Verdier & García-Blanco, 1978; Smith-Verdier, Florencio & García-Blanco, 1979; Drew, Mok, Ang & Tan, 1987; Gallucci, Mathur & Shechter, 1992). The dissymmetric situation in the present compound is to be imputed to the fact that only the N(1)—C(2)═O(2) fragment forms infinite chains of hydrogen-bonded amide groups, the mutual relationships between hydrogen-bond strengthening and π-bond delocalization in conjugated systems having been already discussed in detail in previous papers (Gilli, Bellucci, Ferretti & Bertolasi, 1989; Gilli & Bertolasi, 1990).

The authors are indebted to the chemical company FIS SpA (Alte di Montecchio Maggiore, Italy) for supplying the crystals of nitrofurantoin.

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Structure of *N*-Hexadecylpyridinium Chloride Monohydrate

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(*Received* 6 *May* 1992; *accepted* 3 *November* 1992)

Abstract. $C_{21}H_{38}N^+.Cl^-.H_2O$, $M_r = 358.01$, triclinic, $P\bar{1}$, $a = 7.426\,(1)$, $b = 28.301\,(3)$, $c = 5.227\,(1)$ Å, $\alpha = 92.58\,(1)$, $\beta = 99.18\,(1)$, $\gamma = 92.56\,(1)°$, $V = 1081.8\,(3)$ Å^3, $Z = 2$, $D_x = 1.099$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 16.05$ cm^{-1}, $F(000) = 396$, $T = 158\,(1)$ K, $R = 0.065$, $wR = 0.073$ for 1907 observed reflections. The aliphatic chain is fully extended and inclined to the b axis by 23°. The molecules are packed in a bilayer parallel to the (001) plane. The Cl anion and water of hydration are hydrogen bonded to each other and occupy the interstitial spaces between the hexadecylpyridinium cations. The bond lengths and angles are as expected when the contraction resulting from low-temperature data collection is taken into consideration.

Introduction. The analysis of the title compound was prompted by the very recent understanding that cationic surfactants, in contrast to anionic surfactants, especially of the type of *N*-hexadecylpyridinium chloride (cetylpyridinium-Cl$^-$), exhibit antiviral and antifungal properties at moderate acid pH 6.5 (Paradies & Schröer, 1988; Paradies, 1989, 1991). This work is part of an ongoing study of antiviral biosurfactants with regard to crystal properties and molecular packing of aromatic quaternized N atoms having various alkyl chain lengths, biodegradation properties and different sizes and shapes, when forming micelles in aqueous solutions (Paradies, 1991; Clancy, Tanner, Thies & Paradies, 1992). Counterion dependency of micelle size, structure and shape (Anacker & Ghose, 1968; Anacker, 1958) is also important. In addition, the present

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Table 1. *Experimental conditions and structure-refinement parameters for* CPCl.H_2O *at* 158 (1) K

Scan type	ω–2θ
Scan rate in ω (° min^{-1})	32.0
Scan width (°)	$1.26 + 0.30\tan\theta$
$2\theta_{max}$ (°)	120.1
Range h	$-8 \rightarrow 0$
k	$-31 \rightarrow 31$
l	$-5 \rightarrow 5$
No. of reflections measured	3507
No. of unique reflections	3221
R_{int}	0.047
Corrections:	
Lorentz–polarization	Yes
Absorption, transmission factors	0.75–1.16
Secondary extinction coefficient	0.39810×10^{6}
H atoms included in calculated positions: C—H (Å)	0.95
Function minimized in full-matrix least-squares refinement	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Least-squares weights w	$4F_o^2/\sigma^2(F_o)^2$
p-Factor weighting intense reflections	0.03
Anamolous-dispersion correction	All non-H atoms
No. of observations [$I > 3.00\sigma(I)$]	1907
No. of variables	218
Reflection:parameter ratio	8.75
Residuals R, wR	0.065, 0.073
Goodness-of-fit indicator S	1.77
Maximum shift/e.s.d. in final cycle	0.01
Maximum peak in final difference map (e $Å^{-3}$)	0.45
Minimum peak in final difference map (e $Å^{3}$)	−0.35

