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

A = Donor atom, B = acceptor atom.

A—H···B	A···B	A—H	H···B	A—H···B
MAHS				
O(1)—H(1)···O(1^{i})	2·4448 (5)	0·95 (3)	1·507 (27)	169·5 (34)
N—H(4)···O(2^{ii})	2·8670 (5)	0·92 (1)	1·968 (9)	164·2 (8)
N—H(5)···O(W^{iii})	2·7688 (9)	0·86 (1)	1·913 (13)	174·7 (14)
O(W)—H(W)···O(2)	2·8000 (5)	0·88 (1)	1·956 (12)	163·2 (11)
DIMAHS				
O(1)—H(1)···O(1^{iv})	2·4395 (7)	0·85 (2)	1·670 (22)	150·0 (26)
N—H(3)···O(2)	2·7747 (7)	0·88 (1)	1·911 (12)	171·5 (11)

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

Table 5. *Bond lengths* (Å) *and angles* (°)

	MAHS	DIMAHS
Anion		
C(1)—O(1)	1·2866 (6)	1·2884 (8)
C(1)—O(2)	1·2443 (6)	1·2391 (8)
C(1)—C(2)	1·5130 (6)	1·5137 (8)
C(2)—C(2^{i})	1·5159 (6)	1·5132 (8)
O(1)—C(1)—O(2)	123·45 (4)	124·47 (6)
O(1)—C(1)—C(2)	114·45 (4)	113·98 (5)
O(2)—C(1)—C(2)	122·11 (4)	121·54 (5)
C(1)—C(2)—C(2^{i})	114·20 (4)	114·20 (5)
Cation		
N—C(3)	1·4837 (10)	1·4841 (10)
C(3)—N—C(3^{ii})		112·99 (7)

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

compared to 1·2391 (8) Å]. It is likely that this difference is due to the fact that O(2) is involved in two hydrogen bonds in MAHS and only one in DIMAHS. As commonly observed the average C—O distance of each carboxyl group is nearly the same in MAHS and DIMAHS: 1·2655 and 1·2638 Å, respectively, from Table 5.

Both cations have also been investigated structurally as part of the charge-density studies of hydrogen oxalate ions performed by Thomas (1975) and Thomas & Pramatus (1975). The N—C bond lengths found in the present structure determinations are slightly larger, 1·484 (1) Å, than in the room-temperature studies of the hydrogen oxalate salts, 1·463 (3) Å (methylammonium) and 1·471 (2) and 1·476 (2) Å (dimethylammonium). As these bond lengths have not been corrected for thermal vibration, it is possible that the apparent discrepancy may be due to the effect of thermal vibration.

We thank Flemming Hansen who assisted in the experimental crystallographic work. The Danish Natural Science Research Council supported this research by financing the X-ray diffractometer (J.nr. 11-1837 to SL), and the Carlsberg Foundation provided financial support to NK.

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Structure of 1-{5-Fluoro-4-[(5-oxo-1,3-pentadienyl)amino]-2-pyrimidinyl}pyridinium Chloride Trihydrate

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Abstract. $C_{14}H_{12}FN_4O^+.Cl^-.3H_2O$, $M_r = 360·8$, monoclinic, $P2_1/c$, $a = 7·2493$ (6), $b = 18·611$ (1), $c = 13·0710$ (6) Å, $\beta = 96·973$ (6)°, $V = 1750·5$ (2) Å^3, $Z = 4$, $D_x = 1·369$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 21·5$ cm^{-1}, $F(000) = 752$, $T = 290$ K, $R = 0·041$ for 2134 observed reflections. The aliphatic chain at N(4)

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is *cis* to N(3) and has the all-*trans* conformation. The pyrimidine and pyridinium planes make an angle of 5·66 (7)°. The water molecules and the ions are involved in a three-dimensional network of hydrogen bonds. The three water molecules play three different roles in this network.

Introduction. Pyridine-assisted phosphorylation of *O*-protected thymidine, uridine, inosine and guanosine and of the related bases hypoxanthine and guanine (all of which bear one reactive heteroaromatic lactam system) leads to the formation of the corresponding fluorescent, water-soluble monopyridinium salts (Adamiak, Biała & Skalski, 1985; Adamiak, Biała, Gdaniec, Mielewczyk & Skalski, 1986). The structure of the 6-pyridinium cation derived from guanine was described by Jaskólski, Skalski, Adamiak & Adamiak (1987). Recently, it has been observed (Skalski, Wenska, Gdaniec & Adamiak, 1991) that under analogous conditions the two lactam systems of the nucleobases thymine, uracil and xanthine form dipyridinium dications. The structure of one of them, *N*,*N*′-(2,4-pyrimidinediyl)dipyridinium, has been described as its dichloride monohydrate salt by Jaskólski, Skalski & Adamiak (1988). It appears that its analogue derived from 5-fluorouracil is too reactive to be isolated from the reaction mixture. Here we report the structure of the isolated product, 1-{5-fluoro-4-[(5-oxo-1,3-pentadienyl)amino]-2-pyrimidinyl}pyridinium chloride trihydrate.

