

Fig. 1. ORTEP (Johnson, 1965) drawing of FDH.

Discussion. Final atomic parameters are given in Table 1,* interatomic distances and bond angles in Table 2. The atom-labelling scheme is illustrated in Fig. 1 and a stereoview of the molecules is given in Fig. 2. Like fluorescein dipropionate, FDP (Wang, Wang, Peng, He & Wang, 1990), the molecule of (I) has a somewhat T-like shape with the isobenzofuran moiety nearly perpendicular to the xanthene plane. Unlike FDP, each unit cell of (I) contains eight molecules but no solvent molecule (acetone). In addition, FDP gave strong fluorescence on reacting with

* Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54277 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

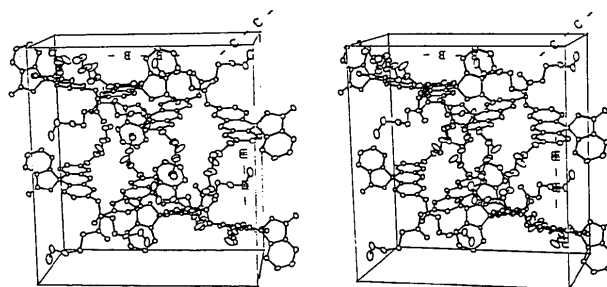


Fig. 2. Stereoview of FDH.

the lipolase within living biocells, but (I) gave only faint fluorescence; the difference is probably due to the fact that (I) contains a larger acyl group than FDP. This result will be discussed subsequently.

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Structure of Methyl (*E*)-4-(3,4-Dimethoxyphenyl)-3-phenyl-1-pyrazoline-3-carboxylate

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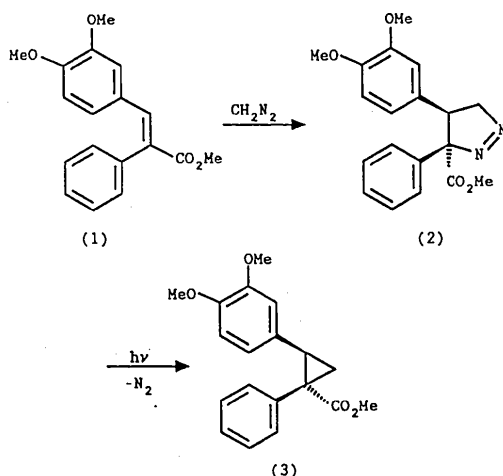
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Abstract. $C_{19}H_{20}N_2O_4$, $M_r = 340.4$, monoclinic, $P2_1/c$, $a = 13.249$ (1), $b = 15.469$ (1), $c = 8.6468$ (9) Å, $\beta = 97.82$ (1)°, $V = 1755.6$ (2) Å³, $Z = 4$, $D_x = 1.29$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu =$

0.76 mm⁻¹, $F(000) = 720$, $T = 291$ (2) K, final $R = 0.034$ for 1733 unique observed reflections. The molecular structure is closely similar to the 3,4-diphenyl analogue [Dewulf, Meunier-Piret, Putzeys & Van Meerssche (1975). *Cryst. Struct. Commun.* **4**, 175–180] confirming the significance of intermolecular contacts in the crystal.

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Introduction. We have been concerned with the synthesis of phenanthrene and 9,10-dihydrophenanthrene derivatives employing intramolecular oxidative cyclization (Halton, Maidment, Officer & Warnes, 1984). In the course of these studies the cyclopropane derivative (3) was required and its preparation was effected as illustrated below. While the addition of diazomethane to stilbene derivatives, *e.g.* (1), usually proceeds with high stereoselectivity (Deleux, Leroy & Weiler, 1973; Verhé & De Kimpe, 1987), the available spectroscopic data did not allow for the unambiguous assignment of configuration to compound (2). We now provide the resulting crystallographic data which shows that compound (2) is methyl (*E*)-4-(3,4-dimethoxyphenyl)-3-phenyl-1-pyrazoline-3-carboxylate formed with high regio- and stereoselectivity.



