$w^{1/2}|F_c| \geq 1$ and $w^{1/2}|\Delta F| < 3$, $w = 0$ otherwise. $R = 0{\cdot}028$, $wR = 0{\cdot}037$, $S = 1{\cdot}126$ for 2580 observed reflections ($w \neq 0$). Max. Δ/σ in the final cycle 0·78. No significant peaks in final difference map, highest peak 0·3 e Å^{-3}. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1, \cdots, 4$) (*International Tables for X-ray Crystallography*, 1974). Calculations performed by FACOM M-150F computer at Shionogi Research Laboratories.

Discussion. Atomic coordinates and equivalent isotropic temperature factors of non-H atoms are listed in Table 1.* Bond lengths and angles are given in Table 2. The perspective view of the molecule with the atom-numbering system drawn using *PLUTO* (Motherwell & Clegg, 1978) is presented in Fig. 1. The absolute configuration of the molecule was determined on the basis of the R configuration of C(6).

A stereoscopic view of the crystal structure is given in Fig. 2. Distances between the molecules and a dichloromethane solvate are longer than the sum of the van der Waals radii. In a layer perpendicular to the b axis, there are two intermolecular hydrogen bonds, one between adjacent molecules along the a axis, O(22)···N(23)($1+x$, y, z) = 2·825 (3) Å, and the other between those along the c axis, O(12)···N(17)(x, y, $1+z$) = 2·789 (3) Å. Another hydrogen bond of O(22)···O(25)($2-x$, $\frac{1}{2}+y$, $1-z$) = 2·839 (3) Å is formed between the layers.

The non-planarity of N(5) is one of the important factors in the investigation of the structure–activity relationship of β-lactam antibiotics. Takasuka, Nishikawa & Tori (1982) proposed the angle θ between the N(5)—C(4) bond and the plane of N(5), C(6) and C(8) as a parameter representing the non-planarity regardless of the relevant bond lengths. For the series 7α*H*-1-oxacephem, 7α-methoxy-1-oxacephem, cephalosporin and 7α-methoxycephalosporin having the same substitutents at the 3, 4 and 7β positions, the wavenumbers of the stretching vibration band of C(8)=O(9), measured in solution, correlate linearly with the cos θ values.

The θ value in flomoxef is 29·4°, which is larger than those of the other 7α-methoxy-1-oxacephems having different substituents at the 3, 4 and 7β positions: 21·8° for diphenylmethyl 7α-methoxy-3-(1-methyl-1*H*-tetrazol-5-ylthio)methyl-7β-phenylacetamido-1-oxa-1-dethia-3-cephem-4-carboxylate (Shiro, Nakai, Onoue & Narisada, 1980), and 18·8 and 16·9° for two independent molecules in an asymmetric unit of latamoxef diammonium salt (Shiro, Nakai, Matsubara & Kikkawa, 1982).

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* Lists of structure factors, anisotropic temperature factors of the non-H atoms and coordinates of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44135 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,5-Di(1-pyrrolidinyl)-4-hexene-1,3-dione

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Abstract. $C_{14}H_{22}N_2O_2$, $M_r = 250$, triclinic, $P\bar{1}$, $a = 7{\cdot}300\,(5)$, $b = 8{\cdot}790\,(7)$, $c = 10{\cdot}923\,(7)$ Å, $\alpha = 96{\cdot}04\,(6)$, $\beta = 103{\cdot}38\,(7)$, $\gamma = 90{\cdot}27\,(5)°$, $V = 678{\cdot}3$ Å^3, $Z = 2$, $D_x = 1{\cdot}224$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 0{\cdot}47$ cm^{-1}, $F(000) = 272$, room temperature. $R = 0{\cdot}056$ for 1829 observed reflections. The molecule consists of two planes inclined at 77 (1)° at the methylene C atom between the carbonyl groups.

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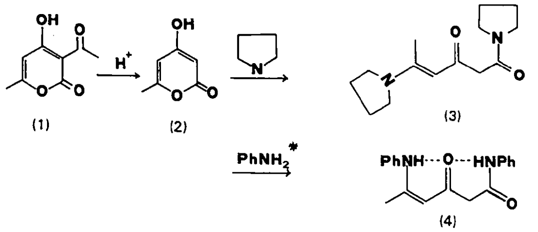
The configuration about the C=C bond is *E*, the pyrrolidine group at position 5 being *trans* with respect to the carbonyl group at position 3. The two pyrrolidine rings have different conformations; one a half-chair, the other an envelope.

Introduction. In nature a large class of aromatic compounds is produced by condensation of poly-β-carbonyl intermediates. Many strategies have been proposed for the synthesis of aromatic natural products. To be profitable such a method depends on the availability of an inexpensive β-diketo compound or derivative as starting material. A promising starting material appears to be dehydroacetic acid (1) (3-acetyl-4-hydroxy-6-methyl-2-pyrone), a dimer of a diketene, which can easily be converted to a β-dicarbonyl (Batelaan, 1976) or a masked dicarbonyl compound. In connection with work on the synthesis of polyketides we prepared the enamine amide of dehydroacetic acid δ-lactone, (2).



