

complete set of 4220 reflexions.* Scattering factors for neutral atoms and f' , f'' from *International Tables for X-ray Crystallography* (1974). Enraf-Nonius (1977) SDP used for all calculations. Computer: MicroVAX II.

Discussion. The crystal structure determination shows clearly that the atomic arrangement is built up by a stacking of $C_7H_8NO_2^+$ and $H_2AsO_4^-$ units [Figs. 1 and 2; *STRUPLO84* (Fischer, 1985)]. $H_2AsO_4^-$ anions are bonded together by O(1)—H(1)···O(4) and O(2)—H(2)···O(3) hydrogen bonds so as to form layers along the (100) planes. Two types of As—O distances are observed in AsO_4 tetrahedra depending on whether O atoms are hydrogen donors (1.702, 1.719 Å) or acceptors (1.660, 1.661 Å). The two cations are found between these planes of anions. They are bonded to $H_2AsO_4^-$ sheets by hydrogen atoms of the NH_3^+ groups: N—H(8)···O(4), N—H(9)···O(2), N—H(7)···O(4), and carboxylic groups: O(5)—H(10)···O(3). Such a type of two-dimensional $H_2XO_4^-$ ($X = P, As$) network has been pointed out by Adams (1977) in aminoguanidinium dihydrogenmonophosphate. Glycine monophosphate

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(Averbuch-Pouchot, Durif & Guitel, 1988) presents layers of $H_2PO_4^-$ connected anions which differ from the previous by the ratio of oxygen donors/oxygen acceptors of H atoms. In the crystal structures of alkali and ammonium dihydrogenmonophosphates two-dimensional or three-dimensional frameworks of $H_2PO_4^-$ connected ions have been found depending on the dimensions of the associated cations. In $R-NH_3^+ \cdot H_2XO_4^-$ ($X = P, As$) organic compounds the steric hindrance of the $R-NH_3^+$ cation has an effect upon the $H_2XO_4^-$ connexion in chains, sheets or three-dimensional networks through the hydrogen bonds. Tables 1 and 2 report the final atomic coordinates and the main interatomic distances.

References

- ADAMS, J. M. (1977). *Acta Cryst.* **B33**, 1513–1515.
 AVERBUCH-POUCHOT, M. T., DURIF, A. & GUITEL, J. C. (1988). *Acta Cryst.* **C44**, 99–102.
 ENRAF-NONIUS (1977). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
 FISCHER, R. X. (1985). *J. Appl. Cryst.* **18**, 258–262.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 MAIN, P., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 TORDJMAN, I., MASSE, R. & GUITEL, J. C. (1988). *Acta Cryst.* **C44**, 2057–2059.

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Structure of 4-Carboxyanilinium Dihydrogenmonoarsenate Monohydrate

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Abstract. $C_7H_8NO_2^+ \cdot H_2AsO_4^- \cdot H_2O$, $M_r = 297.1$, monoclinic, $P2_1/c$, $a = 14.754$ (14), $b = 8.603$ (4), $c = 8.982$ (9) Å, $\beta = 107.65$ (8)°, $V = 1086.4$ (5) Å³, $Z = 4$, $D_x = 1.816$ Mg m⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 3.325$ mm⁻¹, $F(000) = 600$, $T = 295$ K, final $R = 0.033$ for 2939 unique reflexions. Two files of $H_2AsO_4^-$ tetrahedra bridged by water molecules form sheets of anions which alternate with sheets of $C_7H_8NO_2^+$ cations.

Introduction. The interactions between amino acids, primary amines and monophosphoric and mono-

arsenic acids have prompted us to develop an interest in chemical reactions between cyclic amines and these acids. The present work is devoted to a detailed structure of the $C_7H_8NO_2^+ \cdot H_2AsO_4^- \cdot H_2O$ compound obtained by reaction of 4-aminobenzoic acid with H_3AsO_4 acid. In the preceding paper (Tordjman, Masse & Guitel, 1988) we described the preparation method of the three monophosphates and monoarsenates of the aminobenzoic acid isomers.

