

C1—O1—C5—C6	-171.1 (5)	-172.8 (2)	-171.5 (2)
C1—O6—C7—C8	131.1 (4)	113.2 (2)	128.7 (2)
C1—O6—C7—C11	-108.3 (5)	-120.5 (2)	-110.0 (2)
C1—C2—C3—C4	-46.1 (6)	-58.8 (2)	-47.5 (2)
C2—C1—O1—C5	-62.7 (6)	-64.9 (2)	-55.4 (2)
C2—C1—O6—C7	172.2 (4)	155.1 (2)	154.0 (2)
C2—C3—C4—C5	50.8 (6)	57.5 (2)	55.9 (2)
C3—C4—C5—C6	-178.0 (5)	-177.7 (2)	179.2 (2)
C7—C8—C9—C10	-176.8 (4)	166.8 (2)	-160.4 (2)
C8—C7—C11—C12	56.6 (6)	67.4 (2)	173.7 (2)
C9—C8—C7—C11	52.0 (6)	50.5 (2)	168.0 (2)
(4)			
O11—C1—C2—O2	-155.3 (2)	O4—C4—C3—C2	58.8 (3)
O11—C1—C2—C3	82.9 (3)	O4—C4—C5—O5	53.5 (3)
O12—C1—C2—O2	24.4 (3)	O4—C4—C5—C6	-68.5 (3)
O12—C1—C2—C3	-97.5 (3)	O5—C5—C4—C3	-67.2 (3)
O2—C2—C3—O3	-68.8 (3)	O5—C5—C6—O6	61.8 (3)
O2—C2—C3—C4	51.6 (3)	O6—C6—C5—C4	-174.5 (2)
O3—C3—C2—C1	51.9 (3)	C1—C2—C3—C4	172.2 (2)
O3—C3—C4—O4	179.1 (2)	C2—C3—C4—C5	-177.1 (2)
O3—C3—C4—C5	-56.9 (3)	C3—C4—C5—C6	170.8 (2)

Table 5. Asymmetry parameters (Duax & Norton, 1975) of six-membered rings in (1)–(3)

	(1)	(2)	(3)
$\Delta C_3(O1)$	6.8 (3)	2.6 (2)	12.7 (2)
$\Delta C_3(C1)$	15.2 (3)	3.1 (2)	12.4 (2)
$\Delta C_3(C2)$	8.5 (3)	5.4 (2)	1.7 (2)
$\Delta C_2(O1—C1)$	15.6 (3)	1.6 (2)	17.8 (2)
$\Delta C_2(C1—C2)$	16.7 (3)	5.6 (2)	8.5 (2)
$\Delta C_2(C2—C3)$	1.6 (3)	5.9 (2)	9.4 (2)

Data collection and cell refinement were performed using CAD-4 software (Schagen, Straver, van Meurs & Williams, 1989; Frenz, 1986). For all compounds, anisotropic displacement parameters were applied for all non-H atoms. H atoms were found in a difference Fourier map and refined as riding: isotropically for (2) and (4), and with fixed isotropic displacement parameters for (1) and (3). The absolute structures of (1)–(4) were determined by three methods: the Hamilton test (Hamilton, 1965), the Rogers η -test (Rogers, 1981), both using *SHELXTL/PC* (Sheldrick, 1991), and calculation of the Flack parameter x (Flack, 1983) using *SHELXL93* (Sheldrick, 1993). Results for (1): $\eta = 1.04$ (4), $\eta_{inv} = -1.03$ (4); $N = 1587$, $R_{ratio} = 1.006$, $\alpha < 10^{-6}$; $x = 0.06$ (4). For (2): $\eta = 1.01$ (2), $\eta_{inv} = -1.00$ (2); $N = 3035$, $R_{ratio} = 1.688$, $\alpha < 10^{-9}$; $x = 0.02$ (1). For (3): $\eta = 1.4$ (3), $\eta_{inv} = 1.4$ (3); $N = 1581$, $R_{ratio} = 1.0015$, $\alpha = 2.5 \times 10^{-2}$; $x = 0.08$ (16). For (4): $\eta = 1.23$ (4), $\eta_{inv} = -1.19$ (4); $N = 976$, $R_{ratio} = 1.398$, $\alpha < 10^{-9}$; $x = 0.01$ (1). Structure solution and refinement were performed using *SHELXTL/PC*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1260). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CHI 2HU, England.