A crystallographic analysis of (3) was undertaken to confirm the relative positions of the enamine pyrrolidine cycles in the aliphatic chain.

Experimental. A mixture of 2·8 g (22 mmol) of dehydroacetic acid δ-lactone, (2), and 3·2 g (45 mmol) of pyrrolidine in 200 ml toluene were refluxed under a Dean–Stark separator for 4 h. After removal of the solvent and recrystallization, 5·3 g of pure enamine-amide, (3), were isolated (m.p. 389 K) giving a 96·4% yield. Pale-yellow plate-like crystals were grown from a CH_3Cl/heptane solution, dimensions 0·42 × 0·42 × 0·11 mm. Stoe–Siemens AED2 four-circle diffractometer, monochromated Mo $K\alpha$ radiation, ω/θ scan mode, $2\theta_{max} = 50°$, index limits $h \pm 8$, $k \pm 10$, $l \pm 12$, no significant intensity variation for four standard reflections; 4774 measured, 2371 unique reflections, $R_{int} = 0·018$, 1829 reflections with $F > 5\sigma(F)$ considered observed. Cell constants refined from $\pm\omega$ values of 25 reflections in the range $6 < 2\theta < 40°$. No absorption or extinction corrections applied. Structure solved by automatic multisolution direct methods using *SHELX76* (Sheldrick, 1976) which was used for all further calculations. Full-matrix least-squares refinement to $R = 0·056$, $wR = 0·070$, $w^{-1} = 2·624 \times [\sigma^2(F_o) + 0·00151(F_o^2)]$. Anisotropic thermal parameters for all non-H atoms, H atoms in observed positions and refined isotropically. Atoms C(12) and C(13) undergo considerable thermal motion, their protons included in calculated positions with C—H = 1·08 Å, H—C—H = 109·5°. Refined U_{iso} for C(12) H atoms 0·314 Å^2, for C(13) H atoms 0·219 Å^2. In last cycle of refinement $(\Delta/\sigma)_{max} = 0·09$, $(\Delta/\sigma)_{mean} < 0·02$; final difference map $\Delta\rho_{min} = -0·29$, $\Delta\rho_{max} = 0·36$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Final positional and thermal parameters are given in Table 1.*

Table 1. *Final positional* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 ×10^4)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j a_i^* a_j^* (\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	U_{eq}
C(1)	7309 (4)	5694 (3)	2045 (2)	538 (8)
C(2)	7722 (4)	4690 (3)	937 (3)	555 (9)
C(3)	7905 (3)	2993 (3)	1163 (2)	457 (7)
C(4)	7412 (3)	1965 (3)	31 (2)	460 (8)
C(5)	7439 (3)	379 (3)	−72 (2)	419 (7)
N(6)	6935 (3)	−447 (2)	−1198 (2)	457 (6)
N(7)	8792 (3)	6317 (2)	2924 (2)	552 (7)
O(8)	5685 (3)	5944 (3)	2113 (2)	766 (8)
O(9)	8479 (3)	2676 (2)	2254 (2)	616 (7)
C(10)	8022 (4)	−506 (3)	1043 (3)	548 (9)
C(11)	6372 (4)	226 (3)	−2402 (2)	546 (8)
C(12)	5698 (10)	−1105 (5)	−3347 (4)	1271 (19)
C(13)	6348 (6)	−2461 (4)	−2840 (3)	883 (14)
C(14)	6904 (4)	−2129 (3)	−1422 (3)	534 (8)
C(15)	10787 (4)	6008 (4)	2980 (3)	683 (11)
C(16)	11779 (6)	6676 (4)	4297 (4)	812 (14)
C(17)	10551 (5)	7945 (4)	4619 (3)	782 (12)
C(18)	8551 (5)	7336 (3)	4007 (3)	670 (10)

Discussion. Bond distances and angles in the title compound, (3), are given in Table 2. The numbering scheme used is apparent from Fig. 1, drawn using *ORTEP*II (Johnson, 1976). As a result of steric hindrance between atom O(9) and methyl group C(10) [distance H(10*b*)···O(9) 2·231 (3) Å] angles C(3)—C(4)—C(5) and C(4)—C(3)—O(9) are opened to 127·9 (2) and 127·1 (2)°, respectively, while angle C(2)—C(3)—C(4), of 113·7 (2)°, is closed by the corresponding amount. The remaining bond distances and angles in the molecule are normal except those involving atoms C(12) and C(13) which undergo considerable thermal motion. The N(6) pyrrolidine ring has a C_s [C(12)] envelope conformation while the N(7) pyrrolidine ring has a C_2 [N(7)] half-chair conformation. There are no short intermolecular contacts between non-H atoms in the crystal.

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* Briard, Dubourg, Roques, Castillo, Herault & Declercq (1984).

