1A—C2A	100.0 (3)	O11B—Ca1—O11B^{i}—As1B^{i}	169.30 (15)
O12A—As1A—C1A—C6A	−84.1 (3)	O11B—Ca1—O11B^{i}—Ca1^{i}	0.00 (8)
O13A—As1A—C1A—C2A	−140.4 (3)	O1W—Ca1—O12A^{ii}—As1A^{ii}	−4.1 (2)
O13A—As1A—C1A—C6A	35.5 (3)	O2W—Ca1—O12A^{ii}—As1A^{ii}	−171.2 (2)
O12B—As1B—O11B—Ca1	−46.35 (16)	O11A—Ca1—O12A^{ii}—As1A^{ii}	−88.4 (2)
O12B—As1B—O11B—Ca1^{i}	120.84 (13)	O11B—Ca1—O12A^{ii}—As1A^{ii}	91.7 (2)
O13B—As1B—O11B—Ca1	−160.87 (12)	As1A—C1A—C2A—C3A	175.4 (3)
O13B—As1B—O11B—Ca1^{i}	6.33 (16)	C6A—C1A—C2A—C3A	−0.6 (5)
C1B—As1B—O11B—Ca1	80.09 (15)	As1A—C1A—C6A—C5A	−175.5 (3)
C1B—As1B—O11B—Ca1^{i}	−112.72 (15)	C2A—C1A—C6A—C5A	0.4 (5)
O11B—As1B—C1B—C2B	−14.6 (3)	As1B—C1B—C2B—C3B	−177.1 (2)
O11B—As1B—C1B—C6B	168.0 (2)	C6B—C1B—C2B—C3B	0.3 (4)
O12B—As1B—C1B—C2B	113.7 (2)	As1B—C1B—C6B—C5B	178.0 (3)
O12B—As1B—C1B—C6B	−63.7 (3)	C2B—C1B—C6B—C5B	0.6 (5)
O13B—As1B—C1B—C2B	−134.2 (2)	C1A—C2A—C3A—C4A	−0.4 (5)
O13B—As1B—C1B—C6B	48.3 (3)	C1B—C2B—C3B—C4B	−0.8 (5)
O1W—Ca1—O11A—As1A	−40.53 (19)	C2A—C3A—C4A—N4A	179.2 (3)
O2W—Ca1—O11A—As1A	137.75 (19)	C2A—C3A—C4A—C5A	1.4 (6)
O11B^{i}—Ca1—O11A—As1A	−137.90 (18)	C2B—C3B—C4B—N4B	−176.7 (3)
O12A^{ii}—Ca1—O11A—As1A	51.65 (19)	C2B—C3B—C4B—C5B	0.3 (5)
O1W—Ca1—O11B—As1B	73.73 (15)	N4A—C4A—C5A—C6A	−179.4 (3)
O1W—Ca1—O11B—Ca1^{i}	−95.79 (10)	C3A—C4A—C5A—C6A	−1.5 (6)
O2W—Ca1—O11B—As1B	−108.61 (14)	N4B—C4B—C5B—C6B	177.6 (3)
O2W—Ca1—O11B—Ca1^{i}	81.87 (9)	C3B—C4B—C5B—C6B	0.6 (5)

