

part ce groupement diméthylamine dont le désordre et l'agitation thermique importante rendent imprécise la localisation de deux atomes de carbone et de l'atome d'azote, les distances interatomiques et les angles de valence sont en accord avec les grandeurs théoriques et avec les valeurs rapportées par Kilbourn & Owston (1970) pour le dérivé *cis*. Ces distances et angles sont reportés sur la Fig. 2.

Les angles observés entre les plans des cycles phényles restent également en accord avec les résultats antérieurs. Les cycles en position *trans* par rapport à la double liaison centrale font entre eux un angle de 59°, alors que l'on observe 61° pour le broparoestrol (Fornies-Marquina, Courseille, Busetta & Hospital, 1972) et un angle variant de 59 à 70° pour les formes solvatées du diéthylstilboestrol (Busetta, Courseille & Hospital, 1973). Il faut noter cette analogie entre la conformation des cycles en position *trans* du tamoxifène et de la molécule de diéthylstilboestrol 'dissymétrique à cycles croisés' (Precigoux, 1978) (Fig. 3). Les valeurs rapportées par Kilbourn & Owston (1970) pour le dérivé *cis* sont sensiblement plus élevées (67 et 73°).

Les cycles portés par le même carbone font entre eux un angle égal à 87°, plus élevé que celui observé dans le cas du broparoestrol (78°).

La cohésion cristalline est assurée uniquement par un ensemble de forces de van der Waals. Il n'existe pas de recouvrement intermoléculaire au niveau des cycles aromatiques.

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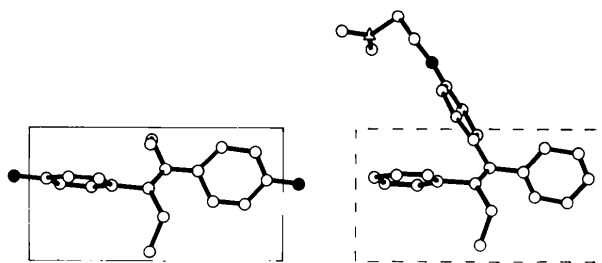


Fig. 3. Conformations des molécules de diéthylstilboestrol (dissymétrique à cycles croisés) et de tamoxifène (*trans*).

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## 5-Iodocytidine

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**Abstract.**  $C_9H_{12}IN_3O_5$ , orthorhombic,  $P2_12_12_1$ ,  $a = 8.565$  (5),  $b = 8.312$  (5),  $c = 18.027$  (10) Å,  $U = 1283.4$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.915$  Mg m<sup>-3</sup>. Diffractometer data, Mo  $K\alpha$  radiation. Final  $R = 0.103$  for 1589 unique reflexions. The molecule is in the *anti* conformation and the C(5')–O(5') bond is in the *gauche-gauche* conformation. The sugar pucker is C(3')-*endo*–C(2')-*exo* relative to the mean plane of the ring.

**Introduction.** Crystals were obtained by slow evaporation of an aqueous solution of 5-iodocytidine.

The intensities of 1592 unique reflexions were measured on a Stoe two-circle diffractometer with graphite-monochromatized Mo  $K\alpha$  radiation. Reflexions were considered unobservable if the counts were less than the background count. Two crystals mounted about *a* and *c* were used for data collection. The crystals were small and no absorption corrections were applied.

The structure was determined by the heavy-atom method. Full-matrix least-squares refinement of the positional and isotropic thermal parameters was made for 1589 reflexions. The reflexions 110, 200 and 020

appeared to suffer from extinction and were omitted. H-atom positions were calculated on the basis of the chemical structure and hydrogen-bonding scheme suggested by the interatomic distances and angles. With fixed H-atom coordinates and temperature factors, refinement of the positional parameters of the non-hydrogen atoms, the anisotropic thermal parameters of the I atom and the isotropic thermal parameters of the other atoms was continued. After refinement  $R$  was 0.103,\* and there were no shifts in

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34675 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters ( $\times 10^4$ ) and isotropic thermal parameters ( $\times 10^3$ ) of the non-hydrogen atoms with e.s.d.'s