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m-Anisidinium Dihydrogenmonoarsenate

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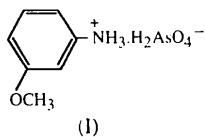
Abstract

In the structure of (1,3-CH₃O-C₆H₄-NH₃)⁺·H₂AsO₄⁻, the H₂AsO₄⁻ and (1,3-CH₃O-C₆H₄-NH₃)⁺ entities alternate in layers perpendicular to the *a* axis. These organic and inorganic layers are held together in the crystal by N—H···O hydrogen bonds. The H₂AsO₄⁻ groups are connected by hydrogen bonds to form pairs, with inter-nal short As···As distances of 4.5 Å.

Comment

The structure and numbering scheme of the title compound, (I), are shown in Figs. 1 and 2. The As···As distance of 4.5 Å between pairs of H₂AsO₄⁻ tetrahedra is significantly shorter than that observed in ethylenediammonium monohydro-

gentetraoxoarsenate(V) (Averbouch-Pouchot & Durif, 1987). All distances and angles in the tetrahedron are generally as expected. The base ring atoms are coplanar



within experimental error and the corresponding plane is perpendicular to the inorganic layer. The structure is stabilized by a hydrogen-bonding network which engages all N and O atoms except O5 belonging to the

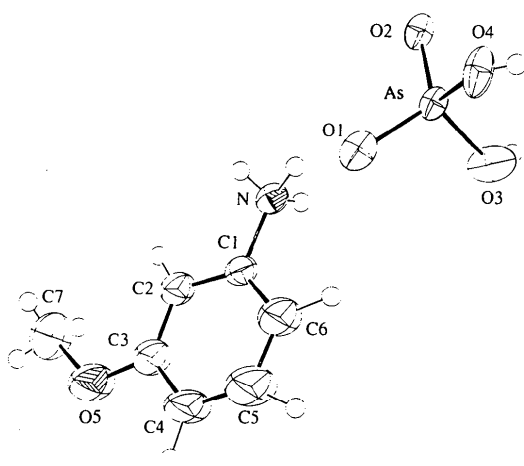


Fig. 1. View of the molecule with 50% probability displacement ellipsoids.

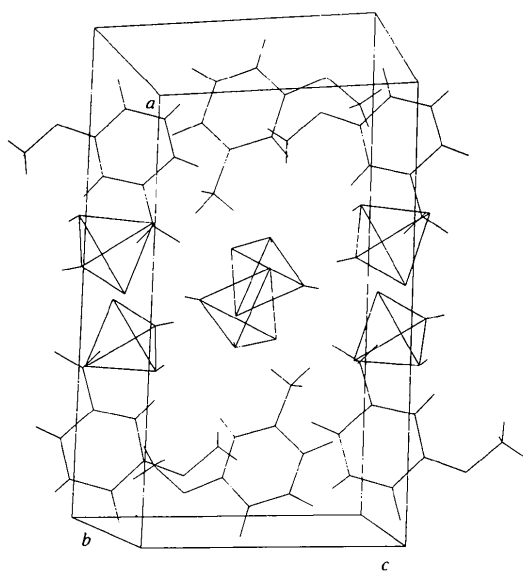


Fig. 2. Diagram showing the packing in the unit cell. Hydrogen bonds are not shown but are reported in Table 3.

organic cation. There is no hydrogen bonding between cations in the inorganic layer. Such a phenomenon is not uncommon in this kind of structure (Abdellahi & Jouini, 1995*a,b*; Averbuch-Pouchot, Durif & Guitel, 1988).

