important que celui observé dans le composé Co-4 DPC.

Ces molécules forment des piles infinies liées entre elles par des liaisons hydrogène autour de l'atome H(7). Les caractéristiques en sont: distances N(7)–H(7)···N(3^{i}) 2,882 (4), H(7)···N(3^{i}) 1,93 (4), N(7)–H(7) 0,96 (4) Å; angle N(7)–H(7)···N(3^{i}) 173 (3)° [code de symétrie: (i) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$]. Ces piles se déduisent les unes des autres par des plans de glissement *c*.

Des contacts de van der Waals {dont la plus proche: 3,219 (7) Å entre les atomes C(2) et C(2^{ii}) [code de symétrie: (ii) $-x, y, \frac{1}{2}-z$]} assurent la cohésion du cristal.

L'existence de liaisons hydrogène rapproche l'arrangement cristallin observé dans la variété monoclinique II de celui rencontré dans la variété monoclinique I. En effet, les molécules forment alors des rubans infinis torsadés alternativement (Nguyen-Huy Dung *et al.*, 1984). Par ailleurs, la présence de piles infinies de molécules est à rapprocher de l'empilement moléculaire déjà décrit dans le composé Co-4 DPC (Viossat *et al.*, 1984).

En conclusion, l'étude de la conformation du noyau cyclohexadiényle (*B*) et de l'arrangement cristallin montre que la structure de la variété monoclinique II du dihydro-5,6 pyrimidino[5,4-*c*]carbazole présente d'étroites filiations avec celle de la variété monoclinique I d'une part, et avec celle du Co-4 DPC d'autre part. Il est probable que l'existence de ces différentes variétés polymorphiques n'entraîne pas une variation de leur biodisponibilité.

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Elementary Binding Patterns in Protein–Nucleic Acid Interactions. Structure of 1-(2-Carbamoylethyl)uracil, $C_7H_9N_3O_3$*

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(*Received* 29 *March* 1984; *accepted* 16 *May* 1984)

Abstract. $M_r = 183{\cdot}17$, triclinic, $P\bar{1}$, $a = 8{\cdot}869$ (1), $b = 11{\cdot}318$ (1), $c = 4{\cdot}444$ (1) Å, $\alpha = 105{\cdot}59$ (1), $\beta = 99{\cdot}75$ (1), $\gamma = 103{\cdot}87$ (1)°, $V = 404{\cdot}0$ (1) Å^3, $Z = 2$, $D_m = 1{\cdot}51$, $D_x = 1{\cdot}506$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 10{\cdot}30$ cm^{-1}, $F(000) = 192$, room temperature, $R = 0{\cdot}040$ for 1224 reflections. The uracil moieties form pairs through N(3)–H···O(4) hydrogen bonds around an inversion center. The carbamoyl group of the adjacent molecule interacts with O(4) of the paired uracil. A CNDO/2 calculation indicates that the interaction of the carbamoyl group with O(4) of uracil is more stable than that with O(2).

* 3-(2,4-Dioxo-1-pyrimidinyl)propionamide.

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0108-2701/84/091605-03$01.50

Introduction. The operator-binding domains of many regulatory proteins contain the asparagine and glutamine residues (Sauer, Yocum, Doolittle, Lewis & Pabo, 1982), which are probably responsible for the specificity. Interactions of the side chains of Asn and Gln have been reported with adenine (Takimoto, Takenaka & Sasada, 1981, 1982) and with cytosine (Fujita, Takenaka & Sasada, 1984) from analyses of model crystals. We prepared and studied the binary model crystal containing uracil and a carbamoyl group.

Experimental. 1-(2-Carboxyethyl)uracil, synthesized from uracil and β-propiolactone in dimethylformamide (Kondo, Sato & Takemoto, 1973), was

esterified in HCl-saturated MeOH and then ammonolyzed in NH_3-saturated MeOH to give title compound. Thin plate-like crystals from MeOH solution; D_m by flotation in mixture of n-C_6H_{14} and CCl_4; crystal size 0·5 × 0·5 × 0·1 mm; Rigaku four-circle diffractometer; Ni-filtered Cu $K\alpha$ radiation; unit-cell dimensions from 70 high-angle reflections; scan range $3 < 2\theta < 123°$; ω-scan mode; scan speed 2° (in ω) min^{-1}; scan width 1·1° (in ω) plus α_1–α_2 divergence; five reference reflections monitored periodically showed no significant deterioration; correction for Lorentz and polarization factors, not for absorption; 1249 independent reflections, 24 weak reflections below background considered as zero reflections; standard deviations estimated by $\sigma^2(F_o) = \sigma_p^2(F_o) + qF_o^2$ (McCandlish & Stout, 1975), $\sigma_p(F_o)$ evaluated by counting statistics and q estimated to be $1·53 \times 10^{-5}$ from measurement of monitored reflections. h −10–9, k −12–11, l 0–5. Normalized structure-factor distribution suggested centrosymmetry, but initial attempts in $P\bar{1}$ failed to yield a solution; structure obtained on basis of $P1$ and refined by full-matrix least squares; when positional and isotropic thermal parameters converged, two independent molecules found to be closely related by inversion; refinement in $P\bar{1}$, all hydrogen atoms found on difference map; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F_o)$; zero reflections with $|F_c| \geq F_{lim}$ (F_{lim} = 0·223) included in the refinement by assuming $|F_o| = F_{lim}$ with $w = w(F_{lim})$; final $R = 0·040$ for 1224 reflections with $F_o > 3\sigma(F_o)$ ($Rw = 0·044$ and S = 6·29); max. Δ of parameters in last cycle $0·02\sigma$; $\Delta\rho$ max. +0·13 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); all calculations with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *LSAP*80 (Takenaka & Sasada, 1980), *LISTUP* (Takenaka & Sasada, 1983), *DCMS*82 (Takenaka & Sasada, 1982) and *CNDO*/2 (Kikuchi, 1971). Final atomic parameters are given in Table 1.*