	$x$	$y$	$z$	$U(\text{\AA}^2)$
I	9812 (2)	6916 (2)	3009 (1)	*
N(1)	7865 (25)	5847 (24)	884 (11)	22 (5)
C(2)	9058 (31)	5989 (29)	378 (13)	23 (6)
O(2)	8723 (23)	5744 (22)	-289 (10)	31 (4)
N(3)	10459 (24)	6557 (21)	606 (10)	22 (5)
C(4)	10656 (26)	6900 (31)	1349 (11)	20 (5)
N(4)	12049 (27)	7529 (26)	1521 (12)	32 (6)
C(5)	9367 (25)	6610 (24)	1856 (11)	15 (5)
C(6)	8078 (30)	6152 (29)	1621 (13)	19 (5)
C(1')	6262 (32)	5310 (30)	606 (13)	25 (6)
O(4')	5175 (22)	5600 (17)	1156 (8)	23 (4)
C(2')	6204 (31)	3515 (27)	425 (13)	22 (6)
O(2')	5135 (21)	3154 (19)	-123 (8)	30 (4)
C(3')	5809 (25)	2838 (27)	1167 (11)	15 (5)
O(3')	5295 (24)	1227 (19)	1193 (8)	30 (4)
C(4')	4546 (32)	4011 (30)	1411 (13)	27 (6)
C(5')	4317 (33)	4092 (31)	2264 (14)	27 (6)
O(5')	5656 (22)	4354 (22)	2644 (10)	32 (5)

\* Anisotropic temperature factors for I ( $\times 10^4$ )

$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
166 (10)	505 (10)	264 (7)	-44 (8)	-57 (8)	-70 (9)

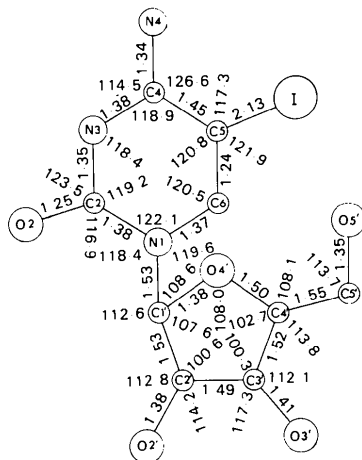


Fig. 1. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) in 5-iodocytidine. The average e.s.d. in bond lengths is 0.03  $\text{\AA}$ , and in bond angles 2.3 $^\circ$ .

parameters  $>0.01\sigma$ . Positional and thermal parameters are given in Table 1, bond lengths and angles in Fig. 1.

**Discussion.** The nucleoside is in the *anti* conformation, like the majority of pyrimidine nucleosides and nucleotides. The pyrimidine base is essentially planar (Table 2) though the attached atoms deviate appreciably from this plane. These deviations, however, are no greater than those observed in similar structures (e.g. Rahman & Wilson, 1970). The sugar ring has a C(3')-*endo*-C(2')-*exo* pucker relative to the mean plane of the ring. C(5')-O(5') is in the *gauche-gauche* orientation relative to C(4')-O(4') and C(4')-C(3'), which is the conformation most favoured in nucleosides and nucleotides, particularly when the sugar pucker is C(3')-*endo*.

Bond lengths and angles are in reasonably good agreement with those in other nucleosides and nucleo-

Table 2. Deviations ( $\text{\AA}$ ) of atoms from the least-squares planes, and torsion angles ( $^\circ$ ), with e.s.d.'s

The atoms marked with an asterisk were used to calculate the mean planes.  $X$ ,  $Y$  and  $Z$  are coordinates, in  $\text{\AA}$ , relative to  $a$ ,  $b$  and  $c$ .

(1) Pyrimidine ring

$$-2.5098X + 7.8443Y - 2.7633Z - 2.3461 = 0$$

N(1)*	0.022 (20)	C(6)*	0.004 (24)
C(2)*	-0.026 (24)	C(1')	0.080 (24)
N(3)*	0.005 (17)	O(2)	0.050 (18)
C(4)*	0.019 (26)	N(4)	0.116 (22)
C(5)*	-0.025 (20)	I	-0.215 (2)

(2) Sugar ring

$$6.6269X + 0.7059Y + 11.3177Z - 5.0894 = 0$$

O(4')*	0.044 (16)	O(2')	-1.603 (16)
C(1')*	0.121 (24)	O(3')	-0.144 (17)
C(2')*	-0.249 (23)	C(5')	0.623 (26)
C(3')*	0.281 (21)	O(5')	1.959 (18)
C(4')*	-0.197 (25)		

Torsion angles

O(4')-C(1')-N(1)-C(6)	14.3 (2.5)
O(5')-C(5')-C(4')-C(3')	52.0 (2.5)