Experimental. A colourless regular crystal, 0.15 × 0.24 × 27 mm, was mounted on a Hilger & Watts Y290 diffractometer. Cell dimensions were determined using 20 reflections ($18 < \theta < 28^\circ$, nickel-filtered Cu K α radiation) at 291 K. Intensity data were measured by conventional $\theta/2\theta$ scan mode to θ of 57° , at fixed scanning speed (2302 measurements; $h - 14$ to 14 , $k 0$ to 16 , $l 0$ to 9) and the resulting 1733 unique observed reflections (intensities 3.0 times their standard deviations, from counting statistics) were corrected for Lorentz and polarization factors. As transmission coefficients ranged from 0.82 to 0.89, no absorption correction was applied. Three standard reflections with $\pm 2\%$ variation in intensity.

Structure solution was by direct methods, with H atoms located by successive difference Fourier syntheses, and refinement (SHELX76; Sheldrick, 1976) by weighted full-matrix least squares on F minimizing $w\Delta^2$ where $\Delta = |F_o| - |F_c|$, $w = [\sigma(F_o)]^{-2}$ and F_o , F_c are the calculated and observed structure factors. All H atoms were refined with isotropic and non-H-atom anisotropic thermal parameters. No features of

significance were noted in the final difference map, highest peak $0.28 \text{ e } \text{\AA}^{-3}$.

Scattering factors for non-H and H atoms were taken from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). The final cycle converged (average $\Delta/\sigma = 0.1$) with $R = 0.034$, $wR = 0.043$. Program ORTEP (Johnson, 1971) as implemented in the GX (Gilmore, Mallinson & Muir, 1985) package was used to produce a view of the molecule (Fig. 1).

Discussion. Final positional and equivalent isotropic parameters of (2) are shown in Table 1, and bond lengths and bond angles, and selected torsion angles are shown in Tables 2 and 3.* The molecule and numbering schemes are shown in Fig. 1.

The structure consists of independent molecules of (2), with only weak van der Waals intermolecular interactions [closest contact for H...H 2.48 (3) Å]. The presence of the two methoxy substituents at positions 3 and 4 on the phenyl ring has scarcely perturbed the structure from that found in the diphenyl analogue (Dewulf, Meunier-Piret, Putzeys & Van Meerssche, 1975; DIP). The average difference in dihedral angles around the pyrazoline and between it and the methoxy-substituted C(16)...C(21) ring is $0.7 (4)^\circ$ (for 16 angles). Indeed this and the DIP pyrazoline rings are superimposable within experimental error (note that the coordinates presented here are for an inverted configuration relative to DIP). The flap atom [C(4)] is $0.463 (4) \text{ \AA}$ from the pyrazoline envelope plane. All other bond lengths and angles are normal.

The methoxy groups appear to be constrained by intramolecular H...H non-bonding contacts [*e.g.* H(17)...H(23A) 2.31 (3) Å] which are similar to those found in the remainder of the molecule [H(4)...H(21) 2.33 (3) Å]. Atoms C(3) and C(4) are essentially coplanar [$0.034 (2)$, $0.118 (4) \text{ \AA}$ out of plane respectively] with the phenyl rings to which they are bound.