* Lists of observed and calculated structure factors, H-atom parameters, anisotropic thermal parameters and selected torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44155 (15 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* (°)

C(1)–C(2)	1·510 (4)	N(6)–C(14)	1·472 (3)
C(1)–O(8)	1·224 (3)	C(11)–C(12)	1·478 (5)
C(1)–N(7)	1·341 (3)	C(12)–C(13)	1·405 (6)
C(2)–C(3)	1·539 (3)	C(13)–C(14)	1·506 (6)
C(3)–C(4)	1·426 (3)	N(7)–C(15)	1·471 (3)
C(3)–O(9)	1·231 (3)	N(7)–C(18)	1·452 (3)
C(4)–C(5)	1·387 (3)	C(15)–C(16)	1·508 (5)
C(5)–N(6)	1·333 (3)	C(16)–C(17)	1·498 (5)
C(5)–C(10)	1·495 (3)	C(17)–C(18)	1·527 (5)
N(6)–C(11)	1·472 (3)		

C(2)–C(1)–N(7)	117·1 (2)	C(11)–N(6)–C(14)	110·7 (2)
C(2)–C(1)–O(8)	120·9 (2)	N(6)–C(11)–C(12)	103·9 (2)
N(7)–C(1)–O(8)	122·0 (2)	C(11)–C(12)–C(13)	109·8 (3)
C(1)–C(2)–C(3)	113·1 (2)	C(12)–C(13)–C(14)	108·2 (3)
C(2)–C(3)–C(4)	113·7 (2)	C(13)–C(14)–N(6)	104·0 (2)
C(2)–C(3)–O(9)	118·4 (2)	C(1)–N(7)–C(15)	126·4 (2)
C(4)–C(3)–O(9)	127·9 (2)	C(1)–N(7)–C(18)	121·5 (2)
C(3)–C(4)–C(5)	127·1 (2)	C(15)–N(7)–C(18)	112·0 (2)
C(4)–C(5)–N(6)	120·8 (2)	N(7)–C(15)–C(16)	103·6 (3)
C(4)–C(5)–C(10)	123·1 (2)	C(15)–C(16)–C(17)	104·4 (3)
N(6)–C(5)–C(10)	116·1 (2)	C(16)–C(17)–C(18)	104·2 (3)
C(5)–N(6)–C(11)	123·7 (2)	C(17)–C(18)–N(7)	103·2 (3)
C(5)–N(6)–C(14)	125·6 (2)		

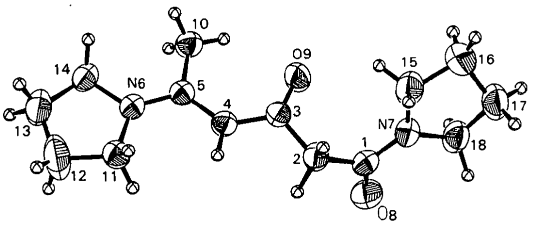

Fig. 1. View of the molecule, (3), showing the numbering scheme and the vibrational ellipsoids (50% probability level).

The best plane through atoms N(7), C(15), C(18), C(1), C(2) and O(8) [planar to within 0·039 (3) Å] is inclined by 77 (1)° to the best plane through atoms N(6), C(11), C(14), C(5), C(10), C(4), C(3), O(9) and C(2) [planar to within 0·022 (3) Å]. This is considerably less than in the analogous compound 3-oxo-5-phenylamino-4-hexenanilide, (4), prepared from aniline and (2) (Briard, Dubourg, Roques, Castillo, Herault & Declercq, 1984). In (4) the best planes through atoms of the phenyl ring, N(6), C(5), C(10), C(4), C(3), O(9), C(2) and atoms of the phenyl ring, N(7), C(1), C(2), O(8) are inclined to one another by 110°. The two compounds also differ in the relative orientation of the C(5)=C(4) substituents. In (4) the aniline group at C(5) is in configuration *Z* (or *cis*) with respect to the carbonyl group at C(3), and the structure is stabilized by two intramolecular hydrogen bonds between the N(6) and N(7) H atoms and the carbonyl O atom at C(3). The molecule will then undergo cyclization by attack of N(6) on C(1) to produce the 2-pyridone. In (3) the pyrrolidine group at C(5) is in configuration *E* (or *trans*) with respect to the carbonyl bond at C(3). A *Z* arrangement is excluded on steric grounds.

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Structure of 5-Methylcytosine Hydrochloride

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Abstract. $C_5H_8N_3O^+.Cl^-$, $M_r = 161·6$, monoclinic, $P2_1/c$, $a = 6·431(1)$, $b = 16·132(2)$, $c = 7·030(1)$ Å, $\beta = 97·33(1)°$, $V = 723·29$ Å^3, $Z = 4$, $D_x = 1·48$, $D_m = 1·49$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1·5418$ Å, $\mu = 0·423$ mm^{-1}, $F(000) = 336$, $T = 295$ K, $R = 0·042$ for 1146 observed reflections with $I > 3\sigma(I)$. The cytosine base is protonated at N(3). The crystal structure is stabilized by hydrogen bonds of the type N(3)–H···Cl and direct electrostatic interactions between Cl and atoms of the base. Molecules related by the *c*-glide are nearly parallel (angular deviation ≃ 17°) and are separated by approximately 3·5 Å. A comparison of the stacking interactions observed in the present structure and in related molecules suggests that 5-methylation of the cytosine base generally results in reduced ring overlap.

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