dimers which crystallize in the space group $P4_32_12$ with $Z = 4$. In addition, the molecules of (2) and floccosin resemble each other with respect to the orientation of the two nearly planar three-ring systems. The inter-atomic distances and angles found for xanthomegnin agree with comparable values reported for floccosin.

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5-[(p-Chlorobenzamido)methylene]-1,3-dimethyl-2,4,6(1H,3H,5H)-pyrimidinetrione

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Abstract. $C_{14}H_{12}ClN_3O_4$, monoclinic, $P2_1/c$, $a = 15.5124$ (11), $b = 7.2375$ (3), $c = 13.3737$ (10) Å, $\beta = 108.69$ (1)°, $V = 1422.3$ (2) Å^3, $Z = 4$, $D_x = 1.495$ Mg m^{-3}, $M_r = 321.72$, $\mu(\text{Cu } K\alpha) = 2.59$ mm^{-1}. The structure was solved by Patterson and Fourier techniques. R and R_w were 0.059 and 0.084 respectively for 2038 observed reflexions. The Cl—C(benzene) distance and the *ipso* angle are 1.741 (2) Å and 122.0 (3)° respectively. There is an intramolecular hydrogen bond [N—H$\cdots$O = 149 (4)°].

Introduction. The title compound is the by-product of a reaction which took place when acylation of the C(5) atom in the nucleus of a pyrimidine was attempted. The structure determination was carried out in order to establish the structural formula (Fig. 1).

Unit-cell dimensions were obtained and refined by a least-squares procedure from the setting angles of 78 reflexions with $\theta \leq 45°$. The data were collected on a four-circle diffractometer in the $\omega/2\theta$ scan mode. Graphite-monochromatized Cu $K\alpha$ radiation was used. 2419 independent reflexions were recorded, 2038 of which were considered as observed $[I > 2\sigma(I)]$. An
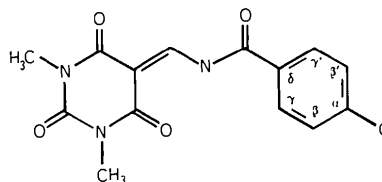

Fig. 1. Structural formula of the title compound.

$C_{14}H_{12}ClN_3O_4$

absorption correction was applied ($\mu = 2.59$ mm^{-1}; crystal size: $0.09 \times 0.24 \times 0.38$ mm) with maximum and minimum transmission factors of 0.802 and 0.536 respectively (ORABS from XRAY 70; Stewart, Kundell & Baldwin, 1970).

The Cl atom was located in a Patterson map and the other non-hydrogen atoms were found in successive Fourier syntheses.

After several cycles of least-squares refinement, a difference synthesis revealed the positions of the H atoms, which were refined isotropically. A weighting scheme (see Table 1) was applied to give no trends in $\langle w\Delta^2 \rangle$ vs $|F_o|$ and $\sin \theta/\lambda$.

The molecule is shown in Fig. 2 (ORTEP, Johnson, 1965) with the numbering system. The residual δR_w full-normal probability plot (Abrahams & Keve, 1971) is shown in Fig. 3 and indicates the consistency of the weighting scheme. DP is defined as $(F_o - F_c)/(\sigma_o^2 + \sigma_c^2)^{1/2}$ and X is the expected DP value for a normal distribution, N_t is the total number of observations (based on F's), R_t is the corresponding interexperimental agreement factor, N is the number of observations in the interval DP, and R is the usual crystallographic residual factor. The equation of the straight line, with e.s.d.'s in parentheses, was obtained through a least-squares fitting. ρ is the correlation coefficient and χ^2 is an estimator of the fitting.*

In Tables 2 and 3 are displayed the atomic coordinates and equivalent isotropic thermal parameters for the non-hydrogen atoms, and the atomic coordinates for the H atoms together with their bond distances.† Table 4 shows bond distances and angles for the heavy atoms.
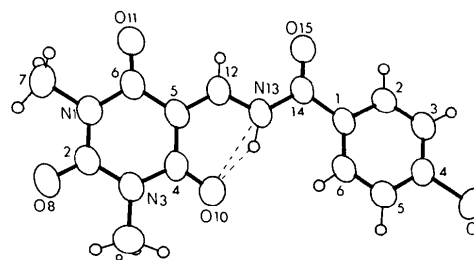
* $p = \mu_{11}/\sigma_x\sigma_y, \chi^2 = \overline{\sigma_y^2(1 - p^2)}, \sigma_x^2 = \mu_{20}, \sigma_y^2 = \mu_{02}, \mu_{ij} = \sum_1^N (DP_n - \overline{DP})^i (X_n - \overline{X})^j$.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36131 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Function and coefficients for the weighting scheme*