The only differences with the DIP structure concern the conformation of the substituents at C(3) of the pyrazoline ring. Both substituent rings are rotated, about the C(3)—C(10) and C(3)—C(6) bonds, by an average of $11 (1)^\circ$. Comparisons of the angles between the mean planes through the phenyl rings, carboxylate and pyrazoline envelope relative to DIP show that compensation for these twists is by one significant change only: the methoxy-substituted phenyl and methyl carboxylate interplanar angle is $10.3 (1)^\circ$ here compared with 15.8° for DIP. None of

* Lists of structure factors, anisotropic thermal parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54275 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final atomic coordinates ($\times 10^4$) and thermal parameters ($\text{\AA}^2 \times 10^2$)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \cdot a_i \cdot a_j$$

	x	y	z	U
N(1)	6287 (2)	1312 (1)	-2088 (2)	6.92
N(2)	6114 (1)	531 (1)	-1929 (2)	6.15
C(3)	7093 (1)	17 (1)	-1671 (2)	5.00
C(4)	7910 (2)	705 (1)	-1142 (3)	5.57
C(5)	7392 (2)	1503 (2)	-1923 (3)	6.86
C(6)	7257 (2)	-363 (1)	-3263 (3)	5.50
O(7)	8077 (1)	-560 (1)	-3584 (2)	8.37
O(8)	6396 (1)	-477 (1)	-4188 (2)	6.40
C(9)	6465 (3)	-850 (2)	-5711 (3)	7.45
C(10)	7013 (1)	-746 (1)	-583 (2)	4.92
C(11)	6098 (2)	-999 (2)	-143 (3)	6.06
C(12)	6038 (2)	-1707 (2)	818 (3)	7.45
C(13)	6893 (2)	-2171 (2)	1349 (3)	7.40
C(14)	7808 (2)	-1931 (2)	919 (3)	7.55
C(15)	8784 (2)	-1225 (2)	-47 (3)	6.54
C(16)	8184 (1)	829 (1)	603 (2)	5.17
C(17)	7441 (2)	998 (1)	1563 (3)	5.17
C(18)	7702 (1)	1176 (1)	3125 (2)	5.00
C(19)	8728 (2)	1193 (1)	3770 (3)	5.80
C(20)	9461 (2)	1023 (2)	2835 (3)	7.11
C(21)	9181 (2)	841 (2)	1260 (3)	6.50
O(22)	7024 (1)	1344 (1)	4151 (2)	6.17
C(23)	5970 (2)	1394 (2)	3553 (4)	6.81
O(24)	8906 (1)	1401 (1)	5325 (2)	7.91
C(25)	9946 (3)	1452 (4)	6024 (5)	11.21
H(4)	8503 (15)	576 (12)	-1598 (23)	5.5 (6)
H(51)	7567 (18)	1563 (15)	-3015 (30)	8.3 (8)
H(52)	7518 (16)	2029 (16)	-1268 (27)	7.9 (7)
H(91)	6740 (20)	-1443 (19)	-5607 (31)	9.4 (9)
H(92)	7028 (18)	-538 (16)	-6213 (29)	8.3 (8)
H(93)	5733 (28)	-812 (20)	-6251 (41)	12.8 (12)
H(11)	5486 (18)	-699 (14)	-519 (28)	7.7 (7)
H(12)	5440 (20)	-1863 (16)	1087 (30)	9.0 (9)
H(13)	6853 (18)	-2676 (18)	2116 (30)	9.5 (8)
H(14)	8396 (17)	-2261 (15)	1255 (26)	8.4 (8)
H(15)	8522 (17)	-1046 (13)	-355 (25)	6.5 (6)
H(17)	6758 (16)	1020 (12)	1164 (24)	5.7 (6)
H(20)	10169 (19)	1020 (14)	3254 (29)	8.0 (8)
H(21)	9680 (18)	713 (14)	638 (29)	8.4 (8)
H(231)	5705 (18)	844 (17)	3059 (31)	8.7 (8)
H(232)	5864 (17)	1836 (16)	2766 (29)	7.6 (8)
H(233)	5617 (19)	1569 (15)	4446 (33)	9.3 (8)
H(251)	10233 (26)	1953 (21)	5469 (41)	14.2 (14)
H(252)	9908 (23)	1586 (19)	7042 (41)	11.4 (11)
H(253)	10286 (37)	866 (27)	5923 (54)	18.6 (24)