Discussion. Fig. 1 shows the crystal structure together with the atom numbering. The bond distances and angles are given in Table 2. The pyrimidine ring is planar within ±0·020 Å. The bond distances and angles of the uracil moiety are in good agreement with those of 1-(2-carboxyethyl)uracil (Fujita, Takenaka & Sasada, 1982) and uridine (Green, Rosenstein, Shiono, Abraham, Trus & Marsh, 1975). The dimensions of the carbamoyl group are close to those of asparagine

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances and angles involving H atoms, least-squares planes for the pyrimidine ring and the carbamoyl group and hydrogen-bond geometry have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39495 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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 principal components of the mean-square displacement matrix U. Values in < > are anisotropicity defined by $[\sum(B_{eq} - 8\pi^2U_i)^2/3]^{1/2}$ and those in ( ) are e.s.d.'s; they refer to last decimal places.

	x	y	z	B_{eq}(Å^2)
N(1)	0·2059 (2)	0·7842 (1)	0·9930 (3)	2·67<22>
C(2)	0·2200 (2)	0·9080 (2)	0·9868 (4)	2·74<36>
O(2)	0·3243 (2)	1·0018 (1)	1·1744 (3)	3·91<159>
N(3)	0·1064 (2)	0·9169 (1)	0·7466 (3)	2·76<65>
C(4)	−0·0172 (2)	0·8176 (2)	0·5247 (4)	2·70<81>
O(4)	−0·1065 (1)	0·8397 (1)	0·3144 (3)	3·57<144>
C(5)	−0·0286 (2)	0·6938 (2)	0·5584 (5)	3·07<96>
C(6)	0·0818 (2)	0·6823 (2)	0·7857 (4)	2·97<78>
C(9)	0·3293 (2)	0·7672 (2)	1·2333 (4)	3·14<65>
C(10)	0·4850 (2)	0·7702 (2)	1·1351 (4)	2·95<62>
C(11)	0·4782 (2)	0·6526 (2)	0·8686 (4)	2·65<45>
O(12)	0·3509 (1)	0·5711 (1)	0·7036 (3)	3·72<167>
N(13)	0·6195 (2)	0·6449 (2)	0·8163 (4)	3·91<211>

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

Standard deviations are given in parentheses.

N(1)–C(2)	1·385 (2)	N(1)–C(6)	1·367 (2)
N(1)–C(9)	1·480 (2)	C(2)–O(2)	1·212 (2)
C(2)–N(3)	1·381 (2)	N(3)–C(4)	1·373 (2)
C(4)–O(4)	1·238 (2)	C(4)–C(5)	1·430 (2)
C(5)–C(6)	1·339 (2)	C(9)–C(10)	1·513 (2)
C(10)–C(11)	1·506 (2)	C(11)–O(12)	1·233 (2)
C(11)–N(13)	1·331 (2)		
C(2)–N(1)–C(6)	120·9 (1)	C(2)–N(1)–C(9)	117·5 (1)
C(6)–N(1)–C(9)	121·7 (1)	N(1)–C(2)–O(2)	123·1 (1)
N(1)–C(2)–N(3)	114·7 (1)	O(2)–C(2)–N(3)	122·1 (1)
C(2)–N(3)–C(4)	126·9 (1)	N(3)–C(4)–O(4)	119·6 (1)
N(3)–C(4)–C(5)	114·9 (1)	O(4)–C(4)–C(5)	125·5 (1)
C(4)–C(5)–C(6)	119·3 (1)	N(1)–C(6)–C(5)	123·1 (1)
N(1)–C(9)–C(10)	113·5 (1)	C(9)–C(10)–C(11)	114·8 (1)
C(10)–C(11)–O(12)	122·6 (1)	C(10)–C(11)–N(13)	115·2 (1)
O(12)–C(11)–N(13)	122·2 (1)		

