Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
C(1)	−0.7484 (1)	0.4269 (2)	−0.3402 (1)	4.7 (1)
C(11)	−0.7706 (1)	0.5256 (2)	−0.4123 (2)	5.6 (1)
C(12)	−0.8001 (1)	0.3248 (3)	−0.3881 (2)	6.8 (2)
C(2)	−0.6730 (1)	0.3861 (2)	−0.2962 (1)	4.8 (1)
C(21)	−0.6679 (2)	0.2891 (4)	−0.3538 (2)	9.8 (2)
C(22)	−0.6304 (2)	0.4922 (4)	−0.2858 (3)	9.2 (2)
C(3)	−0.6501 (1)	0.3388 (2)	−0.1977 (2)	4.3 (1)
C(31)	−0.5742 (1)	0.3175 (2)	−0.1278 (2)	4.2 (1)
C(32)	−0.5454 (1)	0.2011 (2)	−0.1293 (1)	4.0 (1)
C(33)	−0.4878 (1)	0.1941 (2)	−0.1294 (2)	4.8 (1)
C(34)	−0.4622 (1)	0.0851 (2)	−0.1304 (2)	5.3 (1)
C(35)	−0.4915 (1)	−0.0197 (2)	−0.1288 (1)	4.6 (1)
C(36)	−0.5487 (1)	−0.0145 (2)	−0.1283 (1)	5.0 (1)
C(37)	−0.5750 (1)	0.0953 (2)	−0.1286 (1)	4.8 (1)
C(38)	−0.4872 (1)	−0.2329 (2)	−0.1218 (2)	6.3 (2)
C(39)	−0.4400 (2)	−0.3292 (3)	−0.1101 (2)	7.6 (2)
C(4)	−0.6823 (1)	0.4236 (3)	−0.1655 (1)	6.9 (1)
C(41)	−0.7948 (2)	0.5745 (4)	−0.5622 (2)	8.5 (2)
C(5)	−0.7460 (1)	0.4711 (3)	−0.2542 (2)	7.0 (1)
N	−0.5392 (1)	0.4027 (2)	−0.0707 (1)	4.7 (1)
O(1)	−0.4607 (1)	−0.1223 (1)	−0.1269 (1)	6.0 (1)
O(2)	−0.4687 (1)	0.3777 (1)	−0.0067 (1)	5.8 (1)
O(3)	−0.7808 (1)	0.6272 (2)	−0.4026 (2)	9.4 (1)
O(4)	−0.7777 (1)	0.4869 (2)	−0.4909 (1)	6.2 (1)

Table 2. *Selected geometric parameters* (Å, °)