analysis should yield information about counterion location, hydration and alkyl-chain packing with respect to recent reports on the structures of dodecyldimethylpropylammonium bromide monohydrate and hemihydrate (Taga, Machida, Kimura, Hayashi, Umemura & Takenaka, 1986, 1987) as well as hexadecyltrimethylammonium bromide (Campanelli & Scaramuzza, 1986) and of *N*-*n*-butylpyridinium chloride (Ward, Rhinebarger & Popov, 1986).

Experimental. The title compound (CPCl.H_2O) was prepared by refluxing ultra-pure grade pyridine with redistilled 1-chlorohexadecane for 6 h. The crude product obtained on cooling was recrystallized twice from concentrated aqueous NaCl solutions, and subsequently from water–acetone mixtures until CPCl.H_2O showed a constant melting point of 394.6 K, a clearing point of 436 K and a transition temperature of 358–393 K (Jeffrey, 1989). The chemical analysis revealed the composition of the recrystallized material as $C_{21}H_{38}ClN.H_2O$. Colourless prism-shaped crystals were obtained from water–acetone (40/60 *w*/*w*) mixtures, or from water–ethyl acetate (50/50 *w*/*w*) at 293 K.

A crystal of dimensions 0.15 × 0.20 × 0.150 mm was selected for data collection on a Ragaku AFC-5*R* diffractometer with graphite-monochromated Cu $K\alpha$ radiation. Lattice parameters and orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 25 centred reflections with $19.00 < 2\theta < 32.00°$, correspond to a triclinic unit cell. Data-collection details are given in Table 1. ω scans of several intense reflections, made prior to data collection, had an average width at half height of 0.27° with a take-off angle of 6.0°. The weak reflections [$I < 10.0\sigma(I)$] were rescanned (maximum of two rescans) and the counts were accumulated to assure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. Data reduction, structure solution and refinement are summarized in Table 1. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). Complex neutral-atom scattering factors were taken from Cromer & Waber (1974). All computations were performed using the *TEXSAN* (Molecular Structure Corporation, 1985) crystallographic software package.

Discussion. The final atomic parameters are listed in Table 2.* Bond distances and bond angles are given in Table 3. The numbering scheme used is illustrated in Fig. 1. The packing diagram, Fig. 2, reveals that the hexadecyl chains have an extended *trans*-zigzag form. The bond distances and C—C—C angles of the *n*-alkyl chain are similar to those found for *n*-octadecane by Nyburg & Lüth (1972). In addition, the results obtained show a slight shortening of the C—C bonds and a widening of the C—C—C angles compared to pentane (Mathisen, Norman & Pedersen, 1967), which arises from the librational motion of the C atoms in planes normal to the C_{16}-chain axis. This is readily seen in Fig. 1, which reveals a small increase in the size of the thermal ellipsoids in the C_{16} chain as one moves away from the centre of the molecule. This has the known effect of bringing the mean positions of the C atoms inwards towards the chain axis by shortening the C—C bonds while widening the C—C—C angles. The shortest nonbonded contacts are all H···H. A striking feature of this structure is the tilt angle of the pyridine ring with respect to the C_{16} chain of 102.4°, which is similar to the value, determined by X-ray reflectivity experiments, of 103°, when CPCl is attached to silica surfaces (Paradies, 1991).

The cation moieties are arranged in an antiparallel fashion giving rise to interdigitation within the bilayer. This represents a low charge density on its polar surfaces owing to the large bulk of the pyridinium group.