Experimental. Deep-red crystals from water, crystal 0·4 × 0·4 × 0·5 mm, Syntex $P2_1$ diffractometer, Cu $K\alpha$ radiation, graphite monochromator. Cell parameters from least-squares treatment of setting angles of 15 reflections ($23·5 < 2\theta < 33·2°$). θ:2θ profiles measured for 2334 unique $+h+k\pm l$ reflections with $2\theta < 115°$ [max. $(\sin\theta)/\lambda = 0·547$ Å^{-1}] and with $0 \leq h \leq 7$, $0 \leq k \leq 20$, $-14 \leq l \leq 14$; profile analysis according to Lehmann & Larsen (1974). Random intensity variation (within 2·2%) of two standard reflections ($\bar{1}53$, $\bar{1}\bar{1}4$) measured every 1·5 h. No absorption correction. 2134 observed reflections with $I \geq 2\sigma(I)$. Structure solved by the automatic direct-methods program included in *SHELX76* (Sheldrick, 1976), C—H hydrogen atoms generated geometrically, O(N)—H hydrogen atoms located from a difference Fourier map. Full-matrix least-squares refinement, function minimized $\sum w(|F_o| - |F_c|)^2$, $w = 1/[\sigma^2(F_o) + 0·0001F_o^2]$; anisotropic non-H atoms, isotropic H atoms, soft constraints imposed on the geometry of the water molecules and on the amino N—H to prevent the H atoms from drifting towards their heavy atoms [O—H 0·97 (5), H···H 1·53 (10), N—H 1·05 (5) Å]; empirical isotropic extinction parameter x defined as $F_c' = F_c(1 - xF_c^2/\sin\theta)$ converged at $x = 1·06\,(7) \times 10^{-6}$. Final $R = 0·041$, $wR = 0·059$, $S = 3·91$, $(\Delta/\sigma)_{\max} = 0·13$, residual density in final ΔF map between $-0·24$ and $0·20$ e Å^{-3}. Computer programs: local programs (Jaskólski, 1982), *SHELX76* (Sheldrick, 1976) and *PARST* (Nardelli, 1983); molecular illustrations drawn using *ORTEP* (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Atomic coordinates are listed in Table 1.* The geometry of the cation is characterized in Table 2 and its thermal-ellipsoid representation is shown in Fig. 1.

The dimensions of the pyridinium ring agree, within e.s.d.'s, with those found in other crystallographic studies (Jaskólski, Skalski, Adamiak & Adamiak, 1987; Jaskólski, Skalski & Adamiak, 1988; Surma, Adamiak, Gawron & Skalski, 1991). In the C(2) region, the geometry of the pyrimidine ring is similar to that reported for an *N*,*N*′-(2,4-pyrimidinediyl)dipyridinium dication (Jaskólski, Skalski & Adamiak, 1988). The geometry at C(4) reflects the presence of a neutral alkylamino substituent. Compared with a 4-(*N*-pyridinium)-substituted system (Jaskólski, Skalski & Adamiak, 1988), the present pyrimidine ring has the N(3)—C(4)—C(5) angle 4·5 (3)° narrower and the endocyclic N(3)—C(4) and C(4)—C(5) bonds slightly longer [0·014 (4) and 0·017 (4) Å, respectively]. The exocyclic C(4)—N(4) bond is 0·097 (4) Å shorter than in 4-(*N*-pyridinium)pyrimidine. The length of the C(5)—F(5) bond [1·359 (3) Å] is typical for 5-fluoropyrimidines (*e.g.* Ferguson, Scrimgeour, Low & Tollin, 1986). The endocyclic C(4)—C(5)—C(6) angle is 4·9 (3)° wider and the C(5)—C(6) bond 0·0254 (4) Å shorter than in the *N*,*N*′-(2,4-pyrimidinediyl)dipyridinium dication (Jaskólski, Skalski & Adamiak, 1988). Both these changes can be attributed to the electronic effect of the F substituent at C(5). The aliphatic chain at N(4) is disposed *cis* with respect to N(3), away from F(5), and has the extended all-*trans* conformation. The pattern of C—C bond distances along the aliphatic chain is consistent with a system of conjugated double bonds (Table 2). The two aromatic rings are planar with only slight deviations from strict planarity (χ^2 for pyrimidine and pyridinium planes 66·0 and 68·8, respectively). As expected, they are nearly coplanar, the dihedral angle between their best planes being 5·66 (7)°. This twist angle is similar to the corresponding values found in the *N*,*N*′-(2,4-pyrimidinediyl)dipyridinium dication

