C(1)—C(2)—C(4) (126·7°) clearly reflect strong steric interaction between the *syn* methyl group of C(6) and the C(2)—C(4)—N(4) group of the molecule. This steric interaction is also reduced by the deviation of the C(2)—C(4)—N(4) moiety from linearity (175·6°). Nevertheless C(2)—C(3) and C(2)—C(4) are typical C_{sp^2}—C_{sp} bonds with bond order of $\frac{4}{3}$ (Bent, 1961).

The C—N triple-bond lengths are of the same order as the value in the corresponding $(S_2C_4N_2)^{2-}$ and demonstrate the low contribution of the cyanide groups to the π system of the whole molecule (Witt & Britton, 1971).

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Structure of 1-[3-(Imidazol-1-yl)propyl]thymine

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Abstract. $C_{11}H_{14}N_4O_2$, $M_r = 234{\cdot}3$, monoclinic, $P2_1/n$, $a = 8{\cdot}707\ (2)$, $b = 12{\cdot}786\ (4)$, $c = 10{\cdot}643\ (3)$ Å, $\beta = 102{\cdot}89\ (2)°$, $V = 1155{\cdot}0\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}59$ cm^{-1}, $F(000) = 496$, room temperature, $R = 0{\cdot}034$ for 1268 unique reflections. The extended conformation of the molecule prevents any intramolecular photocycloaddition of thymine and imidazole C=C double bonds in the crystal [the shortest distance between the midpoints of C=C and C=N double bonds is 3·549 (3) Å].

Introduction. The photochemistry of purine–pyrimidine dinucleotides and their analogues in aqueous solution has grown in interest in the last few years (Bose, Davies, Sethi & McCloskey, 1983; Bose, Kumar, Davies, Sethi & McCloskey, 1984; Bose & Davies, 1984; Wenska, Paszyc & Skalski, 1983; Wenska, 1985). However, the available information concerning the possibility of photocycloaddition of purine to pyrimidine and the stability of the resulting photodimers is still limited and ambiguous. Thus further structural data are needed to shed more light on these problems.

Irradiation with UV light of an aqueous solution of the title compound leads to a cyclobutane product resulting from cycloaddition of imidazole and thymine C=C double bonds (Wenska, 1985). The product of this photoreaction has a *cis–syn* configuration (Gdaniec, Wenska & Kozioł, 1985). This X-ray study of the title compound was undertaken to determine the conformation of this compound in the crystal and investigate whether or not a solid-state photoreaction might lead to a similar product of intramolecular photocycloaddition.

Experimental. Colourless crystal 0·5 × 0·5 × 0·4 mm from methanol; D_m not determined; Syntex $P2_1$ diffractometer, graphite monochromator; lattice parameters from 15 reflections; profiles measured for 1724 unique reflections with $2\theta \le 48°$ (h 0→9, k 0→14, l −12→12), profile analysis according to Lehmann & Larsen (1974), no significant intensity variation for two standard reflections; absorption ignored; 1268 reflections with $I \ge 1{\cdot}96\sigma(I)$; structure solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); full-matrix least-squares refinement on F with *SHELX*76 (Sheldrick, 1976), $w = 1/[\sigma^2(F) + 0{\cdot}00013F^2]$; final refinement: anisotropic non-H atoms, isotropic H atoms (from ΔF map) and empirical isotropic extinction parameters x used to correct F_c according to $F_c' = F_c(1 - xF_c^2/\sin\theta)$, x converged at 0·0116 (9); $R = 0{\cdot}034$ and $wR = 0{\cdot}040$; $S = 1{\cdot}95$; in final refinement cycle $\Delta/\sigma \le 0{\cdot}02$, largest peak in final ΔF map 0·11, largest hole −0·13 e Å^{-3}; atomic

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scattering factors from *International Tables for X-ray Crystallography* (1974); computer programs: *SHELX76* (Sheldrick, 1976), local programs (Jaskolski, 1982), *PLUTO* (Motherwell & Clegg, 1978), *ORTEP* (Johnson, 1976).

Discussion. Final positional parameters are given in Table 1, molecular dimensions in Table 2.* A stereoview of the molecule with the atom-numbering scheme is shown in Fig. 1. As the molecule adopts an extended form, no intramolecular cyclobutane ring is likely to form upon UV irradiation of the crystals. To reach the conformation suitable for photocycloaddition of the imidazole and thymine C=C double bonds, a rotation of *ca* −120° around the C(9)—C(10) bond would be necessary.

Bond distances and angles in the thymine moiety are in good agreement with the mean values in neutral uracil reported by Taylor & Kennard (1982). The thymine ring is not exactly planar ($\chi^2 = 70$). The deviations from the best plane through the six ring atoms range from −0·009 (2) to 0·011 (2) Å. The atoms O(2), O(4), C(7) and C(8) lie 0·006 (2), −0·045 (2), 0·028 (4), 0·014 (2) Å, respectively, from this plane.

