

pyridine ring is higher than in 4-dimethylamino-pyridine. This suggests that the higher conjugation arises from introduction of the *N*-oxide group.

The N—O bond distance is considerably longer than the mean of 1.293 Å in free *N*-oxide groups and is nearly equal to 1.35 Å in strongly hydrogen-bonded *N*-oxide groups (Eichhorn, 1987). Thus, the long N—O bond length arises from hydrogen bonds with two water molecules which are placed above and below the molecular plane, respectively.

The crystal structure is composed of networks of hydrogen bonds. The hexagonal ring, O(10)—O(11)—O(12')—O(10')—O(11')—O(12)—O(10), in which the primed atoms are related to the corresponding unprimed ones by the operation $(1-x, 1-y, -z)$, is formed by hydrogen bonds. The interatomic distances are O(10)···H(O11) = 1.89 (2) [O···O 2.740 (2)], O(10)···H(O12) = 1.89 (2) [2.731 (2)] and O(12)···H'(O11') = 2.06 (2) Å [2.858 (2) Å]. Each hexagonal ring is linked to adjacent ones along the *c* axis by the

hydrogen bonds between the water molecules, O(11)···H'(O12) (*x, y, z* - 1) = 2.19 (2) Å [O···O 2.903 (2) Å].

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Structure of 1-(2-Carbamoylthyl)thymine

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Abstract. C₈H₁₁N₃O₃, *M_r* = 197.20, monoclinic, *P*2₁/*n*, *a* = 15.537 (2), *b* = 15.845 (2), *c* = 7.446 (1) Å, β = 96.50 (1)°, *V* = 1821.3 (4) Å³, *Z* = 8, *D_m* = 1.439, *D_x* = 1.438 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 1.21 cm⁻¹, *F*(000) = 832, *T* = 293 K, *R* = 0.064 for 1389 observed reflexions (*F_o* ≥ 3σ). Two independent molecules, *A* and *B*, are related by pseudo-centrosymmetry and are linked together by two N(amide)—H···O(2) hydrogen bonds. N(3) of the *B* molecule is also hydrogen-bonded to O(amide) of the *A* molecule. The thymine moieties of *A* related by the crystallographic inversion form a pair through N(3)—H···O(4) hydrogen bonds.

Introduction. As part of serial studies on elementary patterns in protein–nucleic acid interactions, we have been engaged in analysing model crystals that contain the side groups of amino acids and nucleotide bases (e.g. Takenaka & Sasada, 1985). The present paper deals with the interaction between thymine and the carbamoyl group.

Experimental. 1-(2-Carboxyethyl)thymine, synthesized from thymine and β-propiolactone according to Kondo, Sato & Takemoto (1973), was purified by ion-exchange

column chromatography (Diaion SA10, CH₃COO-type). Esterification of 1-(2-carboxyethyl)thymine with HCl-saturated methanol and then ammonolysis with NH₃-saturated methanol gave the carbamoylthyl derivative. The products were purified on an ion-exchange column. Colourless rod-like crystals obtained from methanol solution, space group from Weissenberg photographs; systematic absences *h* + *l* = 2*n* + 1 for *h*0*l* and *k* = 2*n* + 1 for 0*k*0. Crystal size 0.25 × 0.1 × 0.1 mm, Rigaku four-circle diffractometer, graphite-monochromated Mo *K*α. Unit-cell dimensions from 52 reflexions with 10 < 2θ < 37°. Intensities by ω scan, scan width 1.3° (ω), scan rate 4° min⁻¹. Five reference reflexions monitored every 50 reflexions showed no significant intensity deterioration. Out of 3319 independent reflexions, 1930 with *F_o* < 3σ considered unobserved; 2θ_{max} = 41°, *h* -15→15, *k* 0→15, *l* 0→7. Correction for Lorentz and polarization factors but not for absorption effects. σ²(*F_o*) = σ_p²(*F_o*) + *qF_o*² where σ_p evaluated by counting statistics and *q* (=0.001) from measurement of monitored reflexions (McCandlish & Stout, 1975).