$w_T = K\{1/|f(|F_o|)|^2\}\{1/f(\sin \theta/\lambda)\};$ $K = 0.4167;$ $f(|F_o|) = a + b|F_o| + c|F_o|^2$

	a	b	c
$|F_o| \leq 1.0$	0.5102	−0.0561	0.0059
$1.0 < |F_o| \leq 11.0$	0.4178	−0.0459	0.0048
$11.0 < |F_o| \leq 33.0$	0.5516	0.0340	−
$33.0 < |F_o|$	0.2298	0.0142	−

$f(\sin \theta/\lambda) = d$

$\sin \theta/\lambda$ (Å^{-1})	d
0.0 −0.39	0.4432
0.39−0.44	0.9189
0.44−0.60	1.6175

Fig. 2. An ORTEP plot of the molecule showing the atomic numbering.

DP

12

$-3.5 \leq DP \leq 3.0$

$N_t = 2419$ $R_t = 0.0669$

$N = 2407$ $R = 0.0635$

$DP = 0.923(2) X - 0.124(2)$

$\rho = 0.997$

$\chi^2 = 0.006$

Fig. 3. A δR_w full-normal probability plot.

Table 2. *Atomic coordinates* ($\times 10^5$) *and* U_{eq} (Å$^2 \times 10^4$) *for the non-hydrogen atoms*

$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$

	x	y	z	U_{eq}
N(1)	92502 (14)	52111 (37)	27558 (20)	590 (8)
C(2)	93022 (20)	53987 (42)	17541 (25)	588 (10)
N(3)	85490 (16)	49381 (41)	9109 (20)	612 (8)
C(4)	77455 (20)	42749 (44)	10286 (26)	620 (10)
C(5)	77139 (17)	41109 (42)	20986 (23)	581 (10)
C(6)	84949 (19)	45928 (43)	29986 (23)	586 (9)
C(7)	100516 (22)	57136 (65)	36584 (30)	762 (13)
O(8)	99948 (15)	59680 (45)	16167 (21)	813 (10)
C(9)	86082 (29)	51773 (78)	−1524 (29)	841 (16)
O(10)	71053 (16)	38802 (43)	2383 (20)	778 (9)
O(11)	85060 (15)	45164 (43)	39107 (17)	755 (9)
C(12)	69536 (19)	34664 (43)	22920 (26)	592 (10)
N(13)	61845 (16)	30336 (40)	15191 (22)	629 (9)
C(14)	53925 (19)	24299 (45)	17064 (23)	606 (10)
O(15)	53558 (15)	22322 (52)	25827 (19)	852 (10)
C(1')	46073 (15)	20840 (41)	7317 (21)	556 (9)
C(2')	38150 (21)	13987 (52)	8780 (25)	653 (11)
C(3')	30554 (21)	10072 (50)	233 (26)	664 (11)
C(4')	30959 (16)	13084 (40)	−9718 (21)	563 (9)
C(5')	38735 (21)	19592 (47)	−11434 (25)	638 (10)
C(6')	46253 (20)	23463 (50)	−2848 (25)	628 (10)
Cl	21403 (5)	8306 (10)	−20515 (7)	723 (3)

Table 3. Atomic coordinates ($\times 10^3$) for the hydrogen atoms and their bond distances

The H-atom numbering corresponds to that of the atoms to which they are bonded.