Experimental

Colourless plate-shaped crystals were obtained from an aqueous solution of H_3AsO_4 and *m*-anisidine in stoichiometric ratio by slow evaporation at room temperature.

Crystal data

$C_7H_{10}NO^+ \cdot H_2AsO_4^-$

$M_r = 265.10$

Monoclinic

$P2_1/b$

$a = 15.320(3) \text{ \AA}$

$b = 7.915(1) \text{ \AA}$

$c = 8.763(1) \text{ \AA}$

$\gamma = 93.5(1)^\circ$

$V = 1060.6(3) \text{ \AA}^3$

$Z = 4$

$D_x = 1.66 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.7107 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 12\text{--}16^\circ$

$\mu = 3.202 \text{ mm}^{-1}$

$T = 293(2) \text{ K}$

Plate

$0.20 \times 0.11 \times 0.08 \text{ mm}$

Colourless

Data collection

CAD-4 diffractometer

$\omega/2\theta$ scans [θ -scan width
($1.15 + 0.34\tan\theta$) $^\circ$]

Absorption correction:
none

1932 measured reflections

1861 independent reflections

1454 observed reflections

$[I > 2\sigma(I)]$

$R_{int} = 0.0383$

$\theta_{max} = 25.02^\circ$

$h = 0 \rightarrow 18$

$k = -9 \rightarrow 9$

$l = 0 \rightarrow 10$

3 standard reflections

frequency: 60 min

intensity decay: 2%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.0507$

$wR(F^2) = 0.1525$

$S = 1.304$

1861 reflections

170 parameters

All H-atom parameters

refined except those of

H1N and HC6

$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2$
 $+ 0.9240P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.059$

$\Delta\rho_{max} = 0.854 \text{ e \AA}^{-3}$

$\Delta\rho_{min} = -1.062 \text{ e \AA}^{-3}$

Extinction correction:

SHELXL93 (Sheldrick,
1993)

Extinction coefficient:

0.001 (1)

Atomic scattering factors

from *International Tables*
for *Crystallography* (1992,
Vol. C, Tables 4.2.6.8 and
6.1.1.4)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for H atoms: $U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ for all others.

	x	y	z	U_{eq}/U_{iso}
As	0.43245 (4)	0.24242 (7)	0.96687 (8)	0.0292 (3)
O1	0.3676 (3)	0.3274 (6)	0.8423 (6)	0.044 (1)
O2	0.5285 (3)	0.1915 (5)	0.8994 (5)	0.035 (1)
O3	0.3782 (4)	0.0680 (7)	1.0433 (8)	0.055 (2)
O4	0.4466 (4)	0.3851 (7)	1.1111 (7)	0.049 (2)
O5	0.1047 (4)	0.161 (1)	0.1941 (8)	0.094 (3)

N	0.3344 (4)	0.1491 (8)	0.5808 (8)	0.035 (1)
C1	0.2440 (4)	0.1641 (9)	0.5308 (9)	0.039 (2)
C2	0.2233 (5)	0.157 (1)	0.3794 (9)	0.044 (2)
C3	0.1358 (5)	0.166 (1)	0.340 (1)	0.065 (3)
C4	0.0735 (6)	0.185 (2)	0.448 (1)	0.100 (5)
C5	0.0978 (8)	0.196 (3)	0.597 (2)	0.144 (8)
C6	0.1834 (6)	0.183 (2)	0.642 (1)	0.095 (4)
C7	0.165 (1)	0.154 (3)	0.073 (2)	0.098 (6)
HO3	0.398 (6)	0.002 (11)	1.063 (10)	0.04 (3)
HO4	0.458 (4)	0.346 (8)	1.175 (7)	0.01 (2)