Structure solved by direct methods, refined by block-matrix least squares; all H atoms found on a difference map refined isotropically; ∑*w*(|*F_o*| - |*F_c*|)²

Table 1. Fractional coordinates and equivalent isotropic temperature factors

$B_{eq} = 8\pi^2(U_1 + U_2 + U_3)/3$, where U_1 , U_2 , and U_3 are the principal components of the mean-square displacement matrix U . Values in $\langle \rangle$ are the anisotropy defined by $[\sum(B_{eq} - 8\pi^2U_j)^2/3]^{1/2}$ and those in $()$ are e.s.d.'s; they refer to the last decimal places.

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
N(1) <i>A</i>	0.2481 (4)	0.0235 (4)	0.7849 (8)	2.1 (9)
C(2) <i>A</i>	0.1886 (5)	-0.0342 (5)	0.713 (1)	2.0 (6)
O(2) <i>A</i>	0.2033 (3)	-0.1098 (3)	0.7184 (7)	3.0 (10)
N(3) <i>A</i>	0.1118 (4)	-0.0015 (4)	0.6361 (9)	2.5 (16)
C(4) <i>A</i>	0.0896 (5)	0.0822 (5)	0.624 (1)	2.2 (9)
O(4) <i>A</i>	0.0183 (3)	0.1046 (3)	0.5460 (7)	3.2 (17)
C(5) <i>A</i>	0.1547 (5)	0.1412 (5)	0.707 (1)	2.3 (10)
C(5 <i>m</i>) <i>A</i>	0.1341 (5)	0.2338 (5)	0.700 (1)	3.4 (10)
C(6) <i>A</i>	0.2296 (5)	0.1082 (5)	0.782 (1)	2.4 (10)
C(7) <i>A</i>	0.3342 (5)	-0.0073 (5)	0.869 (1)	2.8 (14)
C(8) <i>A</i>	0.3925 (5)	-0.0317 (5)	0.726 (1)	2.4 (12)
C(9) <i>A</i>	0.4116 (5)	0.0416 (5)	0.609 (1)	2.4 (12)
N(9) <i>A</i>	0.3954 (4)	0.0305 (4)	0.4310 (9)	2.9 (7)
O(9) <i>A</i>	0.4401 (4)	0.1093 (4)	0.6726 (8)	3.6 (13)
N(1) <i>B</i>	0.2780 (4)	-0.2603 (4)	0.2214 (8)	2.4 (9)
C(2) <i>B</i>	0.3414 (5)	-0.2022 (6)	0.270 (1)	2.9 (14)
O(2) <i>B</i>	0.3280 (4)	-0.1257 (3)	0.2578 (8)	3.5 (13)
N(3) <i>B</i>	0.4209 (4)	-0.2353 (4)	0.3348 (9)	2.8 (7)
C(4) <i>B</i>	0.4420 (5)	-0.3204 (6)	0.358 (1)	3.2 (7)
O(4) <i>B</i>	0.5153 (4)	-0.3402 (4)	0.4124 (8)	4.5 (17)
C(5) <i>B</i>	0.3704 (5)	-0.3780 (5)	0.310 (1)	2.5 (10)
C(5 <i>m</i>) <i>B</i>	0.3856 (6)	-0.4711 (6)	0.341 (1)	3.9 (11)
C(6) <i>B</i>	0.2935 (5)	-0.3457 (5)	0.247 (1)	2.4 (8)
C(7) <i>B</i>	0.1903 (5)	-0.2298 (5)	0.143 (1)	2.8 (9)
C(8) <i>B</i>	0.1375 (5)	-0.2013 (5)	0.290 (1)	2.6 (10)
C(9) <i>B</i>	0.1063 (5)	-0.2740 (5)	0.400 (1)	2.3 (10)
N(9) <i>B</i>	0.1013 (4)	-0.2599 (4)	0.5730 (9)	2.7 (10)
O(9) <i>B</i>	0.0870 (4)	-0.3436 (3)	0.3317 (7)	3.8 (21)

