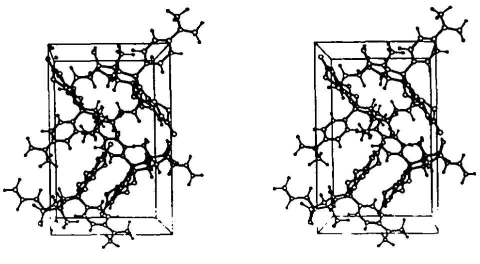

Fig. 3. Stereo packing diagram viewed down **b**.

The above observations lead to the conclusion that, like other $\Delta^{4,9}$ steroids, RU 38 486 and ORG 30761 are flexible molecules and that the angular dimethylaminophenyl group has a fixed position when the steroid molecule is in a flat conformation. It would surely be very interesting to study the flexibility, different conformations and their energy differences of these novel 11β-[4-(dimethylamino)phenyl] $\Delta^{4,9}$ steroids by NMR in solution and by computational techniques like molecular mechanics and molecular dynamics.

The molecular packing is illustrated in Fig. 3, which shows a stereoview down **b**. The steroid molecules are hydrogen-bonded from head to tail, O(17)→O[3, ($\frac{5}{2}-x$, $1-y$, $\frac{1}{2}+z$)] with O···O = 2·926 (5) Å and O—H···O = 171 (5)°. The packing scheme is similar to that of RU 38 486, the difference being the inclusion of the *n*-butyl acetate solvent molecule in the latter crystal structure. There is an unrealistic short intermolecular H···H contact of 1·70 (1) Å between H atoms at C(2*B*) and C(18), indicating that the calculated coordinates of these H atoms are not accurate owing to the only approximate coordinates of C(2*B*); other H···H contact distances are greater than 2·18 Å.

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Hexamethylenetetramine Monohydrochloride*

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Abstract. $C_6H_{13}N_4^+.Cl^-$, $M_r = 176·65$, trigonal, $R3m$, $a = 8·915$ (2), $c = 8·935$ (2) Å (hexagonal axes), $V = 615·0$ (3) Å^3, $Z = 3$, $D_m = 1·41$ (2), $D_x = 1·43$ g cm^{-3}, $F(000) = 282$, Cu $K\alpha$, $\lambda = 1·54178$ Å, $\mu = 36·44$ cm^{-1}, $T = 297$ K, $R = 0·041$ for all 126 independent reflections. The polar adduct possesses full $3m$ symmetry in the crystal. The N^+—H···Cl^- hydrogen bond is also the only close ionic interaction within the crystal.

Introduction. The title compound was prepared during an investigation of hydrated amine salts. Under the crystallization conditions used, however, no water molecules were incorporated into the closely packed

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* 3,5,7-Triaza-1-azoniatricyclo[3.3.1.1^{3,7}]decane chloride.

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and thermal parameters* ($\text{Å}^2 \times 10^3$)

$U_{eq} = \frac{1}{3}$(trace of orthogonalized U_{ij} tensor).

	x	y	z	Site symmetry	Occupancy factor	U_{eq} or U_{iso}
Cl^-	0	0	10000	$3m$	$\frac{1}{6}$	45 (1)
N(1)	0	0	6690 (10)	$3m$	$\frac{1}{6}$	31 (3)
C(2)	931 (4)	1862 (8)	6117 (8)	m	$\frac{1}{2}$	34 (3)
N(3)	914 (3)	1828 (6)	4491 (7)	m	$\frac{1}{2}$	34 (2)
C(4)	1775 (8)	888 (4)	3964 (10)	m	$\frac{1}{2}$	37 (2)
H(1)	0	0	7821	$3m$	$\frac{1}{6}$	37
H(2)	2252	2526	6514	1	1	41
H(4)	1774	887	2755	m	$\frac{1}{2}$	43
H(5)	3094	1547	4366	m	$\frac{1}{2}$	43

Table 2. *Bond distances* (Å) *and angles* (°)

N(1)—C(2)	1·526 (7)	C(2)—N(1)—C(2′)	109·3 (4)
C(2)—N(3)	1·453 (10)	N(1)—C(2)—N(3)	108·6 (5)
N(3)—C(4)	1·469 (5)	C(2)—N(3)—C(4)	109·2 (4)
C—H	1·08 (fixed)	C(4)—N(3)—C(4′)	107·8 (7)
N—H	1·01 (fixed)	N(3)—C(4)—N(3′)	112·6 (5)

crystal structure of the monohydrochloride of hexamethylenetetramine (HMT).

