

The pattern of hydrogen bonding exhibited by mefloquine hydrochloride demonstrates that mefloquine should be able to hydrogen bond with molecules of biological importance such as transport proteins or cellular 'effectors' of antimalarial action.

## References

- ALLEN, F. H., KENNARD, O. & TAYLOR, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
- CARTER, O. L., MCPHAIL, A. T. & SIM, G. A. (1967). *J. Chem. Soc. A*, pp. 365–373.
- DOHERTY, R., BENSON, W. R., MAIENTHAL, M. & STEWART, J. MCD. (1978). *J. Pharm. Sci.* **67**, 1698–1700.
- EARLE, D. P., BERLINER, R. W., TAGGART, J. V., WELCH, W. J., ZUBROD, C. G., WISE, N. B., CHALMERS, T. C., GREIF, R. I. & SHANNON, J. A. (1948). *J. Clin. Invest.* **27**, Suppl., 75–79.
- KARLE, I. L. & KARLE, J. (1981). *Proc. Natl Acad. Sci. USA*, **78**, 5938–5941.
- KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **B21**, 579–586.
- KARLE, J. M. & KARLE, I. L. (1989). *Antimicrob. Agents Chemother.* **33**, 1081–1089.
- KASHINO, S. & HAISSO, M. (1983). *Acta Cryst.* **C39**, 310–312.
- OLEKSYN, B. (1978). *Acta Cryst.* **A34**, S-77.
- OLEKSYN, B. (1982). *Acta Cryst.* **B38**, 1832–1834.
- OLEKSYN, B., LEBIODA, L. & CIECHANOWICZ-RUTKOWSKA, M. (1979). *Acta Cryst.* **B35**, 440–444.
- OLEKSYN, B., STADNICKA, K. M. & HODOROWICZ, S. A. (1978). *Acta Cryst.* **B34**, 811–816.
- PAYNE, D. (1987). *Parasitol. Today*, **3**, 241–246.
- PNIEWSKA, B. & SUSZKO-PURZYCKA, A. (1989). *Acta Cryst.* **C45**, 638–642.
- SHELDRIK, G. M. (1980). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Germany.
- SUSZKO-PURZYCKA, A., LIPINSKA, T., PIOTROWSKA, E. & OLEKSYN, B. (1985). *Acta Cryst.* **C41**, 977–980.
- SWEENEY, T. R. (1981). *Med. Res. Rev.* **1**, 281–301.
- TAGGART, J. V., EARLE, D. P., BERLINER, R. W., ZUBROD, C. G., WELCH, W. J., WISE, N. B., SCHROEDER, E. F., LONDON, I. M. & SHANNON, J. A. (1948). *J. Clin. Invest.* **27**, Suppl., 80–86.
- WYLER, D. J. (1983). *N. Engl. J. Med.* **308**, 875–878.

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## Structure of Fluorescein Dihexanoate\*

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**Abstract.** 3-Oxospiro[isobenzofuran-1(3*H*),9'-[9*H*]-xanthene]-3',6'-diyl hexanoate, C<sub>32</sub>H<sub>32</sub>O<sub>7</sub>, *M<sub>r</sub>* = 528.28, orthorhombic, *Pbca*, *a* = 21.864 (3), *b* = 19.222 (3), *c* = 13.117 (3) Å, *V* = 5512.5 Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.274 g cm<sup>-3</sup>, λ(Mo *Kα*) = 0.7107 Å, μ(Mo *Kα*) = 0.834 cm<sup>-1</sup>, *T* = 298 K, *F*(000) = 2240, *R* = 0.06661 for 2086 unique observed reflections. Unlike fluorescein dipropionate, the title compound did not form an inclusion compound with the solvent acetone. The diffraction analysis reveals that both OH groups in fluorescein participated in esterification.

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**Introduction.** This paper describes a continuation of our investigations on fluorescent materials. The structures of 3,6-dichlorofluoran (Wang, Ren, Wang, He, Wang, Jin & Zhang, 1989) and fluorescein dipropionate (Wang, Wang, Peng, He & Wang, 1990) and the antitumor activity of the former have previously been reported. In this article, we describe the structure of an ester of fluorescein with a larger aliphatic acid, hexanoic acid.