O11B^{i}—Ca1—O11B—As1B	169.52 (15)	C4A—C5A—C6A—C1A	0.6 (6)
O11B^{i}—Ca1—O11B—Ca1^{i}	0.00 (7)	C4B—C5B—C6B—C1B	−1.1 (5)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O13*A*—H13*A*···O12*B*^{ii}	0.88 (2)	1.67 (2)	2.534 (3)	169 (4)
O13*B*—H13*B*···O11*A*^{i}	0.88 (3)	1.70 (3)	2.578 (3)	170 (3)
N4*A*—H41*A*···O12*B*^{iii}	0.88 (2)	2.13 (2)	3.014 (4)	176 (4)
N4*B*—H42*B*···O4*W*^{iv}	0.86 (3)	2.05 (3)	2.901 (5)	172 (4)
O1*W*—H11*W*···N4*B*^{v}	0.90 (4)	2.17 (4)	3.048 (5)	166 (4)
O1*W*—H12*W*···O3*W*	0.89 (2)	1.83 (2)	2.712 (4)	171 (3)
O2*W*—H21*W*···N4*A*^{vi}	0.86 (4)	2.04 (4)	2.893 (4)	173 (4)
O2*W*—H22*W*···O4*W*^{vii}	0.88 (3)	1.92 (3)	2.785 (4)	168 (4)
O3*W*—H31*W*···O13*B*^{viii}	0.88 (4)	1.96 (4)	2.827 (4)	169 (4)
O3*W*—H32*W*···O12*B*	0.89 (3)	1.92 (3)	2.758 (4)	157 (4)
O4*W*—H41*W*···O3*W*^{ix}	0.87 (4)	2.08 (4)	2.915 (4)	160 (3)
O4*W*—H42*W*···O13*A*	0.89 (4)	1.86 (3)	2.748 (4)	176 (2)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*, *y*−1, *z*+1; (iv) *x*, *y*+1, *z*−1; (v) *x*, *y*, *z*+1; (vi) *x*, *y*, *z*−1; (vii) −*x*+1, −*y*, −*z*+1; (viii) −*x*, −*y*+2, −*z*+1; (ix) *x*, *y*−1, *z*.

(III) *catena*-Poly[[triaquastrontium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O']]

Crystal data

$[Sr(C_6H_7AsNO_3)_2(H_2O)_3]$
M_r = 573.76
Monoclinic, *I*2/*a*
Hall symbol: -I 2ya
a = 9.8935 (3) Å
b = 7.5844 (3) Å
c = 23.6669 (9) Å
β = 97.866 (3)°
V = 1759.17 (11) Å^3
Z = 4
F(000) = 1128
D_x = 2.166 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 2201 reflections
θ = 3.6–29.0°
μ = 6.85 mm^{-1}
T = 200 K
Block, colourless
0.25 × 0.12 × 0.12 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.791, T_{max} = 0.980
3326 measured reflections
1737 independent reflections
1606 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 26.0°, θ_{min} = 3.4°
h = −12→12
k = −9→8
l = −29→26