Experimental. Slow evaporation of aqueous solutions containing HCl and HMT in 1:2 ratio yielded crystals of the title compound (Fig. 1) and of HMT itself. D_m was determined by flotation in CCl_4/hexane mixtures. Lattice parameters were obtained by least squares (25 reflections, $32 \leq 2\theta \leq 47°$). Intensity data were collected on a block-shaped crystal, dimensions $0{\cdot}35 \times 0{\cdot}38 \times 0{\cdot}40$ mm, using a Nicolet $R3m$ diffractometer with a graphite monochromator, for $\sin\theta/\lambda \leq 0{\cdot}55$ Å^{-1}, $-9 \leq h \leq 9$, $0 \leq k \leq 9$, $0 \leq l \leq 9$. 329 intensities were measured to give the 126 independent data, all with $I > 1{\cdot}5\sigma_I$, used in structure refinement. Merging $R = 0{\cdot}023$. Three standard reflections showed no appreciable intensity variation. An empirical absorption correction (Sheldrick, 1983) was applied ($T_{max} = 0{\cdot}538$, $T_{min} = 0{\cdot}384$). The space group was determined and the structure solved by analysis of the three-dimensional Patterson function and successive Fourier syntheses, followed by least-squares refinement of 24 parameters to minimize $\sum w(\Delta F)^2$, with weights w proportional to $1/[\sigma^2(F) + 0{\cdot}015F^2]$. The value 0·015 was found to give the best fit of $(\Delta F)^2$ to $1/w(F)$. H-atom positions were calculated, with C—H fixed at 1·08 Å, N—H at 1·01 Å, and $U_H = 1{\cdot}2 \times$ (U_{eq} of atom to which H is bonded). Final $R = 0{\cdot}041$, $wR = 0{\cdot}062$, $S = 0{\cdot}554$. Maximum shift/e.s.d. = 0·003, maximum and minimum heights in final difference Fourier map 0·18 and $-0{\cdot}34$ e Å^{-3}. No correction was made for secondary extinction. Atomic scattering factors for C_{val}, H_{SDS}, N, and Cl^-, including f' and f'', were taken from *International Tables for X-ray Crystallography* (1974). The *SHELXTL* program system (Sheldrick, 1983) was used for all calculations.

Discussion. Final atomic parameters are given in Table 1, bond lengths and angles in Table 2.*

The polar rhombohedral crystal structure arises from close packing of hydrogen-bonded ion pairs $HMTH^+.Cl^-$ (Fig. 1). The $N^+\cdots Cl^-$ distance between hydrogen-bonded chloride and nitrogen is 2·957 Å,

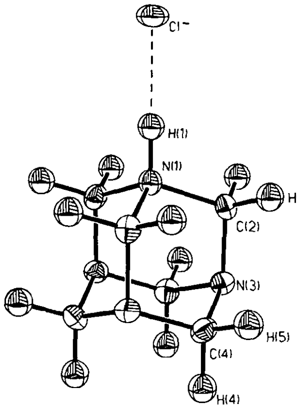

Fig. 1. Perspective view of the title compound.

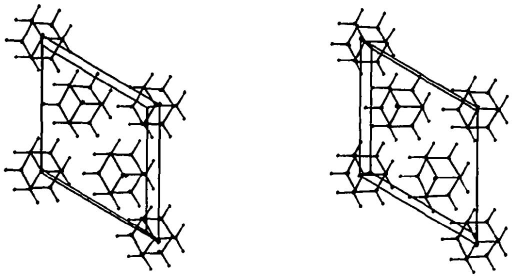

Fig. 2. Packing diagram, viewed along the *c* axis.