	x	y	z	
H(7a)	1028 (5)	442 (11)	418 (6)	1·15 (7) Å
H(7b)	982 (6)	644 (13)	410 (7)	0·94 (10)
H(7c)	1051 (7)	591 (15)	330 (7)	0·98 (12)
H(9a)	795 (5)	531 (9)	−68 (5)	1·04 (6)
H(9b)	860 (5)	404 (11)	−38 (6)	0·88 (8)
H(9c)	916 (4)	581 (8)	−13 (4)	0·96 (7)
H(12)	691 (2)	336 (5)	297 (3)	0·94 (4)
H(13)	632 (3)	320 (6)	87 (4)	0·97 (5)
H(2')	381 (3)	110 (6)	157 (3)	0·95 (5)
H(3')	249 (3)	57 (5)	10 (3)	0·97 (5)
H(5')	394 (4)	220 (8)	−188 (4)	1·04 (6)
H(6')	510 (3)	287 (6)	−46 (3)	0·92 (5)

Table 4. Bond distances (Å) and angles (°)

N(1)—C(2)	1·374 (5)	C(12)—N(13)	1·342 (4)
N(1)—C(6)	1·387 (4)	N(13)—C(14)	1·400 (4)
N(1)—C(7)	1·474 (4)	C(14)—O(15)	1·200 (4)
C(2)—N(3)	1·380 (3)	C(14)—C(1')	1·493 (4)
C(2)—O(8)	1·218 (4)	C(1')—C(2')	1·396 (5)
N(3)—C(4)	1·391 (4)	C(1')—C(6')	1·382 (5)
N(3)—C(9)	1·464 (5)	C(2')—C(3')	1·383 (4)
C(4)—C(5)	1·452 (5)	C(3')—C(4')	1·370 (5)
C(4)—O(10)	1·230 (4)	C(4')—C(5')	1·381 (5)
C(5)—C(6)	1·450 (4)	C(4')—Cl	1·741 (2)
C(5)—C(12)	1·368 (5)	C(5')—C(6')	1·378 (4)
C(6)—O(11)	1·216 (4)		

C(6)—N(1)—C(7)	116·2 (3)	N(1)—C(6)—O(11)	120·6 (3)
C(2)—N(1)—C(7)	118·4 (3)	C(5)—C(12)—N(13)	122·8 (3)
C(2)—N(1)—C(6)	125·3 (3)	C(12)—N(13)—C(14)	123·4 (3)
N(1)—C(2)—O(8)	120·7 (3)	N(13)—C(14)—C(1')	114·4 (3)
N(1)—C(2)—N(3)	118·3 (3)	N(13)—C(14)—O(15)	122·1 (3)
N(3)—C(2)—O(8)	121·0 (3)	O(15)—C(14)—C(1')	123·5 (3)
C(2)—N(3)—C(9)	117·7 (3)	C(14)—C(1')—C(6')	124·6 (3)
C(2)—N(3)—C(4)	123·1 (3)	C(14)—C(1')—C(2')	116·5 (3)
C(4)—N(3)—C(9)	119·2 (3)	C(2')—C(1')—C(6')	118·9 (3)
N(3)—C(4)—O(10)	119·3 (3)	C(1')—C(2')—C(3')	120·8 (3)
N(3)—C(4)—C(5)	117·0 (3)	C(2')—C(3')—C(4')	118·6 (3)
C(5)—C(4)—O(10)	123·7 (3)	C(3')—C(4')—Cl	118·8 (2)
C(4)—C(5)—C(12)	121·2 (3)	C(3')—C(4')—C(5')	122·0 (3)
C(4)—C(5)—C(6)	121·0 (3)	C(5')—C(4')—Cl	119·1 (2)
C(6)—C(5)—C(12)	117·8 (3)	C(4')—C(5')—C(6')	118·8 (3)
N(1)—C(6)—C(5)	115·3 (3)	C(1')—C(6')—C(5')	120·9 (3)
C(5)—C(6)—O(11)	124·1 (3)		

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). A final difference map showed peaks within ±0·28 e Å⁻³.

Discussion. The molecule can be considered as divided into three parts: the pyrimidine ring (plane 1: $\chi^2 = 9·9$), the central chain (plane 2: $\chi^2 = 141·1$), and the benzene nucleus (plane 3: $\chi^2 = 12·2$) with the substituent. The dihedral angles formed by the three planes are 177·3 (6), 176·7 (7) and 1·5 (6)° for planes 1 and 2, 1 and 3, and 2 and 3 respectively.

As far as the central chain is concerned, some kind of delocalization could be considered to contribute significantly to the shortening of C—substituent bonds: C(12)—N(13) and C(5)—C(6) are shorter while C(12)—C(5) is longer than usual (Lide, 1962).