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.051$
$S = 1.09$
1737 reflections
137 parameters
7 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0188P)^2 + 2.3681P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
Sr1	0.75000	0.28095 (5)	0.50000	0.0144 (1)
As1	0.44233 (2)	0.06077 (3)	0.41189 (1)	0.0131 (1)
O1W	0.5937 (2)	0.4181 (3)	0.56983 (10)	0.0265 (7)
O2W	0.75000	0.6274 (4)	0.50000	0.0384 (11)
O11	0.51439 (18)	0.2419 (2)	0.44227 (8)	0.0197 (6)
O12	0.31029 (18)	−0.0183 (3)	0.44109 (8)	0.0198 (6)
O13	0.56339 (18)	−0.1079 (3)	0.42091 (8)	0.0194 (6)
N4	0.3286 (3)	0.0837 (4)	0.15430 (11)	0.0284 (9)
C1	0.3951 (2)	0.0844 (4)	0.33210 (11)	0.0149 (7)
C2	0.2969 (3)	−0.0261 (4)	0.30314 (12)	0.0178 (8)
C3	0.2717 (3)	−0.0238 (4)	0.24435 (12)	0.0198 (8)
C4	0.3452 (3)	0.0894 (4)	0.21317 (12)	0.0189 (8)
C5	0.4396 (3)	0.2040 (4)	0.24267 (12)	0.0209 (9)
C6	0.4645 (3)	0.2017 (4)	0.30139 (12)	0.0188 (8)
H2	0.24690	−0.10380	0.32400	0.0210*
H3	0.20410	−0.09940	0.22490	0.0240*
H5	0.48760	0.28480	0.22210	0.0250*
H6	0.52930	0.28060	0.32090	0.0230*
H11W	0.553 (3)	0.521 (3)	0.5654 (16)	0.0400*
H12W	0.529 (3)	0.344 (3)	0.5764 (14)	0.0400*
H13	0.648 (2)	−0.071 (4)	0.4277 (13)	0.0290*
H21W	0.691 (3)	0.698 (4)	0.5081 (18)	0.0580*
H41	0.254 (2)	0.032 (4)	0.1360 (13)	0.0340*
H42	0.359 (3)	0.177 (3)	0.1374 (14)	0.0340*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sr1	0.0146 (2)	0.0124 (2)	0.0158 (2)	0.0000	0.0004 (1)	0.0000
As1	0.0119 (1)	0.0115 (2)	0.0156 (2)	0.0002 (1)	0.0015 (1)	−0.0001 (1)
O1W	0.0245 (11)	0.0177 (11)	0.0387 (13)	−0.0007 (9)	0.0097 (9)	−0.0027 (10)
O2W	0.051 (2)	0.0175 (17)	0.048 (2)	0.0000	0.0117 (17)	0.0000
O11	0.0218 (10)	0.0129 (9)	0.0224 (10)	−0.0005 (8)	−0.0041 (8)	−0.0018 (9)