Table 2. Selected bond lengths (Å) and angles (°)

As—O1	1.647 (5)	C1—C6	1.36 (1)
As—O2	1.658 (4)	C1—C2	1.36 (1)
As—O4	1.700 (5)	C2—C3	1.39 (1)
As—O3	1.705 (6)	C3—C4	1.36 (2)
O5—C3	1.36 (1)	C4—C5	1.36 (2)
O5—C7	1.40 (2)	C5—C6	1.38 (2)
N—C1	1.463 (9)		
O1—As—O2	115.4 (2)	C2—C1—N	120.4 (6)
O1—As—O4	106.1 (3)	C1—C2—C3	117.5 (7)
O2—As—O4	110.3 (3)	C4—C3—O5	114.0 (7)
O1—As—O3	108.4 (3)	C4—C3—C2	121.4 (8)
O2—As—O3	109.8 (2)	O5—C3—C2	124.6 (8)
O4—As—O3	106.4 (3)	C3—C4—C5	118.8 (9)
C3—O5—C7	118.7 (9)	C4—C5—C6	122.2 (10)
C6—C1—C2	122.9 (7)	C1—C6—C5	117.2 (9)
C6—C1—N	116.7 (7)		

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N—H1N...O1	1.096 (6)	1.696 (5)	2.723 (8)	153.9 (4)
N—H2N...O1 ⁱ	0.84 (9)	1.88 (9)	2.712 (8)	173 (8)
N—H3N...O2 ⁱⁱ	0.82 (8)	2.08 (8)	2.864 (9)	160 (7)
O3—HO3...O2 ⁱⁱⁱ	0.64 (8)	1.98 (8)	2.621 (7)	172 (10)
O4—HO4...O2 ^{iv}	0.67 (6)	2.00 (6)	2.632 (7)	157 (7)

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

Data were corrected for Lorentz-polarization effects. The structure was solved by direct methods and refined by full-matrix least-squares methods.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1130). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Supramolecular Structure of a Cadmium–Silver Complex Forming a Two-Dimensional Network Embracing One-Dimensional Chains in a Layered Crystal Structure

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Abstract

The polymeric coordination compound, poly[*trans*-bis(4-aminopyridine)cadmium(II)-bis[μ -dicyanoargentato(I)-*N:N'*]] – *catena*-poly[(2-aminoethanol-*N,O*)-(4-aminopyridine)[dicyanoargentato(I)-*N*]cadmium(II)- μ -[dicyanoargentato(I)-*N:N'*]] (1/2), {[Cd(4-ampy)₂ – { μ -Ag(CN)₂}]₂}. [Cd(meas)(4-ampy){Ag(CN)₂}]₂ – { μ -Ag(CN)₂}]₂]_n (4-ampy = 4-aminopyridine, C₅H₆N₂; mea = 2-aminoethanol, C₂H₇NO) has a supramolecular structure composed of a two-dimensional network of Cd–NCAgCN–Cd spans, where each Cd is ligated by two 4-ampy ligands in *trans* positions, embracing one-dimensional chains of (–Cd–NCAgCN–)_n. In these chains each Cd is monodentately ligated by an –NCAgCN ‘branch’ and a 4-ampy ligand, and bidentately chelated by mea. The network is stacked along the *b* axis to give a layered crystal structure, in which each monodentate –NCAgCN branch protruding from the chain penetrates through the mesh to form a hydrogen bond with the OH group of the mea ligand in the crystallographically equivalent chain in the adjacent layer.

Comment

Dicyanoargentate(I) linking other coordination centres such as Cd^{II}, Cu^I and Zn^{II} has provided a variety of self-assembled multidimensional structures: a three-dimensional textile interwoven by the two-dimensional networks of *trans*-[Cd(NH₃)₂{Ag(CN)₂}]₂_n (Soma & Iwamoto, 1995); a two-dimensional layer clathrate stacked by the doubly interwoven net-