**Experimental.** The title compound (I) was prepared by refluxing fluorescein (II) with hexanoic acid anhydride (in molar ratio 1:5) in the presence of pyridine and was crystallized from acetone. The colorless crystals of (I) are stable in the atmosphere, m.p. 377–378 K. The infrared spectrum of (I) showed

Table 1. Final positional and equivalent isotropic thermal parameters of non-H atoms of FDH with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub> (Å <sup>2</sup> )
O(1)	0.1691 (2)	-0.1436 (2)	0.4660 (3)	5.18 (9)
C(1A)	0.1574 (2)	-0.0916 (3)	0.3947 (4)	4.0 (1)
C(2A)	0.1534 (2)	-0.0229 (2)	0.4184 (4)	3.6 (1)
C(1)	0.1667 (2)	0.0037 (2)	0.5252 (4)	3.7 (1)
C(2B)	0.1859 (2)	-0.0544 (3)	0.5952 (4)	3.6 (1)
C(1B)	0.1856 (2)	-0.1230 (3)	0.5633 (4)	4.0 (1)
C(3A)	0.1395 (3)	0.0236 (3)	0.3396 (4)	4.6 (1)
C(4A)	0.1316 (3)	-0.0000 (3)	0.2412 (4)	5.2 (1)
C(5A)	0.1378 (2)	-0.0697 (3)	0.2215 (4)	4.5 (1)
C(6A)	0.1502 (2)	-0.1175 (3)	0.2962 (4)	4.7 (1)
C(3B)	0.2042 (2)	-0.0408 (3)	0.6951 (4)	4.3 (1)
C(4B)	0.2217 (2)	-0.0934 (3)	0.7603 (2)	4.7 (1)
C(5B)	0.2209 (2)	-0.1612 (3)	0.7240 (4)	4.5 (1)
C(6B)	0.2031 (3)	-0.1777 (3)	0.6271 (4)	4.6 (1)
O(1A)	0.1348 (2)	-0.0899 (2)	0.1185 (3)	6.3 (1)
O(2A)	0.0609 (3)	-0.1614 (4)	0.1415 (4)	15.1 (2)
C(7A)	0.0971 (3)	-0.1367 (4)	0.0850 (5)	7.4 (2)
C(8A)	0.1033 (3)	-0.1532 (4)	-0.0234 (4)	6.9 (2)
C(9A)	0.0464 (4)	-0.1575 (5)	-0.0824 (5)	9.6 (2)
C(10A)	0.0517 (3)	-0.1682 (4)	-0.1943 (5)	7.1 (2)
C(11A)	-0.0009 (4)	-0.1547 (5)	-0.2548 (6)	8.9 (2)
C(12A)	0.0034 (4)	-0.1672 (4)	-0.3632 (6)	8.9 (2)
O(1B)	0.2352 (2)	-0.2164 (2)	0.7888 (3)	5.14 (9)
O(2B)	0.3344 (2)	-0.1920 (2)	0.7812 (4)	6.8 (1)
C(7B)	0.2951 (2)	-0.2280 (3)	0.8111 (4)	4.6 (1)
C(8B)	0.3003 (3)	-0.2922 (3)	0.8761 (5)	5.7 (1)
C(9B)	0.3630 (3)	-0.3041 (3)	0.9206 (5)	6.3 (2)
C(10B)	0.3631 (3)	-0.3653 (4)	0.9943 (6)	8.4 (2)
C(11B)	0.3539 (5)	-0.4325 (4)	0.9470 (6)	10.5 (3)
C(12B)	0.4057 (7)	-0.4566 (5)	0.8888 (9)	15.5 (4)
C(2)	0.1152 (2)	0.0477 (4)	0.5690 (4)	3.8 (1)
C(7)	0.1358 (2)	0.1142 (3)	0.5860 (4)	4.1 (1)
C(8)	0.2006 (3)	0.1179 (3)	0.5532 (4)	4.7 (1)
O(3)	0.2344 (2)	0.1659 (2)	0.5533 (4)	7.0 (1)
O(2)	0.2178 (2)	0.0543 (2)	0.5169 (3)	4.37 (8)
O(3)	0.0564 (2)	0.0283 (3)	0.5943 (4)	4.5 (1)
C(4)	0.0188 (3)	0.0788 (3)	0.6356 (5)	5.5 (1)
C(5)	0.0400 (3)	0.1462 (3)	0.6525 (5)	6.2 (2)
C(6)	0.0984 (3)	0.1644 (3)	0.6277 (4)	5.3 (1)