minimized; $w = 1/\sigma^2(F_o)$; zero reflexions with $|F_c| > F_{lim}$ ($F_{lim} = 4.97$) included in refinement assuming $F_o = F_{lim}$ and $w = w(F_{lim})$; final R value 0.064 for 1389 reflexions with $|F_o| \geq 3\sigma$ ($wR = 0.064$, $S = 1.23$); maximum shifts of parameters 0.3σ , $\Delta\rho$ peak 0.45 e \AA^{-3} . (R was somewhat high, because the crystal used was too small to give sufficient intensities.) Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs used: *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *LSAP80* (Takenaka & Sasada, 1980), *DCMS82* (Takenaka & Sasada, 1982) and *LISTUP* (Takenaka & Sasada, 1983). Final atomic parameters are given in Table 1.*

Discussion. The two independent molecules, *A* and *B*, are related by pseudo-centrosymmetry. Fig. 1 indicates the hydrogen bonds in which they participate. The crystal structure, as seen in Fig. 2, consists of a three-dimensional hydrogen-bonded network of the two molecules. As for interactions between the carbamoyl

group and thymine, two kinds of hydrogen bonds are found. One is $N(9)\text{---}H\cdots O(2)$ and the other is $N(3)\text{---}H\cdots O(9)$. In the crystal of 1-(2-carbamoyl-ethyl)uracil (Fujita, Takenaka & Sasada, 1984), the corresponding $N(9)$ of the carbamoyl group binds to $O(4)$ of uracil. The thymine moieties of centrosymmetrically related *A* molecules form a hydrogen-bonded pair through the $N(3)\text{---}H\cdots O(4)$ hydrogen bonds, but those of the *B* molecules do not. The remaining hydrogen bonds are between the carbamoyl groups.

Table 2 lists the bond distances and angles. There are no significant differences in molecular dimensions between the two molecules except for those involving $O(4)$. The longer $C(4)\text{---}O(4)$ bond in *A* may result from the hydrogen bond. The molecules adopt a bent conformation as a whole; the torsion angles $C(2)\text{---}$

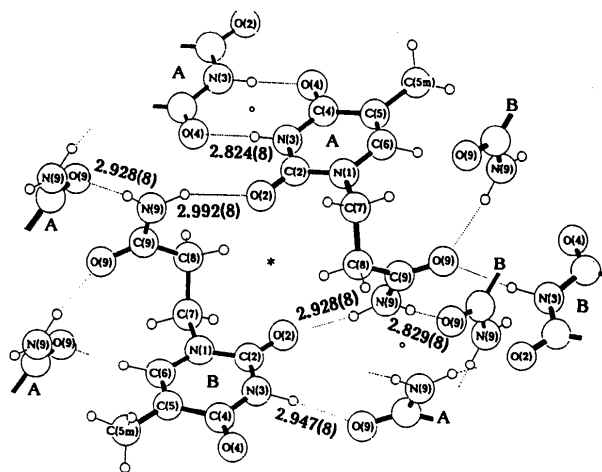


Fig. 1. The two independent molecules and the hydrogen bonds in which they participate (dotted lines). Hydrogen-bond distances (in \AA) are also indicated. The asterisk indicates the pseudo inversion centre.

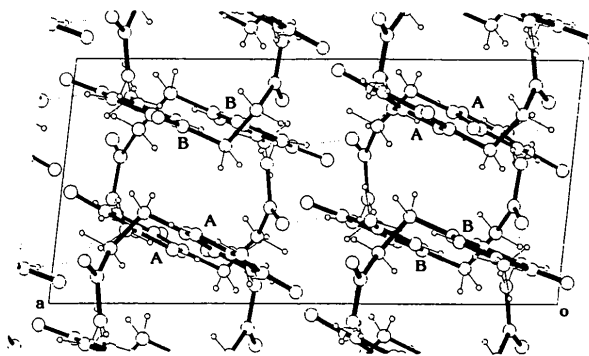


Fig. 2. Crystal structure of 1-(2-carbamoyl-ethyl)thymine, viewed down the *b* axis.

* 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 44476 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

E.s.d.'s are given in parentheses.