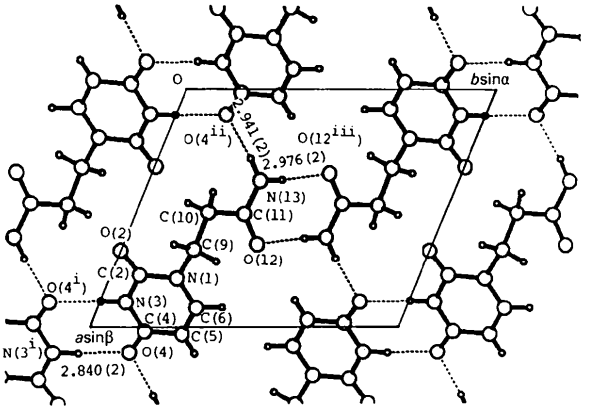

Fig. 1. The crystal structure of 1-(2-carbamoylethyl)uracil viewed down the c axis. The distances are in Å. Atom numbering is also shown. Symmetry code: (i) $1 + x, y - 1, z$; (ii) $-x, 1 - y, -z$; (iii) x, y, z. The molecule with the numbering is at $1 - x, 1 - y, 1 - z$.

(Ramanadham, Shikka & Chidambaram, 1972) and of glutamine (Koetzle, Frey, Lehmann & Hamilton, 1973). The torsion angles in the methylene chain, N(3)–C(2)–N(1)–C(9), C(2)–N(1)–C(9)–C(10), N(1)–C(9)–C(10)–C(11) and C(9)–C(10)–C(11)–N(13), are 176·8 (1), 281·2 (2), 287·3 (2) and 190·4 (1)°, respectively. This conformation is similar to those of the side chains of asparagine and glutamine.

The uracil moieties form a dimer around the inversion center at $(0,1,\frac{1}{2})$ through N(3)–H···O(4) hydrogen bonds with an N···O distance of 2·840 (2) Å and N–H···O angle of 174 (2)°. The carbamoyl group interacts with O(4) of the uracil moiety in the adjacent molecule through an N–H···O hydrogen bond; the N(13)···O(4) distance is 2·941 (2) Å, the N–H···O angle is 170 (2)°, and the torsion angles N(3)–C(4)–O(4)···N(13), C(4)–O(4)···N(13)–C(11) and O(4)···N(13)–C(11)–C(10) are 180·6 (1), 106·6 (1) and 9·6 (2)°, respectively. In addition, the carbamoyl groups form a dimer through the N(13)–H···O(12) hydrogen bonds around the inversion center at $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$; the N···O distance is 2·976 (2) Å and the N–H···O angle is 173 (2)°. The O(2) atom of the uracil moiety does not participate in any hydrogen bonding.

As mentioned above, the carbamoyl group interacts with O(4) of the paired uracil. The peptide moiety is hydrogen bonded with O(4) of uracil in the crystals of *N*-(5-uridinyl)-L-phenylalaninamide (Berman, Hamilton & Rousseau, 1973) and 3-(1-uracilyl)propionylphenylethylamide (Takimoto, Takenaka & Sasada, 1983) and with N(3) and O(4) in the crystal of *N*-(5-uridinyl)-L-leucinamide (Narayanan & Berman, 1977). Thus the O(4) site of the uracil moiety is always involved in hydrogen bonding with amide. On the other hand, the carboxyl group is hydrogen bonded with the O(2) of a paired uracil in the crystal of 3-(1-uracilyl)propionic acid (Fujita, Takenaka & Sasada, 1982) and with N(3) and O(4) in that of 6-methyl-5-uracilylacetic acid (Destro & Marsh, 1972). The formation energies were calculated by a CNDO/2 approximation (Fig. 2). A comparison of energies indicates that the hydrogen bond of the carbamoyl group with O(4) of uracil is about 4 kJ mol^{-1} more stable than that with O(2) and the hydrogen bonds of the carboxyl group with O(2) and with O(4) are comparable. Energy considerations, therefore, explain the observation of the interaction pattern in model crystals.

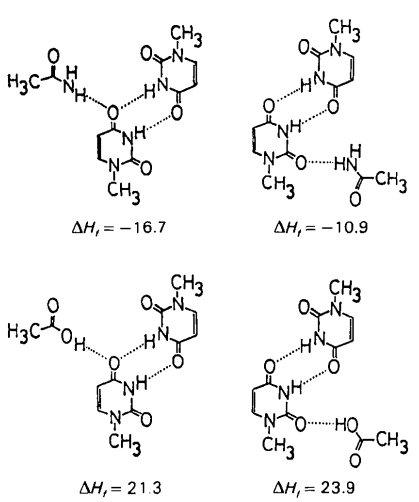

Fig. 2. Formation energies, H_f, in kJ mol^{-1} of the interactions between uracil and the carbamoyl group or the carboxyl group.

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