Table 2. Bond distances (\AA) and angles ($^\circ$)

N(1)—N(2)	1.241 (2)	O(22)—C(23)	1.423 (3)
N(2)—C(3)	1.512 (2)	C(19)—O(24)	1.372 (3)
C(3)—C(4)	1.542 (3)	O(24)—C(25)	1.429 (3)
C(4)—C(5)	1.524 (3)	C(4)—H(4)	0.95 (2)
N(1)—C(5)	1.481 (3)	C(5)—H(51)	1.01 (3)
C(3)—C(6)	1.539 (3)	C(5)—H(52)	0.99 (2)
C(3)—C(10)	1.522 (3)	C(9)—H(91)	0.99 (3)
C(4)—C(16)	1.515 (3)	C(9)—H(92)	1.03 (3)
C(6)—O(7)	1.197 (2)	C(9)—H(93)	1.02 (4)
C(6)—O(8)	1.314 (3)	C(11)—H(11)	0.95 (2)
O(8)—C(9)	1.452 (3)	C(12)—H(12)	0.89 (3)
C(10)—C(11)	1.376 (3)	C(13)—H(13)	1.03 (3)
C(11)—C(12)	1.384 (3)	C(14)—H(14)	0.94 (2)
C(12)—C(13)	1.367 (4)	C(15)—H(15)	0.97 (2)
C(13)—C(14)	1.366 (4)	C(17)—H(17)	0.92 (2)
C(14)—C(15)	1.384 (3)	C(20)—H(20)	0.96 (2)
C(15)—C(10)	1.387 (3)	C(21)—H(21)	0.93 (2)
C(16)—C(17)	1.396 (3)	C(23)—H(231)	1.00 (3)
C(17)—C(18)	1.375 (3)	C(23)—H(232)	0.96 (2)
C(18)—C(19)	1.398 (3)	C(23)—H(233)	0.99 (3)
C(19)—C(20)	1.370 (3)	C(25)—H(251)	1.01 (3)
C(20)—C(21)	1.391 (3)	C(25)—H(252)	0.91 (3)
C(21)—C(16)	1.365 (3)	C(25)—H(253)	1.02 (4)
C(18)—O(22)	1.370 (2)		
N(2)—N(1)—C(5)	112.2 (2)	C(10)—C(11)—C(12)	121.1 (2)
N(1)—N(2)—C(3)	111.2 (2)	C(11)—C(12)—C(13)	120.3 (3)
N(2)—C(3)—C(4)	103.5 (2)	C(12)—C(13)—C(14)	119.4 (2)
N(2)—C(3)—C(6)	106.9 (2)	C(13)—C(14)—C(15)	120.7 (3)
N(2)—C(3)—C(10)	111.6 (2)	C(10)—C(15)—C(14)	120.5 (2)
C(4)—C(3)—C(6)	110.5 (2)	C(4)—C(16)—C(17)	121.5 (2)
C(4)—C(3)—C(10)	117.2 (2)	C(4)—C(16)—C(21)	120.3 (2)
C(6)—C(3)—C(10)	106.6 (2)	C(17)—C(16)—C(21)	118.1 (2)
C(3)—C(4)—C(5)	99.9 (2)	C(16)—C(17)—C(18)	121.2 (2)
C(3)—C(4)—C(16)	116.5 (2)	C(17)—C(18)—C(19)	119.7 (2)
C(5)—C(4)—C(16)	111.8 (2)	C(17)—C(18)—O(22)	125.1 (2)
N(1)—C(5)—C(4)	105.1 (2)	C(19)—C(18)—O(22)	115.2 (2)
C(3)—C(6)—O(7)	123.3 (2)	C(18)—C(19)—C(20)	119.4 (2)
C(3)—C(6)—O(8)	112.4 (2)	C(18)—C(19)—O(24)	115.1 (2)
O(7)—C(6)—O(8)	124.4 (2)	C(20)—C(19)—O(24)	125.6 (2)
C(6)—O(8)—C(9)	116.8 (2)	C(19)—C(20)—C(21)	120.0 (2)
C(3)—C(10)—C(11)	122.0 (2)	C(16)—C(21)—C(20)	121.6 (2)
C(3)—C(10)—C(15)	119.9 (2)	C(18)—O(22)—C(23)	118.1 (2)
C(11)—C(10)—C(15)	118.1 (2)	C(14)—O(24)—C(25)	117.1 (2)