Atom Cl(1) is hydrogen bonded to the water molecule (Table 4). Atom Cl(1) also comes into close contact with atoms H(2) and H(3) of the pyridine

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* Lists of H-atom parameters, anisotropic thermal parameters, structure factors, intermolecular distances involving H atoms and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55769 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1012]

Table 2. *Fractional coordinates and equivalent isotropic thermal parameters* (Å²)

$$B_{eq} = (3/4)\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl(1)	−0.7016 (2)	0.10531 (5)	−1.0969 (3)	2.34 (6)
O(1)	−0.9402 (5)	0.1017 (1)	−0.6537 (7)	3.0 (2)
N(1)	−0.2636 (6)	0.0943 (1)	−0.2897 (8)	1.7 (2)
C(1)	−0.3740 (7)	0.0901 (2)	−0.521 (1)	1.9 (2)
C(2)	−0.3864 (7)	0.0487 (2)	−0.670 (1)	2.2 (2)
C(3)	−0.2866 (8)	0.0118 (2)	−0.581 (1)	2.2 (2)
C(4)	−0.1768 (7)	0.0163 (2)	−0.345 (1)	2.1 (2)
C(5)	−0.1672 (7)	0.0585 (2)	−0.202 (1)	2.1 (2)
C(6)	−0.2532 (7)	0.1398 (2)	−0.132 (1)	2.2 (2)
C(7)	−0.1355 (7)	0.1784 (2)	−0.230 (1)	2.1 (2)
C(8)	−0.1342 (7)	0.2233 (2)	−0.059 (1)	2.2 (2)
C(9)	−0.0143 (8)	0.2645 (2)	−0.131 (1)	2.3 (2)
C(10)	−0.0207 (7)	0.3088 (2)	0.042 (1)	2.2 (2)
C(11)	0.0970 (8)	0.3507 (2)	−0.020 (1)	2.2 (2)
C(12)	0.0878 (7)	0.3945 (2)	0.156 (1)	2.2 (2)
C(13)	0.2055 (8)	0.4371 (2)	0.099 (1)	2.1 (2)
C(14)	0.1959 (8)	0.4803 (2)	0.277 (1)	2.3 (2)
C(15)	0.3116 (8)	0.5229 (2)	0.219 (1)	2.3 (2)
C(16)	0.3028 (8)	0.5664 (2)	0.397 (1)	2.2 (2)
C(17)	0.4192 (8)	0.6089 (2)	0.340 (1)	2.2 (2)
C(18)	0.4104 (8)	0.6523 (2)	0.516 (1)	2.2 (2)
C(19)	0.5274 (8)	0.6949 (2)	0.459 (1)	2.4 (2)
C(20)	0.5186 (8)	0.7383 (2)	0.635 (1)	2.7 (3)
C(21)	0.6372 (9)	0.7809 (2)	0.580 (1)	3.2 (3)

Table 3. *Bond lengths* (Å) *and angles* (°)

