two main points should be noticed: there is no intermolecular bonding between the organic groups; only the external O atoms of the P_4O_{12} ring are involved in hydrogen bonds. Each glycine group is connected by hydrogen bonds to three different phosphoric groups.

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Structure of L-Histidinium Dihydrogenmonophosphate Monohydrate

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Abstract. $C_6H_{10}N_3O_2^+.H_2PO_4^-.H_2O$, $M_r = 271{\cdot}17$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}923\,(5)$, $b = 14{\cdot}383\,(9)$, $c = 8{\cdot}362\,(5)$ Å, $V = 1073\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}678$ Mg m^{-3}, $\lambda(\mathrm{Ag}\,K\alpha) = 0{\cdot}5608$ Å, $\mu = 0{\cdot}163$ mm^{-1}, $F(000) = 568$, $T = 293$ K, final $R = 0{\cdot}036$ for 2024 independent observed reflexions. Planes of $C_6H_{10}N_3O_2^+$ groups perpendicular to the b axis alternate with planes containing $H_2PO_4^-$ groups and water molecules. The $H_2PO_4^-$ groups are linked by hydrogen bonds to form infinite chains, which are themselves interconnected with the histidine planes through several other hydrogen bonds.

Introduction. Recently the first phosphate salt of L-histidine was described (Blessing, 1986). This salt has a 1:2 stoichiometry corresponding to the formula $His.2H_3PO_4$. The compound is in fact a 1:1 salt syncrystallized with a molecule of orthophosphoric acid and must be formulated as $HisH^+.H_2PO_4^-.H_3PO_4$. In the present work we describe the chemical preparation and the crystal structure of the monohydrate of the 1:1 salt: $C_6H_{10}N_3O_2^+.H_2PO_4^-.H_2O$.

Experimental. Crystals of the title compound have been prepared by mixing two dilute aqueous solutions of L-histidine and H_3PO_4 so as to obtain an equimolar ratio in the resulting solution. This solution is then kept at room temperature until a dry solid gel is obtained (2 to 3 months). Then a few drops of distilled water are added every day over several weeks, until some small crystalline areas dispersed in the gel appear. At this step, daily additions of larger quantities of water accelerate the crystallization. After additional weeks large orthorhombic prisms, up to 8 mm long, are obtained. Density not measured. $0{\cdot}42 \times 0{\cdot}30 \times 0{\cdot}30$ mm prism fragment Philips PW 1100 diffractometer, graphite monochromator. Systematic absences: $h00$: $h = 2n$; $0k0$: $k = 2n$; $00l$: $l = 2n$. 16 reflexions ($10{\cdot}5 < \theta < 16°$) for refining unit-cell dimensions. ω scan. Scan width: $1{\cdot}20°$, scan speed: $0{\cdot}025°\ \mathrm{s}^{-1}$, total background measuring time: 10 s. 2708 reflexions measured ($3 < \theta < 30°$), h,k,l, $h_{\max} = 15$, $k_{\max} = 23$, $l_{\max} = 15$.

Two intensity and orientation reference reflexions every 2 h (250 and $\bar{2}\bar{5}0$): no significant variation. Lorentz and polarization corrections, no absorption correction. Direct methods (*MULTAN*77, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1977) used for structure determination. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. H atoms located by difference Fourier map. Unit weights. Final refinement with 2024 reflexions ($I > 4\sigma_I$). Final $R = 0{\cdot}036$ ($wR = 0{\cdot}038$), $S = 0{\cdot}607$, max. $\Delta/\sigma = 0{\cdot}07$. Max. peak height in final difference Fourier synthesis $0{\cdot}42$ e Å^{-3}. No extinction correction. 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 used: VAX 780.

Discussion. Table 1 reports the final atomic coordinates* and Fig. 1 is a projection along the c axis of the atomic arrangement.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and distances and angles involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44708 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final atomic coordinates and* B_{eq} *values* (Å^2)

E.s.d.'s are given in parentheses.