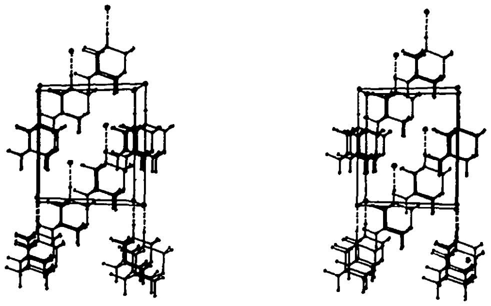

Fig. 3. Packing diagram, viewed normal to the *c* axis.

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* A list of observed and calculated structure factors and a table of anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43388 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

while the next closest distance between Cl^- and N^+ is 5·15 Å, so the only significant ionic attractions are within each ion pair.

One major packing interaction is that between hydrophobic portions of the bulky $HMTH^+$ ions (Fig. 2). The second major packing interaction is the tight surrounding of each hydrogen-bonded chloride ion by six additional non-bonded hydrogen atoms (Fig. 3). Three of these, each at a distance of 2·817 Å from Cl^-, are H(4) atoms from the ion pair translated up along **c** from the given ion pair. The three others, each 2·908 Å from Cl^-, are H(5) atoms in three separate ion pairs related to the given one by rhombohedral centering translations. These $Cl^-\cdots H$ distances are essentially equal to the sum of the ionic radius of Cl^-, 1·81 Å (Pauling, 1970), and the van der Waals radius of aliphatic H, 1·06–1·20 Å (Bondi, 1964).

This crystal is isostructural with that of HMT.HBr (Mak, Li & Yip, 1983).

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Structure of 7a-Hydroxy-4-phenyl-1,2,4,4a,5,6,7,7a-octahydrocyclopenta[*d*][1,3]thiazine-2-thione

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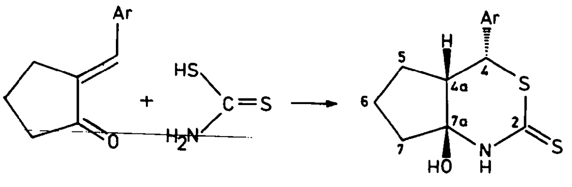
Abstract. $C_{13}H_{15}NOS_2$, $M_r = 265·4$, monoclinic, $C2/c$, $a = 30·482\ (2)$, $b = 6·989\ (1)$, $c = 12·685\ (1)$ Å, $\beta = 111·09\ (1)°$, $V = 2521·4\ (5)$ Å^3, $Z = 8$, $D_x = 1·398$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0·71073$ Å, $\mu = 0·39$ mm^{-1}, $F(000) = 1120$, $T = 293\ (1)$ K. $R = 0·050$ for 2290 unique observed reflections. Only X-ray analysis could reveal the structure of the title compound obtained by the reaction of 2-benzylidenecyclopentanone with dithiocarbamic acid. It has been shown to be a *cis*-fused diastereomer in which the 4-phenyl group is in a *trans* position to the annular H atom.

Introduction. The reactions of 2-arylidenecyclopentanones (1*a*–*c*) with dithiocarbamic acid have been investigated. In these reactions stereohomogenous 4-aryl-7a-hydroxy-1,2,4,4a,5,6,7,7a-octahydrocyclopenta[*d*][1,3]thiazine-2-thiones (2*a*–*c*) were formed. The crude-product analysis (^{1}H NMR) showed that in each case the formation of only one diastereoisomer could be observed as depicted in the scheme. However, as to which of the four possible diastereomers was furnished no spectroscopic answer could be given. Consequently, an X-ray analysis of (2*a*) (melt: 431–434 K) has been performed.



(1) (2)

Ar: (*a*) C_6H_5, (*b*) 4-CH_3–C_6H_4, (*c*) 4-CH_3O–C_6H_4

Experimental. Colourless crystals, *ca* 0·30 × 0·35 × 0·40 mm, CAD-4 diffractometer, $\omega/2\theta$ scan in the range $1·5 < \theta < 27·0°$ with scan width $0·40° + 0·35°\tan\theta$ using graphite-monochromated Mo $K\alpha$ radiation. Cell constants by least squares using 25