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53521 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cl(1)	0·41026 (9)	0·38201 (3)	0·09062 (5)	0·0737 (3)
N(1)	0·5953 (3)	0·0822 (1)	0·2912 (1)	0·0592 (7)
C(2)	0·5795 (3)	0·1465 (1)	0·2515 (2)	0·0473 (7)
N(3)	0·5046 (2)	0·16846 (9)	0·1598 (1)	0·0462 (6)
C(4)	0·4270 (3)	0·1169 (1)	0·0972 (2)	0·0461 (7)
C(5)	0·4362 (3)	0·0462 (1)	0·1322 (2)	0·0548 (8)
F(5)	0·3561 (2)	−0·00590 (7)	0·0687 (1)	0·0751 (6)
C(6)	0·5204 (4)	0·0300 (1)	0·2271 (2)	0·0650 (9)
N(2)	0·6567 (2)	0·2042 (1)	0·3200 (1)	0·0489 (6)
C(22)	0·7218 (3)	0·1885 (2)	0·4186 (2)	0·0631 (9)
C(23)	0·7942 (4)	0·2423 (2)	0·4827 (2)	0·075 (1)
C(24)	0·8028 (4)	0·3110 (2)	0·4477 (2)	0·074 (1)
C(25)	0·7332 (4)	0·3263 (2)	0·3471 (2)	0·069 (1)
C(26)	0·6589 (3)	0·2727 (1)	0·2841 (2)	0·0565 (8)
N(4)	0·3408 (3)	0·13519 (9)	0·0030 (1)	0·0505 (6)
C(42)	0·3106 (3)	0·2056 (1)	−0·0289 (2)	0·0466 (7)
C(43)	0·2320 (3)	0·2229 (1)	−0·1235 (2)	0·0512 (8)
C(44)	0·1856 (3)	0·2949 (1)	−0·1526 (2)	0·0485 (7)
C(45)	0·0990 (3)	0·3142 (1)	−0·2456 (2)	0·0517 (7)
C(46)	0·0503 (3)	0·3874 (1)	−0·2697 (2)	0·0552 (8)
O(46)	−0·0316 (3)	0·40700 (9)	−0·3523 (1)	0·0763 (7)
O(1*W*)	0·1123 (3)	0·4477 (1)	0·3656 (2)	0·0895 (9)
O(2*W*)	0·7489 (3)	0·0152 (2)	0·5643 (2)	0·106 (1)
O(3*W*)	0·0165 (4)	0·1069 (2)	0·6666 (2)	0·103 (1)

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

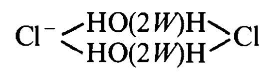
N(1)—C(2)	1·305 (3)	C(25)—C(26)	1·364 (4)
C(2)—N(3)	1·320 (3)	C(26)—N(2)	1·358 (3)
N(3)—C(4)	1·340 (3)	C(4)—N(4)	1·356 (3)
C(4)—C(5)	1·391 (3)	N(4)—C(42)	1·384 (3)
C(5)—C(6)	1·349 (3)	C(42)—C(43)	1·338 (3)
C(6)—N(1)	1·352 (3)	C(43)—C(44)	1·420 (3)
C(2)—N(2)	1·465 (3)	C(44)—C(45)	1·348 (3)
N(2)—C(22)	1·350 (3)	C(45)—C(46)	1·434 (3)
C(22)—C(23)	1·368 (4)	C(46)—O(46)	1·222 (3)
C(23)—C(24)	1·362 (5)	C(5)—F(5)	1·359 (3)
C(24)—C(25)	1·379 (4)		