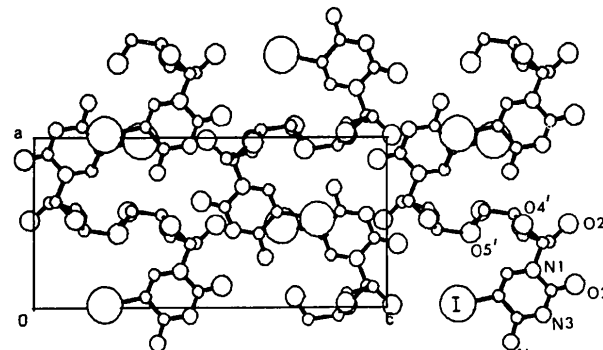


Fig. 2. A projection of the structure viewed along  $b$ .

Table 3. *Hydrogen-bond lengths (Å) and angles (°) with e.s.d.'s*

O(5')—H...O(3') <sup>a</sup>	2.74 (3)	C(5')—O(5')—O(3')	103 (2)
O(2')—H...I <sup>b</sup>	3.37 (2)	C(2')—O(2')—I	135 (2)
O(3')—H...O(2) <sup>c</sup>	2.67 (3)	C(3')—O(3')—O(2)	136 (2)
N(4)—H...O(5') <sup>a</sup>	2.90 (3)	C(4)—N(4)—O(5')	158 (2)

Symmetry code: (a)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (b)  $\frac{1}{2} - x, -y, \frac{1}{2} + z$ ; (c)  $\frac{1}{2} + x, \frac{1}{2} - y, -z$ .

tides (e.g. Voet & Rich, 1970). The molecules are linked together by hydrogen bonds (Table 3), but one of the H atoms attached to N(4) is not involved in hydrogen bonding. There appears to be a hydrogen bond between O(2') and an I atom. There is no overlap of bases but the I atom partly overlaps the base of

another molecule (Fig. 2). The shortest distance between the I atom and atoms in this base is 3.97 Å and involves C(5). The I atoms are in a zigzag arrangement about the symmetry axes parallel to **b**, with a distance of 4.55 Å between adjacent atoms.

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### 17β-Hydroxy-7β-methyl-4,14-estradien-3-one

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**Abstract.** C<sub>19</sub>H<sub>26</sub>O<sub>2</sub>, *M<sub>r</sub>* = 286.4, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 9.8287 (6), *b* = 28.303 (1), *c* = 6.0256 (5) Å (*λ* = 1.5418 Å, *T* = 291 K), *V* = 1676.2 Å<sup>3</sup>, *Z* = 4, *ρ<sub>x</sub>* = 1.14 Mg m<sup>-3</sup>. X-ray analysis of the title compound established unequivocally that the methyl group at C(7) has the β configuration.

**Introduction.** Introduction of a 7α-methyl function and a Δ<sup>14</sup> double bond into 19-nortestosterone enhances androgenic activity to 1000 times that of testosterone in the chick comb assay (Segaloff & Gabbard, 1973). The biological activity of the corresponding 7β-methyl isomer has not been investigated and in order to assess its activity this compound was synthesized (the synthesis will be described elsewhere). The crystal structure determination of this product was undertaken in order to unequivocally establish the configuration of the 7-methyl group.

Crystallographic diffraction data were measured on a specimen crystal of dimensions 0.20 × 0.20 × 0.62 mm with an Enraf–Nonius CAD-4 automated

diffractometer using Ni-filtered Cu *Kα* radiation. The lattice parameters were refined by a least-squares fit to measured 2θ values for 25 reflections in the interval 50° < 2θ < 69°. Integrated relative intensities for 2028 independent reflections with 2θ < 150° were measured as ω–2θ scans; 1643 of these reflections were measured to be observed above background (*I* > 2σ).

The intensities were reduced to structure factor amplitudes, and phase angles sufficient for location of the nonhydrogen atoms were derived using the direct-methods program *MULTAN* (Germain, Main & Woolfson, 1971) in conjunction with the negative-quartet figure of merit (DeTitta, Edmonds, Langs & Hauptman, 1975). All H atoms with the exception of H(O17) were located on a difference electron density map prepared at an intermediate stage in the least-squares refinement of the structural parameters. In the final cycles of full-matrix least-squares refinement, positional parameters for all the atoms, anisotropic thermal vibration parameters for the nonhydrogen atoms and isotropic thermal vibration parameters for