Experimental. Single crystals were easily prepared by slow evaporation at room temperature of an

Table 1. Final atomic coordinates, B_{eq} for non-H atoms and B_{iso} for H atoms
$$B_{eq} = \frac{4}{3} \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}/B_{iso}(\text{\AA}^2)$
As	0.36554 (2)	0.33578 (3)	0.27779 (3)	1.822 (4)
O(1)	0.2935 (1)	0.1861 (2)	0.2136 (3)	2.64 (4)
O(2)	0.4168 (1)	0.4072 (3)	0.1509 (2)	2.13 (3)
O(3)	0.3059 (1)	0.4857 (3)	0.3334 (3)	3.05 (4)
O(4)	0.4565 (1)	0.2841 (3)	0.4390 (3)	3.12 (4)
O(5)	0.1127 (1)	0.2099 (3)	0.0963 (3)	3.31 (5)
O(6)	0.1197 (1)	0.4267 (3)	0.2364 (3)	3.71 (5)
O(7)	0.3892 (1)	0.9067 (3)	0.2528 (2)	2.37 (4)
N	0.3319 (1)	0.8138 (3)	0.5150 (3)	2.10 (4)
C(1)	0.2279 (2)	0.8180 (3)	0.4769 (3)	1.92 (4)
C(2)	0.1810 (2)	0.6927 (4)	0.5162 (4)	2.62 (6)
C(3)	0.0824 (2)	0.6970 (4)	0.4774 (4)	2.73 (6)
C(4)	0.0320 (2)	0.8215 (4)	0.3977 (3)	2.05 (4)
C(5)	0.0800 (2)	0.9475 (4)	0.3608 (4)	2.58 (5)
C(6)	0.1793 (2)	0.9464 (4)	0.4008 (4)	2.59 (5)
C(7)	0.0747 (2)	0.3244 (4)	0.1515 (3)	2.29 (5)
H(1)	0.258 (3)	0.456 (6)	0.291 (5)	5 (1)
H(2)	0.218 (3)	0.890 (5)	0.085 (5)	5 (1)
H(3)	0.055 (3)	0.889 (5)	-0.009 (4)	4.3 (9)
H(4)	0.435 (3)	0.236 (5)	0.480 (5)	4 (1)
H(5)	0.047 (3)	0.037 (5)	0.303 (4)	3.5 (8)
H(6)	0.207 (3)	0.018 (6)	0.359 (5)	5 (1)
H(7)	0.357 (2)	0.602 (4)	0.059 (4)	2.3 (7)
H(8)	0.354 (2)	0.766 (4)	0.066 (3)	2.1 (7)
H(9)	0.359 (2)	0.823 (5)	0.427 (4)	3.1 (8)
H(10)	0.173 (4)	0.201 (7)	0.144 (6)	7 (1)
H(11)	0.450 (3)	0.918 (5)	0.280 (4)	4.0 (9)
H(12)	0.361 (4)	-0.032 (8)	0.223 (7)	10 (2)

Table 2. Main interatomic distances (\AA) and bond angles ($^\circ$)

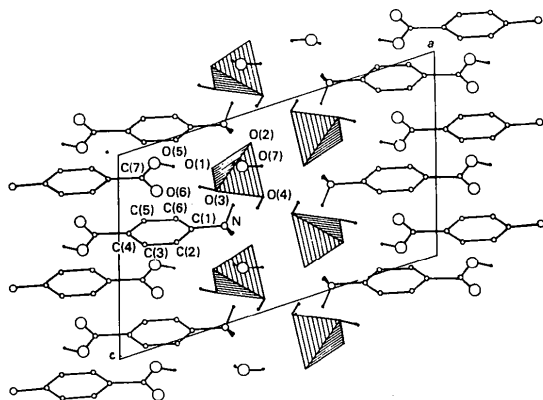
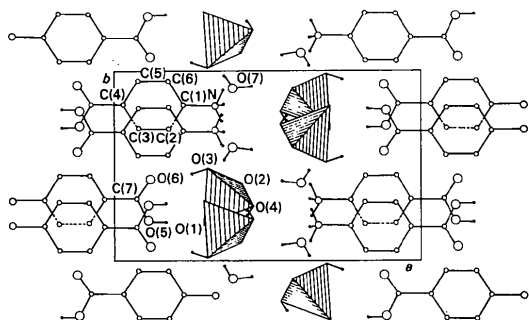
AsO ₄ tetrahedron			
As	O(1)	O(2)	O(3)
O(1)	1.658 (2)	2.805 (3)	2.778 (3)
O(2)	115.2 (1)	1.665 (2)	2.729 (3)
O(3)	110.7 (1)	107.5 (1)	1.719 (2)
O(4)	110.4 (1)	105.9 (1)	106.8 (1)