C(6)—N(1)—C(2)	113·8 (2)	C(23)—C(24)—C(25)	119·2 (3)
N(1)—C(2)—N(3)	130·7 (2)	C(24)—C(25)—C(26)	119·8 (3)
C(2)—N(3)—C(4)	115·4 (2)	C(25)—C(26)—N(2)	119·9 (2)
N(3)—C(4)—C(5)	118·5 (2)	N(3)—C(4)—N(4)	119·2 (2)
C(4)—C(5)—C(6)	120·7 (2)	C(5)—C(4)—N(4)	122·4 (2)
C(5)—C(6)—N(1)	120·8 (2)	C(4)—N(4)—C(42)	123·4 (2)
N(1)—C(2)—N(2)	115·0 (2)	N(4)—C(42)—C(43)	122·7 (2)
N(3)—C(2)—N(2)	114·4 (2)	C(42)—C(43)—C(44)	122·4 (2)
C(2)—N(2)—C(22)	119·2 (2)	C(43)—C(44)—C(45)	124·1 (2)
C(2)—N(2)—C(26)	119·8 (2)	C(44)—C(45)—C(46)	121·8 (2)
C(26)—N(2)—C(22)	121·0 (2)	C(45)—C(46)—O(46)	123·9 (2)
N(2)—C(22)—C(23)	119·4 (2)	C(4)—C(5)—F(5)	118·3 (2)
C(22)—C(23)—C(24)	120·7 (3)	C(6)—C(5)—F(5)	120·9 (2)

N(1)—C(2)—N(2)—C(22)	−5·5 (3)	C(4)—N(4)—C(42)—C(43)	177·3 (2)
N(3)—C(2)—N(2)—C(22)	174·2 (2)	N(4)—C(42)—C(43)—C(44)	173·9 (2)
N(1)—C(2)—N(2)—C(26)	175·6 (2)	C(42)—C(43)—C(44)—C(45)	−177·0 (2)
N(3)—C(2)—N(2)—C(26)	−4·7 (3)	C(43)—C(44)—C(45)—C(46)	178·0 (2)
N(3)—C(4)—N(4)—C(42)	−7·1 (3)	C(44)—C(45)—C(46)—O(46)	−178·4 (2)
C(5)—C(4)—N(4)—C(42)	172·2 (2)		

[6·2 (3) and 6·1 (3)°] (Jaskólski, Skalski & Adamiak, 1988). Another way to describe coplanarity in a polycyclic system is through the use of torsion angles. Applying an algorithm similar to that of Winkler & Dunitz (1971) one can determine the twist (τ) between the connected triatomic groups as well as the nonplanarity (pyramidalization, χ) of the three bonds around each of the connected atoms. In the present case $\tau_{\rm C(2)—N(2)} = 5{\cdot}1\ (2)°$, $\chi_{\rm C(2)} = 0{\cdot}3\ (2)$, $\chi_{\rm N(2)} = 1{\cdot}1\ (2)°$.

The ions and the water molecules are involved in a network of hydrogen bonds (Table 3). Water 2 and the Cl$^-$ ion form centrosymmetric hydrogen-bonded clusters,



$$\mathrm{Cl}^-\begin{matrix}\diagup\mathrm{HO(2}W\mathrm{)H}\diagdown\\ \diagdown\mathrm{HO(2}W\mathrm{)H}\diagup\end{matrix}\mathrm{Cl}^-$$

(at 0·5,0,0·5 in Fig. 2). Within these clusters, two out of three hydrogen-bond-acceptor functions of the Cl$^-$ anion are saturated. The two hydrogen-bond-donor functions of water 2 are also utilized here. The cation is hydrogen bonded exclusively with water 1, which donates its H(2*W*1) hydrogen to O(46) from one cation and accepts H(4) from a screw-axis-related cation leading to infinite chains running along **b**. Water 3 connects these chains with the (water 2)/Cl$^-$ clusters. Water 3 donates one hydrogen bond to O(2*W*) in one (water 2)/Cl$^-$ cluster and another hydrogen bond to Cl$^-$ in an **a**-translated cluster. At the same time, it accepts a hydrogen bond from water 1. In this way, a truly three-dimensional network of hydrogen bonds is created in which all available proton donors are engaged. The O atoms accept one hydrogen bond each, and Cl$^-$ is a threefold acceptor. In contrast to the situation in crystals of *N*,*N*′-(2,4-pyrimidinediyl)dipyridinium dichloride monohydrate (Jaskólski, Skalski & Adamiak, 1988), the C—H groups show very little activity as hydrogen-bond donors [except for a weak C(24)—H···O(46) interaction]. This is due to competition from the O—H donors in these highly hydrated crystals.