The dimensions of the imidazole moiety are in good agreement with those reported for the imidazole molecule (Craven, McMullan, Bell & Freeman, 1977). The five imidazole ring atoms are coplanar ($\chi^2 = 2{\cdot}0$), C(10) being 0·034 (2) Å from their best plane.

The arrangement of the molecule in the crystal lattice is shown in Fig. 2. The molecules are joined by

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

$$U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}.$$

	x	y	z	U_{eq}
N(1)	0·9861 (2)	0·2841 (1)	0·4059 (1)	0·0403 (6)
C(2)	1·0768 (2)	0·3139 (2)	0·5232 (2)	0·0427 (7)
O(2)	1·2113 (2)	0·2820 (1)	0·5636 (1)	0·0634 (7)
N(3)	1·0061 (2)	0·3815 (1)	0·5922 (2)	0·0413 (6)
C(4)	0·8550 (2)	0·4213 (2)	0·5564 (2)	0·0424 (7)
O(4)	0·8050 (2)	0·4797 (1)	0·6297 (1)	0·0622 (6)
C(5)	0·7672 (2)	0·3886 (2)	0·4309 (2)	0·0431 (7)
C(6)	0·8358 (2)	0·3220 (2)	0·3638 (2)	0·0414 (7)
C(7)	0·6040 (3)	0·4299 (3)	0·3827 (3)	0·069 (1)
C(8)	1·0535 (3)	0·2126 (2)	0·3243 (2)	0·0497 (8)
C(9)	1·1454 (3)	0·2686 (2)	0·2395 (2)	0·0501 (8)
C(10)	1·0435 (3)	0·3436 (2)	0·1480 (2)	0·0471 (7)
N(1*P*)	1·1216 (2)	0·3852 (1)	0·0501 (1)	0·0412 (6)
C(2*P*)	1·0771 (3)	0·3700 (2)	−0·0778 (2)	0·0486 (8)
N(3*P*)	1·1665 (2)	0·4187 (1)	−0·1423 (2)	0·0512 (7)
C(4*P*)	1·2756 (3)	0·4687 (2)	−0·0496 (2)	0·0542 (8)
C(5*P*)	1·2492 (3)	0·4491 (2)	0·0684 (2)	0·0502 (8)

Table 2. *Molecular dimensions: bond lengths* (Å), *bond angles* (°) *and important torsional angles* (°) *with e.s.d.'s in parentheses*

N(1)—C(2)	1·374 (3)	C(8)—N(1)	1·471 (3)
N(1)—C(6)	1·374 (3)	C(8)—C(9)	1·513 (3)
N(1)—C(8)	1·471 (3)	C(9)—C(10)	1·506 (3)
C(2)—O(2)	1·224 (3)	C(10)—N(1*P*)	1·465 (3)
C(2)—N(3)	1·367 (3)	N(1*P*)—C(2*P*)	1·343 (3)
N(3)—C(4)	1·382 (3)	N(1*P*)—C(5*P*)	1·358 (3)
C(4)—O(4)	1·227 (3)	C(2*P*)—N(3*P*)	1·305 (3)
C(4)—C(5)	1·445 (3)	N(3*P*)—C(4*P*)	1·366 (3)
C(5)—C(6)	1·335 (3)	C(4*P*)—C(5*P*)	1·350 (3)
C(5)—C(7)	1·495 (4)		
C(2)—N(1)—C(6)	120·7 (2)	N(1)—C(8)—C(9)	113·0 (2)
C(2)—N(1)—C(8)	119·0 (2)	C(8)—C(9)—C(10)	112·0 (2)
C(6)—N(1)—C(8)	120·2 (2)	C(9)—C(10)—N(1*P*)	113·0 (2)
N(1)—C(2)—O(2)	122·5 (2)	C(10)—N(1*P*)—C(2*P*)	126·4 (2)
N(1)—C(2)—N(3)	115·3 (2)	C(10)—N(1*P*)—C(5*P*)	127·6 (2)
O(2)—C(2)—N(3)	122·2 (2)	C(2*P*)—N(1*P*)—C(5*P*)	105·9 (2)
C(2)—N(3)—C(4)	126·9 (2)	N(1*P*)—C(2*P*)—N(3*P*)	113·1 (2)
N(3)—C(4)—C(5)	115·1 (2)	N(3*P*)—C(4*P*)—C(5*P*)	110·5 (2)
N(3)—C(4)—O(4)	120·0 (2)	C(2*P*)—N(3*P*)—C(4*P*)	104·1 (2)
C(4)—C(5)—C(6)	118·1 (2)	C(6)—C(5)—C(7)	123·1 (2)
C(4)—C(5)—C(7)	118·8 (2)	N(1)—C(6)—C(5)	123·9 (2)
O(4)—C(4)—C(5)	125·0 (2)	N(1*P*)—C(5*P*)—C(4*P*)	106·4 (2)
C(2)—N(1)—C(8)—C(9)	−86·3 (2)	C(8)—C(9)—C(10)—N(1*P*)	−170·0 (2)
C(6)—N(1)—C(8)—C(9)	92·4 (2)	C(9)—C(10)—N(1*P*)—C(2*P*)	116·2 (2)
N(1)—C(8)—C(9)—C(10)	−60·9 (2)	C(9)—C(10)—N(1*P*)—C(5*P*)	−66·3 (2)