N(1)—C(5)	1.322 (6)	C(5)—N(1)—C(1)	120.7 (5)
N(1)—C(1)	1.344 (6)	C(5)—N(1)—C(6)	120.4 (4)
N(1)—C(6)	1.490 (6)	C(1)—N(1)—C(6)	118.9 (4)
C(1)—C(2)	1.370 (7)	N(1)—C(1)—C(2)	120.2 (5)
C(2)—C(3)	1.364 (7)	C(3)—C(2)—C(1)	119.3 (5)
C(3)—C(4)	1.363 (7)	C(2)—C(3)—C(4)	119.9 (5)
C(4)—C(5)	1.372 (7)	C(3)—C(4)—C(5)	118.9 (5)
C(6)—C(7)	1.525 (7)	N(1)—C(5)—C(4)	121.0 (5)
C(7)—C(8)	1.518 (7)	N(1)—C(6)—C(7)	112.7 (4)
C(8)—C(9)	1.530 (7)	C(8)—C(7)—C(6)	109.0 (4)
C(9)—C(10)	1.517 (7)	C(7)—C(8)—C(9)	114.3 (4)
C(10)—C(11)	1.519 (8)	C(10)—C(9)—C(8)	112.2 (4)
C(11)—C(12)	1.521 (8)	C(9)—C(10)—C(11)	114.3 (4)
C(12)—C(13)	1.525 (7)	C(12)—C(11)—C(10)	113.2 (4)
C(13)—C(14)	1.514 (8)	C(11)—C(12)—C(13)	114.3 (4)
C(14)—C(15)	1.518 (7)	C(14)—C(13)—C(12)	113.6 (4)
C(15)—C(16)	1.520 (7)	C(13)—C(14)—C(15)	113.6 (4)
C(16)—C(17)	1.518 (7)	C(14)—C(15)—C(16)	113.9 (4)
C(17)—C(18)	1.510 (7)	C(17)—C(16)—C(15)	114.1 (4)
C(18)—C(19)	1.522 (7)	C(18)—C(17)—C(16)	114.2 (4)
C(19)—C(20)	1.511 (8)	C(17)—C(18)—C(19)	114.2 (4)
C(20)—C(21)	1.527 (8)	C(20)—C(19)—C(18)	114.1 (5)
		C(19)—C(20)—C(21)	114.1 (5)

Table 4. *Special contacts involving* Cl *anions* (< 3.2 Å) *and hydrogen-bond geometry* (Å, °)

		Symmetry			Symmetry
Cl(1)···H(40)	2.199	(i)	Cl(1)···H(38)	3.149	(iv)
Cl(1)···H(39)	2.265	(ii)	Cl(1)···H(5)	3.150	(iii)
Cl(1)···H(3)	2.699	(v)	Cl(1)···H(8)	3.000	(iii)
Cl(1)···H(6)	2.744	(ii)	Cl(1)···H(1)	3.045	(i)
Cl(1)···H(2)	2.746	(i)	Cl(1)···O(1)	3.152 (4)	(ii)
Cl(1)···O(1)	3.136 (4)	(i)			

O—H···Cl	O—H	H···Cl	O···Cl	O—H···Cl
O(1^{ii})—H(39^{ii})···Cl(1)	0.99	2.26	3.152 (4)	148
O(1)—H(40)···Cl(1)	0.95	2.20	3.136 (4)	171

Symmetry code: (i) x, y, z; (ii) $x, y, z-1$; (iii) $x-1, y, z-1$; (iv) $-x, 1-y, -z$; (v) $1-x, -y, 2-z$.

ring (2.70 and 2.75 Å) and with atom H(6) of the first methylene group of the alkyl chain (2.74 Å). The hydrogen-bonding network consists of chains of alternating water molecules and Cl anions running along the *c* axis to form a one-dimensional network. There is a very short H···H contact between H(5) on the pyridine ring and H(4) of the water molecule (2.15 Å), while the distance O(1)···H(5) is 2.35 Å.

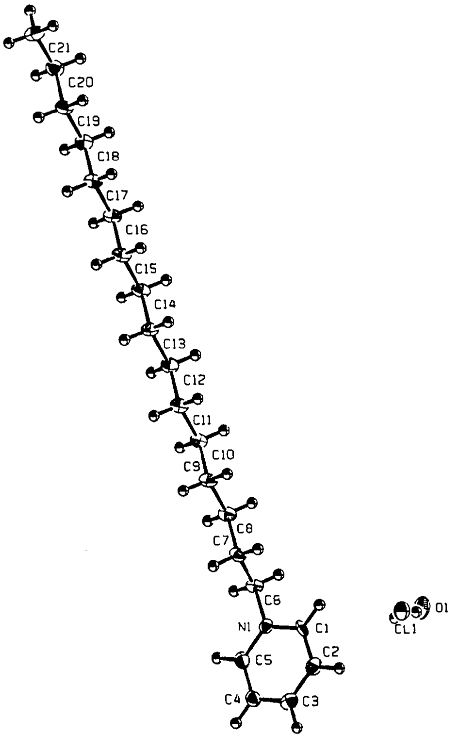

Fig. 1. Perspective view of the CPCl.H$_2$O compound with the atomic numbering scheme.