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i \sum_j \mathbf{a}_i . \mathbf{a}_j \beta_{ij}.$$

	x	y	z	B_{eq}
P	0·58363 (6)	0·24840 (4)	0·14133 (7)	1·396 (6)
O(1)	0·6221 (2)	0·3553 (1)	0·1298 (3)	2·17 (3)
O(2)	0·7336 (2)	0·1985 (1)	0·0992 (2)	2·29 (3)
O(3)	0·5466 (2)	0·2227 (1)	0·3128 (2)	1·99 (3)
O(4)	0·9638 (2)	0·2778 (1)	0·9768 (2)	2·13 (3)
O(5)	0·5819 (2)	0·0411 (1)	0·5509 (2)	2·30 (3)
O(6)	0·8747 (2)	0·0511 (1)	0·8401 (3)	2·62 (3)
O(*W*)	0·0047 (3)	0·2822 (2)	0·3150 (3)	3·71 (5)
N(1)	0·3010 (2)	0·3410 (2)	0·5270 (3)	2·13 (4)
N(2)	0·1148 (2)	0·0534 (2)	0·2312 (2)	1·78 (3)
N(3)	0·7967 (2)	0·3774 (1)	0·6503 (3)	1·90 (3)
C(1)	0·3432 (3)	0·5144 (2)	0·0677 (3)	1·59 (3)
C(2)	0·3033 (2)	0·4342 (2)	0·6032 (3)	1·34 (3)
C(3)	0·0599 (3)	0·5100 (2)	0·0467 (3)	1·71 (3)
C(4)	0·5830 (2)	0·4489 (2)	0·6069 (2)	1·41 (3)
C(5)	0·6981 (3)	0·4047 (2)	0·5330 (3)	1·76 (3)
C(6)	0·7437 (3)	0·4027 (2)	0·7911 (3)	1·95 (4)

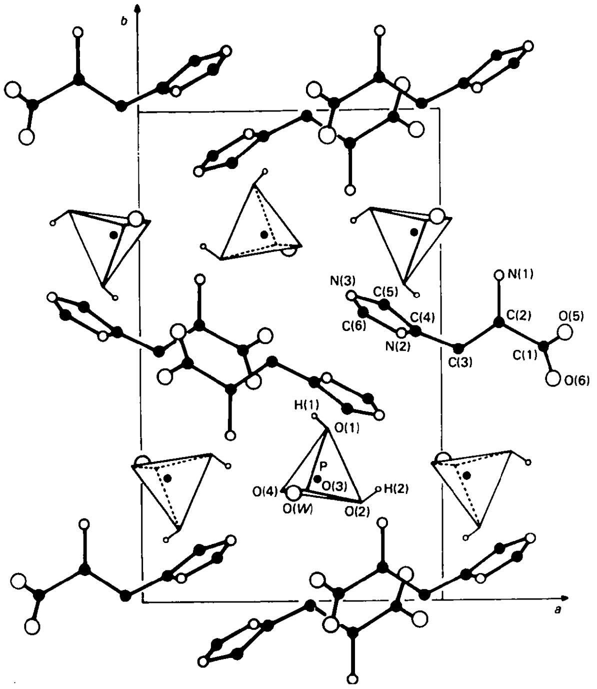

Fig. 1. Projection along the c axis of the atomic arrangement of L-histidinium dihydrogenmonophosphate monohydrate.

The structure can be described as being built up by chains of $H_2PO_4^-$ groups spreading in planes $y \simeq 0{\cdot}25$ and 0·75 alternating with planes ($y \simeq 0{\cdot}0$ and 0·5) containing the histidine groups.