O12	0.0153 (9)	0.0228 (10)	0.0220 (11)	0.0016 (8)	0.0052 (7)	0.0053 (9)
O13	0.0130 (9)	0.0144 (9)	0.0304 (11)	0.0012 (8)	0.0011 (8)	−0.0007 (9)
N4	0.0347 (15)	0.0312 (16)	0.0192 (14)	0.0027 (13)	0.0031 (11)	0.0018 (12)
C1	0.0142 (12)	0.0151 (13)	0.0154 (13)	0.0026 (11)	0.0023 (10)	−0.0007 (12)
C2	0.0186 (14)	0.0139 (13)	0.0211 (15)	−0.0026 (11)	0.0036 (11)	−0.0007 (12)
C3	0.0210 (14)	0.0157 (13)	0.0214 (15)	−0.0019 (12)	−0.0012 (11)	−0.0029 (12)
C4	0.0188 (13)	0.0195 (14)	0.0181 (14)	0.0091 (12)	0.0015 (10)	−0.0009 (12)
C5	0.0183 (14)	0.0214 (15)	0.0241 (16)	0.0016 (12)	0.0067 (11)	0.0077 (13)
C6	0.0164 (13)	0.0157 (14)	0.0238 (15)	−0.0018 (11)	0.0010 (11)	0.0005 (12)

Geometric parameters (Å, º)

Sr1—O1W	2.628 (2)	O13—H13	0.88 (2)
Sr1—O2W	2.628 (3)	N4—C4	1.381 (4)
Sr1—O11	2.5527 (18)	N4—H41	0.89 (2)
Sr1—O1W^{i}	2.628 (2)	N4—H42	0.89 (3)
Sr1—O11^{i}	2.5527 (18)	C1—C2	1.391 (4)
Sr1—O12^{ii}	2.549 (2)	C1—C6	1.388 (4)
Sr1—O12^{iii}	2.549 (2)	C2—C3	1.380 (4)
As1—O11	1.6650 (16)	C3—C4	1.399 (4)
As1—O12	1.6705 (19)	C4—C5	1.392 (4)
As1—O13	1.746 (2)	C5—C6	1.378 (4)
As1—C1	1.891 (3)	C2—H2	0.9500
O1W—H11W	0.88 (2)	C3—H3	0.9500
O1W—H12W	0.88 (3)	C5—H5	0.9500
O2W—H21W	0.83 (3)	C6—H6	0.9500
O2W—H21W^{i}	0.83 (3)		
O1W—Sr1—O2W	66.68 (5)	Sr1—O1W—H11W	125 (2)
O1W—Sr1—O11	79.03 (6)	Sr1—O1W—H12W	111.3 (18)
O1W—Sr1—O1W^{i}	133.36 (7)	H11W—O1W—H12W	105 (3)
O1W—Sr1—O11^{i}	106.39 (6)	H21W—O2W—H21W^{i}	100 (3)
O1W—Sr1—O12^{ii}	76.31 (7)	Sr1—O2W—H21W^{i}	130 (2)
O1W—Sr1—O12^{iii}	148.81 (7)	Sr1—O2W—H21W	130 (2)
O2W—Sr1—O11	96.66 (4)	As1—O13—H13	114.2 (19)
O1W^{i}—Sr1—O2W	66.68 (5)	C4—N4—H41	118.6 (19)
O2W—Sr1—O11^{i}	96.66 (4)	H41—N4—H42	116 (3)
O2W—Sr1—O12^{ii}	141.41 (5)	C4—N4—H42	116 (2)
O2W—Sr1—O12^{iii}	141.41 (5)	As1—C1—C6	120.8 (2)
O1W^{i}—Sr1—O11	106.39 (6)	As1—C1—C2	119.7 (2)