Table 3. Selected dihedral angles ($^\circ$)

C(5)—N(1)—N(2)—C(3)	-1.9 (1)	C(10)—C(3)—C(6)—O(8)	92.4 (1)
N(2)—N(1)—C(5)—C(4)	-16.5 (1)	N(2)—C(3)—C(10)—C(11)	10.5 (2)
N(1)—N(2)—C(3)—C(4)	19.3 (1)	N(2)—C(3)—C(10)—C(15)	-171.6 (2)
N(1)—N(2)—C(3)—C(6)	-97.5 (2)	C(4)—C(3)—C(10)—C(11)	129.6 (2)
N(1)—N(2)—C(3)—C(10)	146.1 (2)	C(4)—C(3)—C(10)—C(15)	-52.6 (2)
N(2)—C(3)—C(4)—C(5)	-26.7 (1)	C(6)—C(3)—C(10)—C(11)	-106.0 (2)
N(2)—C(3)—C(4)—C(16)	94.0 (2)	C(6)—C(3)—C(10)—C(15)	71.9 (2)
C(6)—C(3)—C(4)—C(5)	87.5 (2)	C(3)—C(4)—C(5)—N(1)	26.0 (1)
C(6)—C(3)—C(4)—C(16)	-151.8 (2)	C(16)—C(4)—C(5)—N(1)	-97.9 (2)
C(10)—C(3)—C(4)—C(5)	-149.9 (1)	C(3)—C(4)—C(16)—C(17)	-53.8 (1)
C(10)—C(3)—C(4)—C(16)	-29.2 (2)	C(3)—C(4)—C(16)—C(21)	131.1 (2)
N(2)—C(3)—C(6)—O(7)	155.4 (2)	C(5)—C(4)—C(16)—C(17)	60.2 (2)
N(2)—C(3)—C(6)—O(8)	-27.2 (1)	C(5)—C(4)—C(16)—C(21)	-114.9 (2)
C(4)—C(3)—C(6)—O(7)	43.3 (1)	C(3)—C(6)—O(8)—C(9)	-178.8 (2)
C(4)—C(3)—C(6)—O(8)	-139.2 (2)	O(7)—C(6)—O(8)—C(9)	-1.4 (1)
C(10)—C(3)—C(6)—O(7)	-85.0 (1)		

these (minor) differences seem to be in response to inter- or intramolecular interactions and so suggest the order of deviations that should be expected in the crystal structures of similar molecules.

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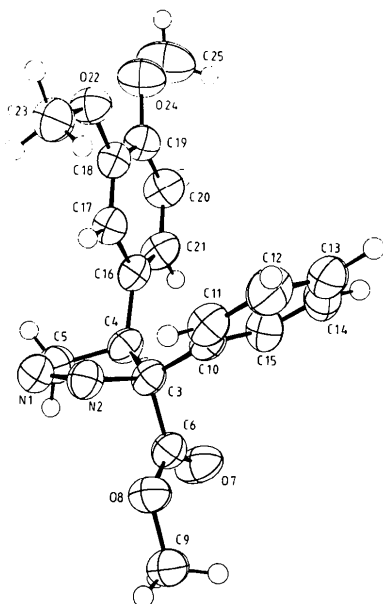


Fig. 1. ORTEP (Johnson, 1971) plot of the molecule

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Structure and ³⁵Cl NQR of (±)-Methyl 4,4,4-Trichloro-3-hydroxybutyrate