The $H_2PO_4^-$ groups are connected in the chain by hydrogen bonds [O(2)–H···O(4)] with a P···P distance of 5·049 Å. These chains are themselves interconnected with the histidine planes through several other hydrogen bonds whose geometrical features are reported in Table 2. Water molecules located in the same planes as the phosphoric groups are connected by hydrogen bonds to an O atom [O(4)] of the $H_2PO_4^-$ group and to an O atom [O(6)] of the carboxylic group.

Table 2. *Main interatomic distances* (Å) *and bond angles* (°) *in the atomic arrangement*

E.s.d.'s are given in parentheses.

The PO_4 tetrahedron

P	O(1)	O(2)	O(3)	O(4)
O(1)	1·578 (3)	2·477 (4)	2·536 (4)	2·540 (4)
O(2)	104·3 (1)	1·558 (2)	2·469 (3)	2·513 (3)
O(3)	110·0 (2)	106·8 (1)	1·517 (2)	2·532 (3)
O(4)	111·0 (2)	110·3 (1)	113·9 (1)	1·504 (2)

P–O(1)–H(1)	118 (8)	P–O(2)–H(2)	116 (4)

The histidine group

C(1)–O(5)	1·257 (4)	C(4)–C(5)	1·357 (4)
C(1)–O(6)	1·249 (4)	C(4)–N(2)	1·384 (4)
C(1)–C(2)	1·531 (4)	C(5)–N(3)	1·375 (4)
C(2)–N(1)	1·485 (4)	C(6)–N(3)	1·320 (5)
C(2)–C(3)	1·535 (4)	C(6)–N(2)	1·325 (4)
C(3)–C(4)	1·493 (4)		

C(2)–C(1)–O(5)	117·4 (3)	C(3)–C(4)–C(5)	132·7 (3)
C(2)–C(1)–O(6)	115·9 (3)	C(3)–C(4)–N(2)	121·3 (3)
O(5)–C(1)–O(6)	126·8 (3)	N(2)–C(4)–C(5)	106·2 (3)
N(1)–C(2)–C(1)	109·9 (3)	C(4)–C(5)–N(3)	107·1 (3)
N(1)–C(2)–C(3)	110·5 (3)	N(2)–C(6)–N(3)	108·5 (3)
C(1)–C(2)–C(3)	111·5 (2)	C(4)–N(2)–C(6)	109·1 (3)
C(2)–C(3)–C(4)	111·6 (2)	C(5)–N(3)–C(6)	109·1 (3)

The hydrogen bonds

	O(N)–H	H···O	O(N)···O	O(N)–H···O
O(1)–H(1)···O(6)	0·49 (6)	2·13 (6)	2·598 (4)	160 (10)
O(2)–H(2)···O(4)	0·73 (5)	1·85 (5)	2·562 (3)	164 (5)
O(*W*)–H(1*W*)···O(5)	0·71 (6)	2·16 (6)	2·861 (5)	171 (6)
O(*W*)–H(2*W*)···O(4)	0·71 (7)	2·15 (7)	2·852 (4)	169 (6)
N(1)–H(1N1)···O(*W*)	0·82 (4)	2·06 (5)	2·862 (5)	166 (4)
N(1)–H(2N1)···O(3)	0·84 (5)	2·00 (5)	2·791 (4)	156 (4)
N(1)–H(3N1)···O(5)	0·88 (5)	2·55 (6)	2·668 (4)	88 (4)*
N(2)–H(N2)···O(5)	0·79 (4)	1·94 (4)	2·672 (4)	157 (4)
N(3)–H(N3)···O(3)	0·73 (6)	1·97 (6)	2·684 (4)	162 (6)

H(1*W*)–O(*W*)–H(2*W*)	112 (6)

* Cannot be considered as a hydrogen bond. See *Discussion*.

The electronic situation of the histidine molecule is that reported in Fig. 2. Main interatomic distances and bond angles in the molecule are reported in Table 2.