Table 2. Bond lengths (Å) and bond angles (°) in FDH

C(1)—C(2A)	1.519 (4)	C(1)—C(2B)	1.505 (4)
C(2A)—C(1A)	1.361 (4)	C(2B)—C(1B)	1.384 (4)
C(1A)—O(1)	1.391 (4)	C(1B)—O(1)	1.384 (4)
C(2A)—C(3A)	1.400 (4)	C(2B)—C(3B)	1.394 (4)
C(3A)—C(4A)	1.379 (5)	C(3B)—C(4B)	1.379 (5)
C(4A)—C(5A)	1.370 (5)	C(4B)—C(5B)	1.388 (5)
C(5A)—C(6A)	1.370 (5)	C(5B)—C(6B)	1.366 (5)
C(6A)—C(1A)	1.394 (5)	C(6B)—C(1B)	1.396 (5)
C(5A)—O(1A)	1.407 (4)	C(5B)—O(1B)	1.395 (4)
O(1A)—C(7A)	1.296 (5)	O(1B)—C(7B)	1.361 (4)
C(7A)—O(2A)	1.183 (5)	C(7B)—O(2B)	1.171 (4)
C(7A)—C(8A)	1.464 (6)	C(7B)—C(8B)	1.503 (5)
C(8A)—C(9A)	1.467 (6)	C(8B)—C(9B)	1.508 (5)
C(9A)—C(10A)	1.487 (6)	C(9B)—C(10B)	1.523 (6)
C(10A)—C(11A)	1.421 (6)	C(10B)—C(11B)	1.446 (7)
C(11A)—C(12A)	1.447 (7)	C(11B)—C(12B)	1.442 (10)
C(1)—C(2)	1.522 (4)	C(2)—C(7)	1.374 (5)
C(7)—C(8)	1.483 (5)	C(8)—O(3)	1.182 (4)
C(8)—O(2)	1.365 (4)	O(2)—C(1)	1.485 (3)
C(2)—C(3)	1.379 (4)	C(3)—C(4)	1.384 (5)
C(4)—C(5)	1.393 (5)	C(5)—C(6)	1.366 (6)
C(6)—C(7)	1.378 (5)		
C(1)—O(2)—C(8)	110.7 (2)	O(2)—C(8)—C(7)	108.7 (3)
O(2)—C(1)—C(2)	102.7 (2)	C(2)—C(7)—C(8)	108.1 (3)
C(1)—C(2)—C(7)	109.6 (3)	O(3)—C(8)—O(2)	121.8 (4)
O(3)—C(8)—C(7)	129.4 (4)	C(2)—C(3)—C(4)	117.3 (3)
C(3)—C(2)—C(7)	121.2 (3)	C(4)—C(5)—C(6)	120.8 (4)
C(3)—C(4)—C(5)	121.1 (4)	C(6)—C(7)—C(2)	121.5 (3)
C(5)—C(6)—C(7)	118.0 (4)	C(6)—C(7)—C(8)	130.4 (3)
C(1)—C(2)—C(3)	129.1 (5)	C(2)—C(1)—C(2B)	112.8 (3)
C(2)—C(1)—C(2A)	113.2 (3)	O(2)—C(1)—C(2B)	108.7 (2)
O(2)—C(1)—C(2A)	107.3 (2)	C(2A)—C(1)—C(2B)	111.5 (3)
C(1A)—O(1)—C(1B)	117.6 (3)	O(1)—C(1B)—C(2B)	123.5 (3)
O(1)—C(1A)—C(2A)	123.8 (3)	C(1B)—C(2B)—C(1)	121.4 (3)
C(1A)—C(2A)—C(1)	121.6 (3)	C(6B)—C(1B)—C(2B)	122.4 (3)
C(6A)—C(1A)—C(2A)	123.4 (3)	C(1B)—C(2B)—C(3B)	117.6 (3)
C(1A)—C(2A)—C(3A)	117.8 (3)	C(2B)—C(3B)—C(4B)	121.7 (3)
C(2A)—C(3A)—C(4A)	120.5 (3)	C(3B)—C(4B)—C(5B)	118.2 (3)
C(3A)—C(4A)—C(5A)	119.1 (3)	C(4B)—C(5B)—C(6B)	122.7 (3)
C(4A)—C(5A)—C(6A)	122.7 (3)	C(5B)—C(6B)—C(1B)	117.5 (3)
C(5A)—C(6A)—C(1A)	116.5 (3)	C(1)—C(2B)—C(3B)	121.0 (3)
C(1)—C(2A)—C(3A)	120.5 (3)	O(1)—C(1B)—C(6B)	114.1 (3)
O(1)—C(1A)—C(6A)	112.8 (3)	C(4B)—C(5B)—O(1B)	120.1 (3)
C(4A)—C(5A)—O(1A)	116.5 (3)	C(6B)—C(5B)—O(1B)	117.1 (3)
C(6A)—C(5A)—O(1A)	120.7 (3)	C(5B)—O(1B)—C(7B)	118.1 (3)
C(5A)—O(1A)—C(7A)	123.1 (3)	O(1B)—C(7B)—C(8B)	109.2 (3)
O(1A)—C(7A)—C(8A)	114.9 (4)	C(7B)—C(8B)—C(9B)	114.3 (3)
C(7A)—C(8A)—C(9A)	116.5 (4)	C(8B)—C(9B)—C(10B)	111.3 (4)
C(8A)—C(9A)—C(10A)	117.5 (4)	C(9B)—C(10B)—C(11B)	114.7 (4)
C(9A)—C(10A)—C(11A)	117.5 (5)	C(10B)—C(11B)—C(12B)	113.9 (6)
C(10A)—C(11A)—C(12A)	117.7 (5)	O(1B)—C(7B)—O(2B)	122.5 (3)
O(1A)—C(7A)—O(2A)	119.4 (4)	O(2B)—C(7B)—C(8B)	128.3 (4)
O(2A)—C(7A)—C(8A)	125.7 (5)		