C(34)—C(35)	1.380 (3)	C(1)—C(5)	1.537 (3)
C(36)—C(35)	1.388 (3)	C(2)—C(1)	1.580 (2)
O(1)—C(35)	1.359 (3)	C(12)—C(1)	1.533 (3)
C(33)—C(34)	1.374 (3)	C(11)—C(1)	1.512 (3)
C(32)—C(33)	1.396 (3)	C(21)—C(2)	1.525 (3)
C(37)—C(32)	1.388 (3)	C(22)—C(2)	1.513 (4)
C(31)—C(32)	1.484 (3)	C(39)—C(38)	1.496 (3)
C(36)—C(37)	1.384 (3)	O(1)—C(38)	1.421 (3)
C(3)—C(31)	1.518 (2)	O(3)—C(11)	1.197 (3)
N—C(31)	1.274 (2)	O(4)—C(11)	1.338 (3)
C(4)—C(3)	1.522 (3)	O(4)—C(41)	1.435 (3)
C(2)—C(3)	1.547 (3)	O(2)—N	1.417 (2)
C(5)—C(4)	1.502 (3)		
C(36)—C(35)—C(34)	119.3 (2)	C(12)—C(1)—C(5)	108.8 (2)
O(1)—C(35)—C(34)	116.1 (2)	C(12)—C(1)—C(2)	113.2 (2)
O(1)—C(35)—C(36)	124.7 (2)	C(11)—C(1)—C(5)	110.9 (2)
C(33)—C(34)—C(35)	121.1 (2)	C(11)—C(1)—C(2)	111.4 (2)
C(32)—C(33)—C(34)	120.5 (2)	C(11)—C(1)—C(12)	108.3 (2)
C(37)—C(32)—C(33)	118.1 (2)	C(1)—C(2)—C(3)	101.6 (1)
C(31)—C(32)—C(33)	121.8 (2)	C(21)—C(2)—C(3)	111.2 (2)
C(31)—C(32)—C(37)	120.1 (2)	C(21)—C(2)—C(1)	113.6 (2)
C(36)—C(37)—C(32)	121.4 (2)	C(22)—C(2)—C(3)	110.9 (2)
C(37)—C(36)—C(35)	119.7 (2)	C(22)—C(2)—C(1)	110.2 (2)
C(3)—C(31)—C(32)	119.1 (2)	C(22)—C(2)—C(21)	109.2 (3)
N—C(31)—C(32)	124.3 (2)	O(1)—C(38)—C(39)	107.5 (2)
N—C(31)—C(3)	116.6 (2)	O(3)—C(11)—C(1)	125.6 (2)
C(4)—C(3)—C(31)	116.5 (2)	O(4)—C(11)—C(1)	111.9 (2)
C(2)—C(3)—C(31)	114.9 (2)	O(4)—C(11)—O(3)	122.5 (3)
C(2)—C(3)—C(4)	104.9 (2)	O(2)—N—C(31)	114.2 (2)
C(5)—C(4)—C(3)	106.7 (2)	C(38)—O(1)—C(35)	118.5 (2)
C(1)—C(5)—C(4)	108.1 (2)	C(41)—O(4)—C(11)	116.9 (2)
C(2)—C(1)—C(5)	104.2 (2)		

Program used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Refinement was by full-matrix least-squares methods (*SHELX*76; Sheldrick, 1976). Program used for calculation of dihedral angles: *XANADU* (Roberts & Sheldrick, 1975).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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6′-Diethylamino-2′-nitrospiro[isobenzofuran-1(3*H*),9′-[9*H*]xanthen]-3-one

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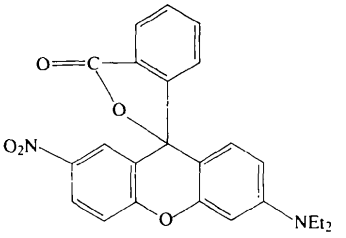
Abstract

The title molecule, $C_{24}H_{20}N_2O_5$, is composed of two parts, namely, a butterfly-like xanthene moiety and an almost planar isobenzofuran fragment. The isobenzofuran plane is almost perpendicular to the two xanthene planes. The C—O bond length in the five-membered lactone ring is 1.487 (5) Å, which is longer than the usual lactone C—O single-bond length.

Comment

Since Meyer & Hoffmeyer (1892) first synthesized fluoran, several derivatives have been prepared and

studies of their spectral properties undertaken (Kramer, Klapper & Miller, 1968; Gronowska, Dabkowska & Walerys, 1979; Matsuoka, Uedam & Kitao, 1982; Wang, Ren, Yun, He & Wang, 1990). We have synthesized the title compound (I) and determined its crystal structure as, up to now, only a few structures of fluoran derivatives have been reported.



(I)

In the title molecule, the xanthene group has a butterfly-like conformation in which the two planar parts form a dihedral angle of 6.6 (5)°. The isobenzofuran moiety is almost planar and is perpendicular to each of the xanthene planes, the dihedral angles being 87.97 (11) and 93.86 (14)°. The structural characteristics of the present molecule are in good agreement with the results reported by Wang *et al.* (1989) and Osborn & Rogers (1975).