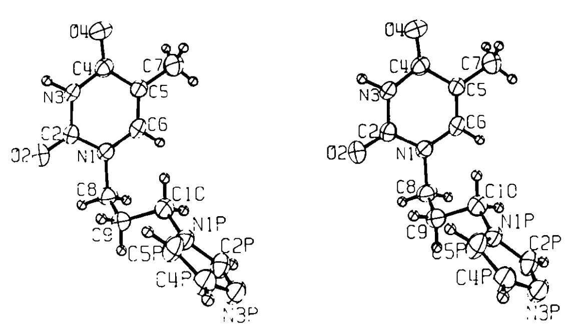

Fig. 1. Stereoview of the molecule and atom-numbering scheme. The thermal ellipsoids of non-H atoms are for 45% probability (Johnson, 1976).

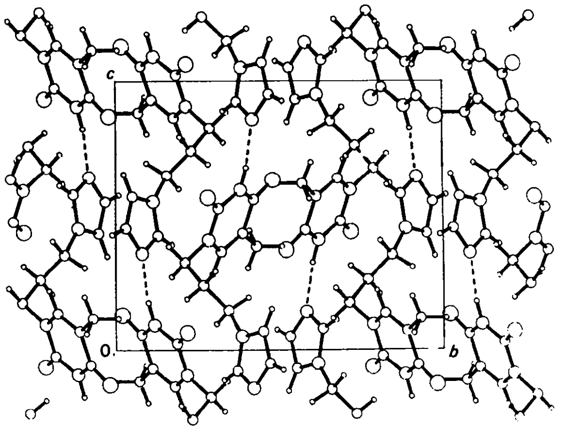

Fig. 2. Projection of the structure on the *bc* plane. Hydrogen bonds are indicated by dotted lines.

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* Lists of observed and calculated structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43180 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

$N(3)—H(3)\cdots N(3P^{i})$ hydrogen bonds [$N(3)\cdots N(3P^{i})$ 2·899 (3), $H(3)\cdots N(3P^{i})$ 1·96 (3) Å, $N(3)—H(3)\cdots N(3P^{i})$ 171 (2)°; (i): $x, y, 1+z$] to form chains along the c axis.

Two thymine residues at x, y, z and $2-x, 1-y, 1-z$ show some ring–ring overlap. The shortest distances between the two overlapping moieties are $N(3)\cdots C(4)$ [3·343 (3) Å] and $C(2)\cdots O(4)$ [3·380 (3) Å] with N(3) almost directly above C(4) and C(2) above O(4). There is no other overlap between the thymine or imidazole rings.

We have made a careful inspection of the thymine and imidazole C=C and C=N bond surroundings to find those double bonds which could be reactant in a possible solid-state photoreaction. The shortest contact between the mid-points of the double bonds is between C(5)—C(6) and $C(2P^{ii})—N(3P^{ii})$ [(ii): $-\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$] and equals 3·549 (3) Å. However, as these bonds are far from being parallel the orientation of their π-electron orbitals is unfavorable for a photochemical reaction (Frank & Paul, 1973). All other distances between the mid-points of the C=C and C=N bonds are longer than 4·2 Å.

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Structure de la (−) Bromo-10 Méthoxycarbonyl-1 Nitro-16 Aspidospermidine-(2*R*,7*R*,16*S*,20*S*,21*S*)

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Abstract. $C_{21}H_{26}BrN_3O_4$, $M_r = 464\cdot4$, orthorhombic, $P2_12_12_1$, $a = 7\cdot966$ (1), $b = 8\cdot646$ (1), $c = 29\cdot947$ (7) Å, $V = 2062\cdot6$ Å^3, $Z = 4$, $D_x = 1\cdot495$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 2\cdot00$ mm^{-1}, $F(000) = 960$, $T = 294$ (1) K, $R = 0\cdot037$ for 1753 independent reflections [$I > 2\sigma(I)$]. The ring conformations are planar for *A* and *B*, approximately half-chair for *C*, chair for *D* and boat for *E*. An intramolecular hydrogen bond $C—H\cdots N$ stabilizes the boat conformation of ring *E*. The ring-junction configurations are *cis* for *B*/*E*, *C*/*E* and *D*/*E* and *trans* for *C*/*D*. The *R* value allows a choice between the two enantiomers. One, corresponding to the absolute configuration, is consistent with starting from (−)-

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