NH ₃ C ₆ H ₄ COOH group			
C(1)—C(2)	1.383 (4)	C(6)—C(1)—C(2)	121.7 (2)
C(2)—C(3)	1.391 (4)	C(1)—C(2)—C(3)	118.8 (3)
C(3)—C(4)	1.375 (4)	C(2)—C(3)—C(4)	120.8 (3)
C(4)—C(5)	1.390 (4)	C(3)—C(4)—C(5)	119.8 (3)
C(5)—C(6)	1.399 (4)	C(4)—C(5)—C(6)	120.2 (3)
C(6)—C(1)	1.380 (4)	C(5)—C(6)—C(1)	118.3 (3)
C(2)—H(2)	0.99 (4)	C(1)—C(2)—H(2)	120 (3)
C(3)—H(3)	0.87 (4)	C(3)—C(2)—H(2)	121 (3)
C(5)—H(5)	0.97 (4)	C(2)—C(3)—H(3)	116 (3)
C(6)—H(6)	0.89 (5)	C(4)—C(3)—H(3)	122 (3)
C(7)—C(4)	1.500 (3)	C(4)—C(5)—H(5)	122 (2)
C(7)—O(5)	1.304 (4)	C(6)—C(5)—H(5)	117 (2)
C(7)—O(6)	1.221 (4)	C(5)—C(6)—H(6)	118 (3)
O(5)—O(6)	2.235 (4)	C(1)—C(6)—H(6)	122 (3)
C(1)—N	1.468 (3)	C(4)—C(7)—O(5)	114.1 (2)
		C(4)—C(7)—O(6)	121.3 (3)
		O(5)—C(7)—O(6)	124.6 (2)
		C(3)—C(4)—C(7)	121.3 (3)
		C(5)—C(4)—C(7)	118.8 (2)
		C(2)—C(1)—N	119.4 (2)
		C(6)—C(1)—N	119.0 (2)

Hydrogen bonds

(O,N)—H	H...O	(O,N)—O	(O,N)—H...O
O(3)—H(1)...O(6)	0.74 (4)	1.96 (4)	2.667 (3)
O(4)—H(4)...O(2)	0.69 (5)	2.05 (4)	2.711 (3)
O(5)—H(10)...O(1)	0.86 (5)	1.71 (5)	2.563 (3)
O(7)—H(11)...O(2)	0.86 (4)	1.88 (4)	2.728 (2)
O(7)—H(12)...O(1)	0.68 (6)	2.11 (7)	2.755 (3)
N—H(7)...O(2)	0.85 (3)	1.96 (3)	2.809 (3)
N—H(8)...O(7)	0.84 (3)	2.00 (3)	2.789 (3)
N—H(9)...O(7)	0.99 (4)	1.90 (4)	2.850 (3)
			159 (5)
			161 (5)
			172 (5)
			170 (4)
			160 (7)
			177 (3)
			156 (3)
			161 (3)

ethanolic solution of 4 aminobenzoic acid and H₃AsO₄ in a stoichiometric ratio. Large, multifaced monoclinic prisms of up to 5 mm size may be obtained. Crystal size used for data collection: 0.50 × 0.50 × 0.45 mm. Density not measured. Nicolet-XRD diffractometer, graphite monochromator. Systematic absences: $h0l, l = 2n$; $0k0, k = 2n$. 21 reflexions ($10.5 < \theta < 12.5^\circ$) used for refining unit-cell dimensions. ω scan. 5224 non-zero reflexions collected ($1.5 < \theta < 35^\circ$). $\pm h, k, l, h_{max} = 24, k_{max} = 14, l_{max} = 15$. Scan width 1.40° , scan speed from 0.017 to $0.050^\circ \text{ s}^{-1}$, total background measuring time: 6 to 17 s. Orientation reference reflexions: 631, $6\bar{3}1, 6\bar{3}\bar{1}$, no variation. Lorentz and polarization correction, no absorption correction. Structure solved by direct methods (MULTAN77; Main, Lessinger, Germain, Declercq & Woolfson, 1977) and successive Fourier syntheses. H atoms located from difference Fourier map. Anisotropic full-matrix least-squares refinement (on F), isotropic for H atoms. Unit weights. Final refinement cycles with 2939 reflexions corresponding to $I > 3\sigma_I$. Final $R = 0.033$ ($wR = 0.036$). Max. $\Delta/\sigma = 0.15$. $S = 1.04$. Max. peak height in the final difference

Fig. 1. Projection along the b direction of the $C_7H_5O_2NH_3^+ \cdot H_2AsO_4^- \cdot H_2O$ framework. Aromatic H atoms are omitted.Fig. 2. ab projection of the $C_7H_5O_2NH_3^+ \cdot H_2AsO_4^- \cdot H_2O$ framework.