O11—Sr1—O11^{i}	166.68 (5)	C2—C1—C6	119.4 (2)
O11—Sr1—O12^{ii}	86.40 (6)	C1—C2—C3	120.5 (3)
O11—Sr1—O12^{iii}	83.19 (6)	C2—C3—C4	120.3 (3)
O1W^{i}—Sr1—O11^{i}	79.03 (6)	N4—C4—C5	120.3 (3)
O1W^{i}—Sr1—O12^{ii}	148.81 (7)	N4—C4—C3	121.0 (3)
O1W^{i}—Sr1—O12^{iii}	76.31 (7)	C3—C4—C5	118.7 (3)
O11^{i}—Sr1—O12^{ii}	83.19 (6)	C4—C5—C6	120.9 (3)
O11^{i}—Sr1—O12^{iii}	86.40 (6)	C1—C6—C5	120.2 (3)
O12^{ii}—Sr1—O12^{iii}	77.18 (7)	C1—C2—H2	120.00
O11—As1—O12	115.50 (10)	C3—C2—H2	120.00
O11—As1—O13	107.81 (9)	C2—C3—H3	120.00
O11—As1—C1	112.69 (11)	C4—C3—H3	120.00
O12—As1—O13	104.40 (10)	C4—C5—H5	120.00
O12—As1—C1	110.29 (9)	C6—C5—H5	119.00
O13—As1—C1	105.26 (10)	C1—C6—H6	120.00
Sr1—O11—As1	129.24 (9)	C5—C6—H6	120.00
Sr1^{ii}—O12—As1	139.61 (11)		

O1W—Sr1—O11—As1	−127.66 (13)	O12—As1—C1—C6	156.6 (2)
O2W—Sr1—O11—As1	167.73 (11)	O13—As1—C1—C2	83.8 (2)
O1W^{i}—Sr1—O11—As1	100.13 (13)	O13—As1—C1—C6	−91.3 (2)
O12^{ii}—Sr1—O11—As1	−50.91 (12)	As1—C1—C2—C3	−173.0 (2)
O12^{iii}—Sr1—O11—As1	26.60 (12)	C6—C1—C2—C3	2.2 (4)
O12—As1—O11—Sr1	111.52 (12)	As1—C1—C6—C5	172.8 (2)
O13—As1—O11—Sr1	−4.75 (14)	C2—C1—C6—C5	−2.3 (4)
C1—As1—O11—Sr1	−120.47 (12)	C1—C2—C3—C4	0.3 (5)
O11—As1—O12—Sr1^{ii}	−109.92 (16)	C2—C3—C4—N4	175.0 (3)
O13—As1—O12—Sr1^{ii}	8.26 (18)	C2—C3—C4—C5	−2.6 (4)
C1—As1—O12—Sr1^{ii}	120.88 (16)	N4—C4—C5—C6	−175.1 (3)
O11—As1—C1—C2	−159.0 (2)	C3—C4—C5—C6	2.6 (5)
O11—As1—C1—C6	25.9 (3)	C4—C5—C6—C1	−0.1 (5)
O12—As1—C1—C2	−28.3 (3)		