All cations in the unit cell have their aromatic rings roughly coplanar and parallel to the (10$\bar{1}$) lattice planes. The aromatic rings in the crystal do not show any noticeable overlap. Also, there is no overlap between F(5) and the pyrimidine system but

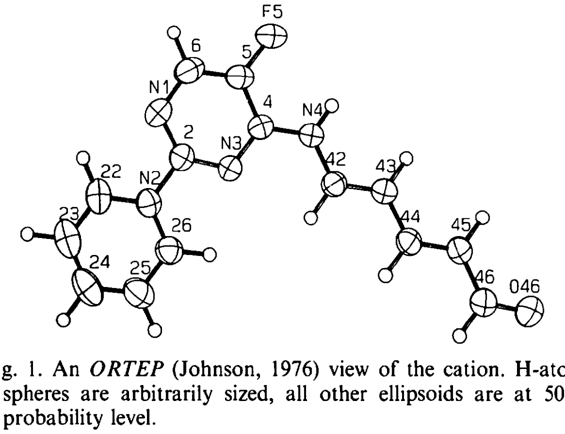


Fig. 1. An *ORTEP* (Johnson, 1976) view of the cation. H-atom spheres are arbitrarily sized, all other ellipsoids are at 50% probability level.

Table 3. *Geometry of the hydrogen bonds* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(4)—H(4)···O(1W^{i})	0·93 (2)	1·88 (3)	2·761 (3)	158 (2)
O(1*W*)—H(1*W*1)···O(3W^{i})	0·93 (3)	1·87 (3)	2·800 (3)	175 (3)
O(1*W*)—H(2*W*1)···O(46^{ii})	0·91 (3)	1·87 (3)	2·768 (3)	169 (2)
O(2*W*)—H(1*W*2)···Cl(1^{iii})	0·89 (4)	2·27 (4)	3·163 (3)	174 (4)
O(2*W*)—H(2*W*2)···Cl(1^{iv})	0·99 (4)	2·33 (4)	3·318 (3)	174 (4)
O(3*W*)—H(1*W*3)···O(2W^{v})	0·96 (4)	1·88 (4)	2·798 (4)	159 (3)
O(3*W*)—H(2*W*3)···Cl(1^{iii})	0·94 (4)	2·22 (4)	3·141 (3)	170 (4)

Symmetry code (i) x, $0·5-y$, $z-0·5$; (ii) $-x$, $1-y$, $-z$; (iii) x, $0·5-y$, $0·5+z$; (iv) $1-x$, $y-0·5$, $0·5-z$; (v) $x-1$, y, z.

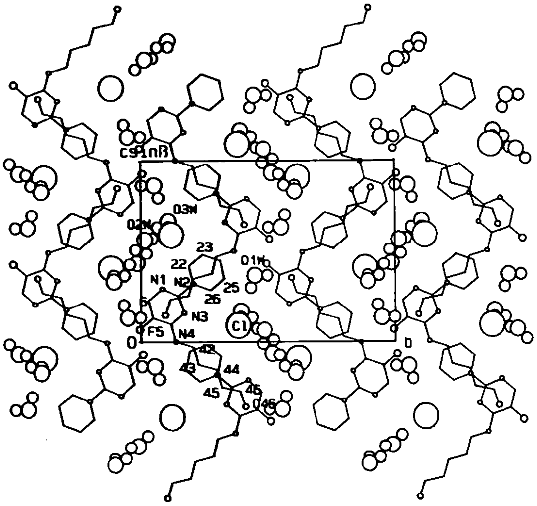


Fig. 2. Crystal packing viewed along **a**. The water molecules are represented as van der Waals space-filling models, and the Cl^- ions as spheres.

the F substituents from two inversion-related cations form a close contact of 2·923 (2) Å (Fig. 2).

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Structure of (−)-Narcotine Hemiacetal

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Abstract. 1,3-Dihydro-6,7-dimethoxy-3-(5,6,7,8-tetrahydro-4-methoxy-6-methyl-1,3-dioxolo[4,5-*g*]-isoquinolin-5-yl)-1-isobenzofuranol, $C_{22}H_{25}NO_7$, $M_r = 415·4$, orthorhombic, $P2_12_12_1$, $a = 12·302$ (2), $b = 8·022$ (1), $c = 20·929$ (3) Å, $V = 2065·4$ (5) Å^3, $Z = 4$, $D_m = 1·29$, $D_x = 1·34$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 7·91$ cm^{-1}, $F(000) = 880$, room temperature, $R = 0·039$ for 1496 observed reflections. The heterocyclic isoquinoline ring exhibits a half-chair conformation and the two five-membered rings

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