The hydrogen bonds are what can be expected in this kind of compound with the exception of an unusual situation for one H atom [H(3N1)] belonging to the $N(1)H_3$ group. Within a range of 3 Å, N(1) has three oxygen neighbours [O(3), O(5) and O(*W*)] with respective distances of 2·791, 2·668 and 2·862 Å. The shortest one suggests an N(1)–H···O(5) bond but the corresponding N–H···O angle is quite unusual (88°) and in addition such a possibility corresponds to an intramolecular bonding. It is probable that N(1)–

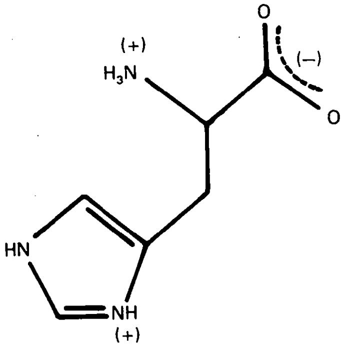

Fig. 2. Electronic situation of the L-histidine group in the title compound.

H(3N1) has difficulties in finding an acceptor which is properly located. This situation is not uncommon, mainly when a hydrogen-rich group like NH_3^+ belongs to a large organic group (Olovsson & Jönsson, 1976).

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(5*RS*)-3,4-Dichloro-5-dichloromethyl-5-acetoxy-2(5*H*)-furanone

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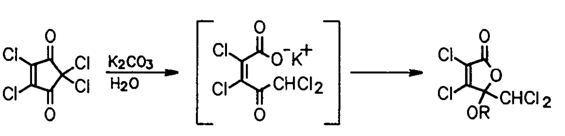
Abstract. 3,4-Dichloro-5-dichloromethyl-2(5*H*)-oxo-5-furyl acetate, $C_7H_4Cl_4O_4$, $M_r = 293{\cdot}92$, monoclinic, $P2_1/n$, $a = 16{\cdot}139\ (1)$, $b = 8{\cdot}9486\ (5)$, $c = 15{\cdot}994\ (1)$ Å, $\beta = 96{\cdot}669\ (8)°$, $V = 2294{\cdot}2\ (3)$ Å^3, $Z = 8$, $D_x = 1{\cdot}702$ g cm^{-3}, Cu $K\alpha$, $\lambda_{\alpha_1} = 1{\cdot}540562$ Å, $\mu = 95{\cdot}8$ cm^{-1}, $F(000) = 1168$, 295 K, $R = 0{\cdot}062$ ($wR = 0{\cdot}067$) for 2689 unique reflections with $I \geq 2\sigma(I)$. The asymmetric unit contains two virtually identical molecules having normal geometry. Molecules are linked by possible weak C—H···O hydrogen bonds, C···O = 3·311 (9) and 3·23 (1) Å.

Introduction. Treatment of 2,2,4,5-tetrachlorocyclopentene-1,3-dione (1), a constituent of pulp mill bleach liquors (McKague, de Sousa, Strömberg & Kringstad, 1987), with potassium carbonate and acetic anhydride gives a crystalline acetate which is identical to a compound which appears during the analysis of chlorophenols in the liquors (Strömberg, de Sousa, Ljungquist, McKague & Kringstad, 1987). Thus, a simple method was available by which material could be prepared for identification much more easily than by isolation from the liquor. This paper describes the X-ray structure of the acetate; other spectral data have been published separately (Strömberg *et al.*, 1987).



(1) (2) (3*a*) R=OH
(3*b*) R=OAc

Experimental. Colorless crystals, 0·25 × 0·32 × 0·35 mm, faces {10$\bar{1}$}, {110}, {011}, {100}. Enraf–Nonius CAD-4F diffractometer, lattice parameters from 25 reflections with $\theta = 25$–41°, intensities for $\theta \leq 75°$, *hkl*: 0 to 20, 0 to 11, −20 to 20, ω–2θ scan, ω scan width $(0{\cdot}75 + 0{\cdot}14\tan\theta)°$ at 1·1–10·0° min^{-1}, extended 25% on each side for background measure-

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