Symmetry codes: (i) −*x*+3/2, *y*, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*+1/2, −*y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11*W*···O11^{iv}	0.88 (2)	1.92 (2)	2.791 (3)	173 (3)
O1*W*—H12*W*···O13^{ii}	0.88 (3)	2.02 (2)	2.845 (3)	156 (3)
O13—H13···O12^{iii}	0.88 (2)	1.73 (2)	2.606 (3)	176 (3)
O2*W*—H21*W*···O11^{iv}	0.83 (3)	2.52 (3)	3.267 (2)	149 (3)
N4—H41···O1*W*^{v}	0.89 (2)	2.24 (3)	3.115 (4)	165 (2)
N4—H42···O13^{vi}	0.89 (3)	2.33 (3)	3.210 (4)	170 (3)

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

(IV) *catena*-Poly[[triaquabarium]-bis[μ_2-hydrogen (4-aminophenyl)arsonato-κ^2O:O']]

Crystal data

$[Ba(C_6H_7AsNO_3)_2(H_2O)_3]$
$M_r = 623.92$
Monoclinic, $I2/a$
Hall symbol: -I 2ya
$a = 9.9997$ (8) Å
$b = 7.7305$ (6) Å
$c = 23.979$ (2) Å
$\beta = 98.214$ (7)°
$V = 1834.7$ (3) Å^3
$Z = 4$
$F(000) = 1200$
$D_x = 2.257$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1721 reflections
$\theta = 4.6$–$29.1°$
$\mu = 5.79$ mm^{-1}
$T = 200$ K
Block, colourless
0.36 × 0.22 × 0.16 mm

Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
$T_{min} = 0.470$, $T_{max} = 0.970$
3485 measured reflections
1801 independent reflections
1696 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.4°$
$h = -12 \rightarrow 10$
$k = -9 \rightarrow 7$
$l = -29 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.055$
$S = 1.10$
1801 reflections
137 parameters
7 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0262P)^2 + 0.1187P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.85$ e Å^{-3}
$\Delta\rho_{min} = -0.95$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ba1	0.75000	0.28700 (3)	0.50000	0.0145 (1)
As1	0.43401 (3)	0.05954 (4)	0.40830 (1)	0.0122 (1)
O1W	0.5902 (2)	0.4300 (3)	0.57359 (12)	0.0261 (8)
O2W	0.75000	0.6465 (5)	0.50000	0.0350 (11)

O11	0.5036 (2)	0.2376 (3)	0.43911 (10)	0.0208 (7)
O12	0.30116 (19)	−0.0164 (3)	0.43546 (9)	0.0205 (7)
O13	0.55376 (19)	−0.1064 (3)	0.41966 (10)	0.0182 (7)
N4	0.3301 (3)	0.0886 (4)	0.15278 (13)	0.0264 (10)
C1	0.3921 (3)	0.0829 (4)	0.32900 (13)	0.0143 (9)
C2	0.2955 (3)	−0.0255 (4)	0.29931 (14)	0.0174 (9)
C3	0.2734 (3)	−0.0207 (4)	0.24105 (13)	0.0174 (9)
C4	0.3463 (3)	0.0915 (4)	0.21100 (13)	0.0175 (9)
C5	0.4402 (3)	0.2028 (4)	0.24149 (15)	0.0202 (10)
C6	0.4626 (3)	0.1983 (4)	0.30002 (14)	0.0180 (9)
H2	0.24500	−0.10250	0.31910	0.0210*
H3	0.20750	−0.09500	0.22110	0.0210*
H5	0.48910	0.28230	0.22190	0.0240*
H6	0.52650	0.27470	0.32020	0.0220*
H11W	0.555 (3)	0.534 (3)	0.5741 (18)	0.0390*
H12W	0.534 (3)	0.358 (4)	0.5849 (17)	0.0390*
H13	0.636 (2)	−0.071 (4)	0.4302 (14)	0.0270*
H21W	0.688 (4)	0.715 (5)	0.509 (2)	0.0520*
H41	0.259 (3)	0.040 (5)	0.1339 (14)	0.0320*
H42	0.355 (4)	0.180 (4)	0.1350 (15)	0.0320*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ba1	0.0157 (1)	0.0125 (2)	0.0147 (2)	0.0000	0.0004 (1)	0.0000
As1	0.0107 (2)	0.0117 (2)	0.0143 (2)	0.0007 (1)	0.0019 (1)	0.0008 (1)
O1W	0.0243 (12)	0.0181 (13)	0.0382 (16)	−0.0006 (9)	0.0120 (11)	−0.0007 (12)
O2W	0.048 (2)	0.0165 (19)	0.042 (2)	0.0000	0.0120 (19)	0.0000
O11	0.0238 (11)	0.0140 (11)	0.0230 (13)	0.0004 (9)	−0.0026 (10)	−0.0031 (10)
O12	0.0134 (10)	0.0255 (13)	0.0236 (13)	0.0007 (9)	0.0066 (9)	0.0071 (11)
O13	0.0114 (10)	0.0137 (11)	0.0289 (13)	0.0020 (9)	0.0006 (9)	0.0009 (11)
N4	0.0287 (16)	0.0326 (18)	0.0177 (16)	0.0031 (13)	0.0025 (13)	0.0022 (14)
C1	0.0143 (14)	0.0159 (15)	0.0125 (16)	0.0046 (12)	0.0013 (12)	0.0006 (14)
C2	0.0141 (14)	0.0161 (16)	0.0225 (18)	−0.0022 (12)	0.0041 (12)	0.0012 (15)
C3	0.0147 (14)	0.0146 (15)	0.0221 (17)	−0.0014 (12)	−0.0004 (13)	−0.0016 (14)
C4	0.0167 (15)	0.0184 (16)	0.0172 (17)	0.0074 (13)	0.0023 (13)	−0.0002 (15)
C5	0.0187 (16)	0.0210 (18)	0.0221 (19)	0.0022 (13)	0.0067 (14)	0.0083 (15)
C6	0.0141 (14)	0.0168 (16)	0.0229 (18)	−0.0003 (12)	0.0015 (13)	0.0000 (14)