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Abstract. C₅H₇Cl₃O₃, *M_r* = 221.47, triclinic, *P* $\bar{1}$, *a* = 10.214 (3), *b* = 9.865 (3), *c* = 9.375 (3) Å, α = 93.21 (1), β = 98.76 (1), γ = 97.38 (1)°, *V* = 923.13 Å³, *Z* = 4, *D_m* = 1.57 Mg m⁻³, *D_x* = 1.593 Mg m⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71069 Å, μ = 0.955 mm⁻¹, *F*(000) = 448, *T* = 296 K, final *R* = 0.041 for 2398 unique reflexions. The title compound crystallizes with two molecules [(I) and (II)] in the asymmetric unit; two molecules of (I) and two molecules of (II), respectively, are connected by hydrogen bonds to form pairs. The ³⁵Cl nuclear quadrupole resonance shows a six-line spectrum with two distinct triplets, differentiated by the temperature behavior of the ³⁵Cl frequencies, $\nu(^{35}\text{Cl}) = f(T)$, 77 ≤ *T* ≤ 330 K.

Introduction. CCl₃ groups have a tendency to undergo an order–disorder transition far below the melting point of the compound considered. This transition can be observed by ³⁵Cl nuclear quadrupole resonance, NQR, but no decision can be made concerning its nature. The freely rotating state has not been observed, either by NQR or by diffraction methods [for a recent discussion of the problem, see Basaran, Dou & Weiss (1991)].

Experimental. Colorless plates of the title compound (Aldrich) were obtained by slow evaporation from an ethanolic solution. *D_m* measured pycnometrically with water as liquid. [For the synthesis and the optical properties of the compound, see Ross (1936).] Crystal size 0.25 × 0.4 × 0.5 mm, Stoe Stadi-4 four-circle diffractometer, graphite(002)-monochromated Mo *K*α radiation, lattice parameters from setting 50 reflexions with 35.0 ≤ 2θ ≤ 38.6°, ω–2θ scan.

Lorentz–polarization corrections, empirical absorption correction, min., max. transmission factors 0.674, 0.765; (sinθ/λ)_{max} = 0.5384 Å⁻¹, –10 ≤ *h* ≤ 10, –10 ≤ *k* ≤ 10, –10 ≤ *l* ≤ 1. Three standard reflexions (222, $\bar{2}\bar{1}3$, $\bar{2}11$) measured every 120 min to monitor the crystal showed about 30% decrease in intensity. 3130 reflexions measured, 2218 independent (unique) reflexions with *F* ≥ 2σ(*F*), *R*_{int} = 0.011. Structure solved by direct methods using *SHELXS86* (Sheldrick, 1986) refined by full-matrix least squares (on *F*) with minimization of $\sum w(|F_o| - |F_c|)^2$, *w* = 1/σ²(*F_o*) (*SHELX76*; Sheldrick, 1976). All H atoms except those from the methyl group of molecule (II) and H(O¹), H(C¹) of molecule (I) were assigned from difference Fourier maps and refined with fixed isotropic temperature factors. 233 free parameters, final *R* = 0.041, *wR* = 0.037 for 2218 reflexions with *F* ≥ 2σ(*F*), (Δ/σ)_{max} = 0.016, (Δ/σ)_{mean} = 0.004, (Δρ)_{max} = +0.32, (Δρ)_{min} = –0.26 e Å⁻³ in the final difference Fourier synthesis. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1* contains the atomic coordinates and the equivalent isotropic thermal parameters *U*_{eq}. Bond lengths and bond angles are given in Table 2.

³⁵Cl NQR spectra were registered with a super-regenerative oscillator-type spectrometer; 2 cm³ sample of polycrystalline material of the title compound. At a few temperatures the ³⁷Cl NQR spec-

* 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 54266 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.