Fourier synthesis $0.55 \text{ e } \text{Å}^{-3}$. No extinction correction. $R = 0.068$ for the complete set of 4731 reflexions.* Scattering factors for neutral atoms and f' , f'' from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1977) *SDP* used for all calculations. Computer: MicroVAX II.

Discussion. The framework is built up of stacks of H_2AsO_4^- anions, $\text{C}_7\text{H}_8\text{NO}_2^+$ cations and water molecules [Figs. 1 and 2; *STRUPLO84* (Fischer, 1985)]. Two separate lines of H_2AsO_4^- tetrahedra run along a plane parallel to bc . In each line, H_2AsO_4^- tetrahedra are bonded together through $\text{O}(4)–\text{H}(4) \cdots \text{O}(2)$ bridges. The two adjacent lines are bridged by water molecules $\text{O}(7)$. The $(\text{H}_2\text{AsO}_4^-)–\text{H}_2\text{O}–(\text{H}_2\text{AsO}_4^-)$ anionic framework extends in a plane parallel to bc and forms a thick layer of tetrahedra. The 4-carboxyanilinium groups connect two anionic layers of tetrahedra through hydrogen bonds of the NH_3^+ and

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Tetraethylammonium *catena*- μ_4 -Chloro-di- μ -chloro-diargentate(I) and Tetraethylammonium *catena*- μ_4 -Bromo-di- μ -bromo-diargentate(I)

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Abstract. $[\text{N}(\text{C}_2\text{H}_5)_4][\text{Ag}_2\text{Cl}_3]$, $M_r = 452.4$, orthorhombic, $Pnma$, $a = 20.773$ (5), $b = 11.895$ (3), $c = 16.683$ (3) Å, $V = 4122$ (2) Å³, $Z = 12$, $D_x = 2.19 \text{ Mg m}^{-3}$, $\text{Mo } K\alpha$, $\lambda = 0.71069$ Å, $\mu = 3.38 \text{ mm}^{-1}$, $F(000) = 2640$, $T = 290 \text{ K}$, $R = 0.047$ for 2310 unique observed [$I \geq 3\sigma(I)$] reflections and 311 parameters. $[\text{N}(\text{C}_2\text{H}_5)_4][\text{Ag}_2\text{Br}_3]$, $M_r = 585.7$, orthorhombic, $Pnma$, $a = 21.161$ (7), $b = 12.207$ (3), $c = 17.208$ (4) Å, $V = 4445$ (2) Å³, $Z = 12$, $D_x = 2.63 \text{ Mg m}^{-3}$, $\text{Mo } K\alpha$, $\lambda = 0.71069$ Å, $\mu = 10.6 \text{ mm}^{-1}$, $F(000) = 3288$, $T = 290 \text{ K}$, $R = 0.062$ for 2964 unique observed [$I \geq 3\sigma(I)$] reflections and 311 parameters. Both compounds contain infinite double-chain anions composed of edge-sharing silver(I) halide tetrahedra. Ag–Cl distances range from 2.514 (2) to 2.778 (2) Å

and Ag–Br distances from 2.630 (2) to 2.844 (2) Å. The Ag \cdots Ag contacts are 3.348 (2)–3.486 (2) Å and 3.250 (3)–3.547 (2) Å in $[\text{N}(\text{C}_2\text{H}_5)_4][\text{Ag}_2\text{Cl}_3]$ and $[\text{N}(\text{C}_2\text{H}_5)_4][\text{Ag}_2\text{Br}_3]$, respectively.

carboxylic groups. As in the previous structure we observe a layer structure with planes of anions separated from planes of cations. The anionic arrangement is near that described for $(\text{H}_2\text{PO}_4^-, \text{H}_2\text{O}, \text{H}_2\text{PO}_4^-)$ in $\text{N}(\text{CH}_3)_4\text{H}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ material (Ohama, Machida, Nakamura & Kunifujii, 1987).

Tables 1 and 2 report the final atomic coordinates and the main interatomic distances.

References

- Enraf–Nonius (1977). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
FISCHER, R. X. (1985). *J. Appl. Cryst.* **18**, 258–262.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
MAIN, P., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
OHAMA, N., MACHIDA, M., NAKAMURA, T. & KUNIFUJI, Y. (1987). *Acta Cryst.* **C43**, 962–964.
TORDJMAN, I., MASSE, R. & GUITEL, J. C. (1988). *Acta Cryst.* **C44**, 2055–2057.

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