Geometric parameters (Å, º)

Ba1—O1W	2.774 (2)	O13—H13	0.87 (2)
Ba1—O2W	2.779 (4)	N4—C4	1.382 (4)
Ba1—O11	2.706 (2)	N4—H41	0.87 (3)
Ba1—O1W^{i}	2.774 (2)	N4—H42	0.88 (3)
Ba1—O11^{i}	2.706 (2)	C1—C2	1.395 (4)
Ba1—O12^{ii}	2.694 (2)	C1—C6	1.384 (4)
Ba1—O12^{iii}	2.694 (2)	C2—C3	1.383 (5)

As1—O11	1.667 (2)	C3—C4	1.398 (4)
As1—O12	1.668 (2)	C4—C5	1.400 (4)
As1—O13	1.749 (2)	C5—C6	1.390 (5)
As1—C1	1.897 (3)	C2—H2	0.9500
O1W—H11W	0.88 (2)	C3—H3	0.9500
O1W—H12W	0.86 (3)	C5—H5	0.9500
O2W—H21W	0.87 (4)	C6—H6	0.9500
O2W—H21W^{i}	0.87 (4)		

O1W—Ba1—O2W	66.52 (5)	Ba1—O1W—H11W	130 (3)
O1W—Ba1—O11	80.60 (7)	Ba1—O1W—H12W	114 (2)
O1W—Ba1—O1W^{i}	133.03 (7)	H11W—O1W—H12W	108 (3)
O1W—Ba1—O11^{i}	106.02 (7)	H21W—O2W—H21W^{i}	105 (4)
O1W—Ba1—O12^{ii}	76.71 (7)	Ba1—O2W—H21W^{i}	128 (3)
O1W—Ba1—O12^{iii}	148.07 (6)	Ba1—O2W—H21W	128 (3)
O2W—Ba1—O11	98.11 (5)	As1—O13—H13	114 (2)
O1W^{i}—Ba1—O2W	66.52 (5)	C4—N4—H41	120 (2)
O2W—Ba1—O11^{i}	98.11 (5)	H41—N4—H42	111 (3)
O2W—Ba1—O12^{ii}	140.93 (5)	C4—N4—H42	118 (2)
O2W—Ba1—O12^{iii}	140.93 (5)	As1—C1—C6	120.6 (2)
O1W^{i}—Ba1—O11	106.02 (7)	As1—C1—C2	119.5 (2)
O11—Ba1—O11^{i}	163.77 (7)	C2—C1—C6	119.8 (3)
O11—Ba1—O12^{ii}	88.13 (7)	C1—C2—C3	119.9 (3)
O11—Ba1—O12^{iii}	79.25 (6)	C2—C3—C4	121.2 (3)
O1W^{i}—Ba1—O11^{i}	80.60 (7)	N4—C4—C5	120.7 (3)
O1W^{i}—Ba1—O12^{ii}	148.07 (6)	N4—C4—C3	121.1 (3)
O1W^{i}—Ba1—O12^{iii}	76.71 (7)	C3—C4—C5	118.2 (3)
O11^{i}—Ba1—O12^{ii}	79.25 (6)	C4—C5—C6	120.8 (3)
O11^{i}—Ba1—O12^{iii}	88.13 (7)	C1—C6—C5	120.2 (3)
O12^{ii}—Ba1—O12^{iii}	78.13 (7)	C1—C2—H2	120.00
O11—As1—O12	115.03 (11)	C3—C2—H2	120.00
O11—As1—O13	107.67 (10)	C2—C3—H3	119.00
O11—As1—C1	112.60 (12)	C4—C3—H3	119.00
O12—As1—O13	104.29 (11)	C4—C5—H5	120.00
O12—As1—C1	110.54 (12)	C6—C5—H5	120.00
O13—As1—C1	105.91 (12)	C1—C6—H6	120.00
Ba1—O11—As1	130.25 (11)	C5—C6—H6	120.00
Ba1^{ii}—O12—As1	136.46 (11)		

O1W—Ba1—O11—As1	−129.08 (16)	O12—As1—C1—C6	155.9 (2)
O2W—Ba1—O11—As1	166.49 (14)	O13—As1—C1—C2	84.0 (3)
O1W^{i}—Ba1—O11—As1	98.70 (15)	O13—As1—C1—C6	−91.7 (3)
O12^{ii}—Ba1—O11—As1	−52.24 (15)	As1—C1—C2—C3	−173.9 (2)
O12^{iii}—Ba1—O11—As1	26.03 (14)	C6—C1—C2—C3	1.9 (5)
O12—As1—O11—Ba1	113.92 (14)	As1—C1—C6—C5	173.8 (2)
O13—As1—O11—Ba1	−1.84 (18)	C2—C1—C6—C5	−1.9 (5)
C1—As1—O11—Ba1	−118.21 (16)	C1—C2—C3—C4	−0.1 (5)
O11—As1—O12—Ba1^{ii}	−106.90 (17)	C2—C3—C4—N4	176.0 (3)

O13—As1—O12—Ba1^{ii}	10.78 (18)	C2—C3—C4—C5	−1.7 (5)
C1—As1—O12—Ba1^{ii}	124.21 (17)	N4—C4—C5—C6	−176.0 (3)
O11—As1—C1—C2	−158.6 (2)	C3—C4—C5—C6	1.7 (5)
O11—As1—C1—C6	25.7 (3)	C4—C5—C6—C1	0.1 (5)
O12—As1—C1—C2	−28.4 (3)		

Symmetry codes: (i) −*x*+3/2, *y*, −*z*+1; (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*+1/2, −*y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H11*W*···O11^{iv}	0.88 (2)	1.87 (2)	2.738 (3)	168 (4)
O1*W*—H12*W*···O13^{ii}	0.86 (3)	2.13 (3)	2.902 (3)	149 (3)
O13—H13···O12^{iii}	0.87 (2)	1.77 (2)	2.626 (3)	167 (3)
O2*W*—H21*W*···O11^{iv}	0.87 (4)	2.46 (4)	3.229 (2)	149 (3)
N4—H41···O1*W*^{v}	0.87 (3)	2.23 (3)	3.092 (4)	171 (3)
N4—H42···O13^{vi}	0.88 (3)	2.37 (3)	3.241 (4)	170 (3)

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