	<i>A</i>	<i>B</i>
N(1)—C(2)	1.36 (1)	1.37 (1)
N(1)—C(6)	1.37 (1)	1.38 (1)
N(1)—C(7)	1.49 (1)	1.50 (1)
C(2)—O(2)	1.219 (9)	1.23 (1)
C(2)—N(3)	1.37 (1)	1.38 (1)
N(3)—C(4)	1.37 (1)	1.39 (1)
C(4)—O(4)	1.243 (9)	1.21 (1)
C(4)—C(5)	1.46 (1)	1.45 (1)
C(5)—C(5 <i>m</i>)	1.50 (1)	1.51 (1)
C(5)—C(6)	1.34 (1)	1.34 (1)
C(7)—C(8)	1.52 (1)	1.51 (1)
C(8)—C(9)	1.50 (1)	1.53 (1)
C(9)—N(9)	1.33 (1)	1.32 (1)
C(9)—O(9)	1.23 (1)	1.24 (1)
C(2)—N(1)—C(6)	121.2 (6)	120.9 (6)
C(2)—N(1)—C(7)	118.7 (6)	118.8 (6)
C(6)—N(1)—C(7)	120.1 (6)	120.3 (6)
N(1)—C(2)—O(2)	122.1 (6)	122.2 (7)
N(1)—C(2)—N(3)	115.5 (6)	115.3 (6)
O(2)—C(2)—N(3)	122.5 (6)	122.6 (7)
C(2)—N(3)—C(4)	126.6 (6)	126.9 (6)
N(3)—C(4)—O(4)	120.8 (6)	119.5 (7)
N(3)—C(4)—C(5)	115.7 (6)	114.6 (7)
O(4)—C(4)—C(5)	123.5 (6)	125.9 (7)
C(4)—C(5)—C(5 <i>m</i>)	118.5 (6)	118.4 (7)
C(4)—C(5)—C(6)	116.9 (7)	118.4 (7)
C(5 <i>m</i>)—C(5)—C(6)	124.6 (7)	123.2 (7)
N(1)—C(6)—C(5)	124.0 (7)	123.9 (7)
N(1)—C(7)—C(8)	111.4 (6)	111.0 (6)
C(7)—C(8)—C(9)	112.4 (6)	113.4 (6)
C(8)—C(9)—N(9)	116.6 (6)	117.1 (6)
C(8)—C(9)—O(9)	122.3 (7)	121.8 (7)
N(9)—C(9)—O(9)	121.7 (7)	121.1 (7)

N(1)—C(7)—C(8) are 76.7 (8) and $-80.7 (8)^\circ$, N(1)—C(7)—C(8)—C(9) 61.7 (8) and $-72.0 (8)^\circ$, C(7)—C(8)—C(9)—O(9) 52 (1) and $-34 (1)^\circ$, and C(7)—C(8)—C(9)—N(9) $-127.0 (7)$ and $145.8 (7)^\circ$, for *A* and *B*, respectively.

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Structure of 3-(2-Hydroxy-4-methylphenyl)-3-phenylphthalide

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Abstract. $C_{21}H_{16}O_3$, $M_r = 316.36$, monoclinic, $P2_1/n$, $a = 8.099 (2)$, $b = 11.178 (3)$, $c = 18.524 (6)$ Å, $\beta = 99.01 (2)^\circ$, $V = 1656.3 (8)$ Å³, $Z = 4$, $D_m = 1.26$, $D_x = 1.268$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.091$ mm⁻¹, $F(000) = 664$, $T = 293$ K. Final $R = 0.037$ for 1514 unique observed reflections. The geometry of the five-membered ring is generally comparable to other γ -lactones. The tetrahedral symmetry at the central tetra-substituted C atom is distorted by its association with the fused-ring system

and by the overcrowding in this molecule, the valence angles range from 101.8 (2) to 113.9 (2)°. The packing is determined by an intermolecular hydrogen bond between the hydroxyl and carbonyl groups with an O...O distance of 2.758 (3) Å and an O—H...O angle of 170 (3)°.

Introduction. 3,3-Diarylpthalides display laxative properties (Loewe & Hubacher, 1941; Hubacher, Doernberg & Horner, 1953) and serve as the sub-