The distortions in the benzene ring have been analysed following the data given by Domenicano, Vaciago & Coulson (1975). We have considered two points of their paper: (a) Table 9 (C—Cl bond lengths and α angles in *para*-substituted chlorobenzenes) and (b) Fig. 3 (plot of the internal angle α *vs* the C—substituent bond length for the phenyl derivatives of second-row elements).

(a) From the references given for compounds with *para* $C(sp^2)$ substituents we found the following ranges: Cl—C [1·745 (5) − 1·728 (6) Å], α [122·2 (5) − 121·0 (2)°], β [119·7 (2) − 117·5 (5)°], γ [121·9 (5) − 119·3 (5)°] and δ [121·0 (5) − 118·2 (3)°] (Domenicano & Vaciago, 1979). In our case, Cl—C = 1·741 (2) Å, α = 122·0 (3), β = 118·3 (3), γ = 120·9 (3) and δ = 118·9 (3)° (see Fig. 1). Of these, the first two results are almost at the extremes of the given ranges.

(b) A straight line (see Fig. 4) has been fitted for the dependence of the angle α on the C—X (benzene) distance (point 1 of the figure is not included in the fitting). The mean deviation from each point to the fitted line is 0·0062, being 0·0192 for the values obtained in the present compound.

Thermal motion has been analysed in terms of the rigid-body **TLS** model (Schomaker & Trueblood, 1968; Filippini & Gramaccioli, 1969). The corrections in distances and angles were not significant in terms of the standard deviations.

There is an intramolecular hydrogen bond which seems to have some influence on the planarity of the central chain and on the stability of the molecule. O(10)···H(13) = 1·75 (5) Å, N(13)—H(13) = 0·97 (5) Å, and N(13)···O(10) = 2·630 (4) Å. The
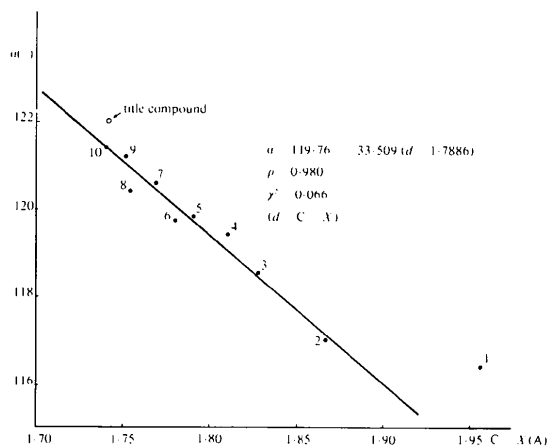

Fig. 4. The fitted straight line showing the dependence of the angle α on the C—X distance.

$C_{14}H_{12}ClN_3O_4$

angles involved are: C(14)—N(13)—H(13) 131 (2), C(12)—N(13)—H(13) 105 (2), N(13)—H(13)—O(10) 149 (4), H(13)—O(10)—C(4) 131 (1)°.

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Structure du (+)-p-Chlorobenzylidène-3,3' Di(bornanone-2) [(+)-Di(bornanone-2 yl-3) p-Chlorophényl Méthane]

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Abstract. $C_{27}H_{35}ClO_2$, orthorhombic, $P2_12_12_1$, $a = 6.835$ (2), $b = 11.932$ (6), $c = 29.039$ (18) Å, $Z = 4$, $d_m = 1.05$, $d_c = 1.19$ Mg m^{-3}. The structure was solved by direct methods with MULTAN 78 and refined by full-matrix least-squares calculations to $R = 0.046$ for 1485 observed reflections. Although four stereoisomers are theoretically possible, depending on the stereochemical course of the reaction, only one is formed, $|\alpha|_D = +174°$ (1.092 g dm^{-3}, dioxane). The structural study shows that the two 2-oxobornyl groups are linked to the exocyclic carbon atom by their endo bonds, which establishes, with certainty, the stereochemical course of the reaction.

Introduction. La réaction de la (+)-bornanone-2 (I) et d'un (−)-arylidène-3 bornanone-2 (II), en milieu basique, suivant la Fig. 1 peut théoriquement donner

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* L'auteur auquel toute correspondance doit être adressée.