that the OH group absorption band (3400 cm<sup>-1</sup>) of the parent compound (II) had disappeared, whereas absorption bands of 1370 and 1420 cm<sup>-1</sup> (aliphatic C—H) could be seen clearly, signifying complete esterification of two OH groups in (II). MS study indicated the presence of two ester acyl groups and a molecular weight of 528 (Yuan & Wang, 1985).

A colorless approximately cubic crystal of (I) (0.37 × 0.30 × 0.30 mm) was studied using an Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo Kα radiation; cell parameters by least-squares fitting of diffractometer setting angles for 25 reflections (20 < 2θ < 32°); ω-2θ scan; speed 2 to 10° min<sup>-1</sup> in θ; width = (0.4 + 0.35tanθ)°, (sinθ/λ)<sub>max</sub> = 0.594 Å<sup>-1</sup>, *h*: 0 to 26, *k*: 0 to 22, *l*: 0 to 15. Three standard reflections monitored every hour, intensity variation 0.3%. Decay correction; Lorentz and polarization correction and ψ semi-empirical absorption correction, transmission factors 0.964 to 0.990. 5390 independent reflections, 2092 observed

reflections with *I* ≥ 3σ(*I*). Structure solved by direct methods and subsequent difference Fourier techniques. Refinement by full-matrix least squares on *F*, anisotropic *U*<sub>ij</sub>'s for all non-H atoms. H atoms located from Δρ map, 32 H atoms included in structure-factor calculations but not refined. The final cycle of refinement included 352 variable parameters, final *R* = 0.066, *wR* = 0.065 and *S* = 2.878 for 2086 observed reflections, with weight *w* = 4*F*<sup>2</sup>/σ<sup>2</sup>(*F*<sub>o</sub>)<sup>2</sup>, (Δ/σ)<sub>max</sub> = 0.02, Δρ < 0.62 e Å<sup>-3</sup>. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974). All calculations performed on a PDP 11/44 computer using the *SDP* package (Frenz, 1985).

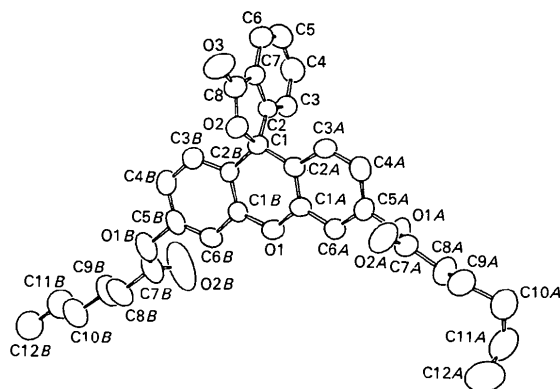


Fig. 1. ORTEP (Johnson, 1965) drawing of FDH.

**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.

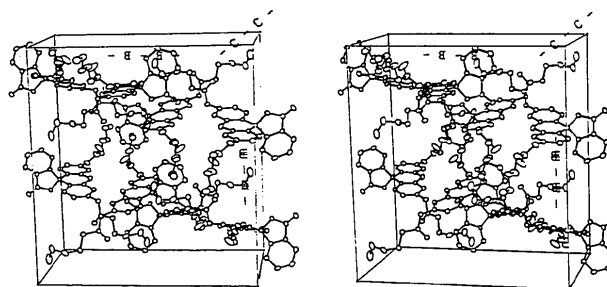


Fig. 2. Stereoview of FDH.

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.

#### References

- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71, 148. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- FRENZ, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- WANG, L., REN, Y., WANG, Q., HE, F., WANG, Q., JIN, D. & ZHANG, P. (1989). *Chin. Sci. Bull.* **34**(21), 1789.
- WANG, L., WANG, X., PENG, Z., HE, F. & WANG, Q. (1990). *Acta Cryst.* **C46**, 1676–1678.
- YUAN, X. Z. & WANG, L. (1985). *J. Chin. Mass Spectrom. Soc.* **6**(2), 1.

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

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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) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.29$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu =$

$0.76$  mm<sup>-1</sup>,  $F(000) = 720$ ,  $T = 291$  (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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