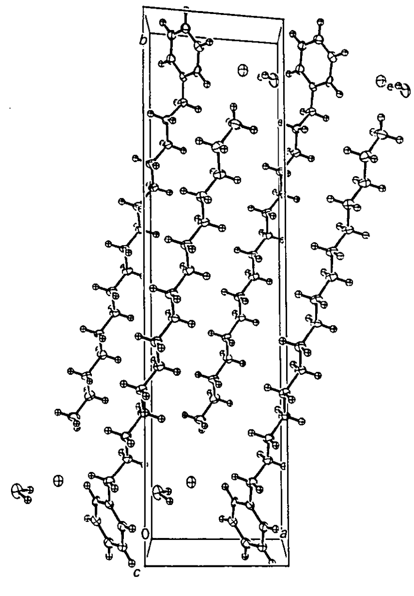

Fig. 2. Packing of molecules of the CPCl.H$_2$O compound on the *bc* plane.

This work has been supported by a grant from the BMFT (Bonn, Germany). We thank Mrs Kirsten Huschke for typing the manuscript.

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Acta Cryst. (1993). C**49**, 747–749

Structure of 5-Benzoyl-1-[4-(dimethylamino)phenylmethyleneamino]-4-phenyl-1*H*-pyrimidin-2-one

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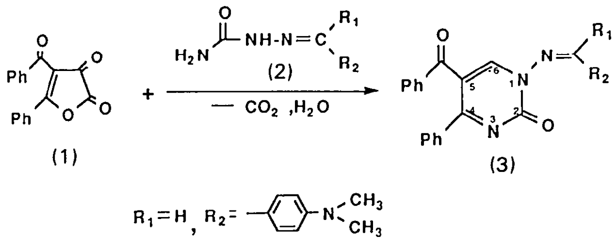
(*Received* 10 *January* 1992; *accepted* 20 *July* 1992)

Abstract. $C_{26}H_{22}N_4O_2$, $M_r = 422.485$, monoclinic, $P2_1/c$, $a = 9.564\,(2)$, $b = 11.305\,(1)$, $c = 20.389\,(3)$ Å, $\beta = 91.27\,(1)°$, $V = 2203.95$ Å^3, $Z = 4$, $D_x = 1.273$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.54184$ Å, $\mu = 6.3$ cm^{-1}, $F(000) = 888$, $T = 295$ K, $R = 0.050$ for 2816 observed reflections. The pyrimidine ring is significantly non-planar, the angles between the planes of atoms N1, C6, C5 and C2, N3, C4 (using standard numbering for pyrimidines) is 7.5°. In 1-amino-5-benzoyl-4-phenyl-1*H*-pyrimidine-2-thione, the corresponding angle is 0.74°.

Introduction. The pyrimidines are effective in antibacterial and insecticidal action (Cheng, 1969; McNair-Scott, Ulbricht, Rogers, Chu & Rose, 1959; Sankyo Co. Ltd & Ube Industries Ltd, 1984; Ziegler, Eder, Belegratis & Prewedorakis, 1967; Akçamur, Altural, Sarıpınar, Kollenz, Kappe, Peters, Peters & von Schnering, 1988; Özbey, Kendi, Akçamur, Yıldırım, Elerman & Soylu, 1991).

Experimental. An equimolar mixture of 4-benzoyl-5-phenyl-2,3-furandione and 4-(dimethylamino)benzaldehyde semicarbazone (2) was heated at 388 K for 15 min. The mixture was cooled to room temperature, and the product (3) extracted with ether (Sarıpınar, 1988). Yellow parallelepiped transparent



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0108-2701/93/040747-03$06.00