The C—O bond length in the five-membered lactone ring is 1.487 (5) Å, which is longer than the normal lactone C—O single-bond value, *e.g.* 1.463 (7) Å (Cameron, Jochem & Linden, 1989) and shorter than the value of 1.525 (3) Å found in fluorescein (Osborn & Rogers, 1975). When reacted with acidic material such as acetic acid the C—O bond in the lactone ring ruptures to give the fluorescent form of the molecule and its colour simultaneously changes to red.

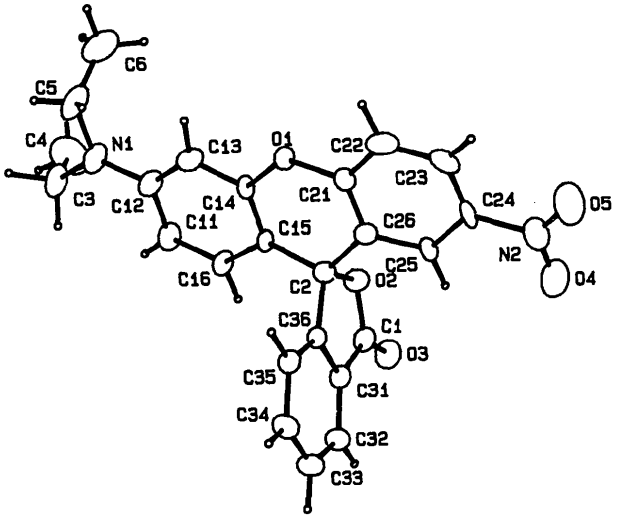


Fig. 1. Perspective drawing of the title compound with the atomic numbering scheme. The displacement ellipsoids have been scaled to 30% probability.

Experimental

A mixture of 2′-carboxy-4-diethylamino-2-hydroxybenzophenone and 4-nitrophenol (molar ratio 1:1) was heated in concentrated sulfuric acid for 3 h at 403 K to yield the title compound. The product was recrystallized from ethanol.

Crystal data

$C_{24}H_{20}N_2O_5$
$M_r = 416.44$
Triclinic
$P\bar{1}$
$a = 8.342$ (3) Å
$b = 9.861$ (2) Å
$c = 12.735$ (2) Å
$\alpha = 80.41$ (2)°
$\beta = 79.91$ (2)°
$\gamma = 80.96$ (2)°
$V = 1008.1$ (5) Å^3
$Z = 2$
$D_x = 1.372$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 0.091$ mm^{-1}
$T = 295$ K
Prism
0.4 × 0.3 × 0.3 mm
Light yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2022 measured reflections
1728 independent reflections
1262 observed reflections [$|F_o| > 3.0\sigma(|F_o|)$]
$R_{int} = 0.012$
$\theta_{max} = 25°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 14$
2 standard reflections
frequency: 60 min
intensity decay: 0.1%

Refinement

Refinement on F
$R = 0.035$
$wR = 0.034$
$S = 0.721$
1262 reflections
280 parameters
All H-atom parameters refined
Unit weights applied
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.141$ e Å^{-3}
$\Delta\rho_{min} = -0.120$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O1	0.6912 (3)	0.4355 (2)	0.1180 (2)	3.58 (8)
O2	0.9686 (3)	0.7396 (2)	0.1105 (3)	3.69 (8)
O3	1.0858 (3)	0.9290 (3)	0.1116 (3)	4.62 (9)
O4	0.9293 (5)	0.8466 (4)	−0.2853 (4)	8.5 (3)
O5	0.8371 (6)	0.6974 (4)	−0.3576 (3)	9.4 (2)
N1	0.6553 (5)	0.3107 (4)	0.4952 (3)	5.0 (1)
N2	0.8688 (5)	0.7414 (4)	−0.2812 (4)	6.3 (1)
C1	0.9622 (4)	0.8780 (4)	0.1164 (4)	3.4 (1)
C2	0.8009 (4)	0.7010 (4)	0.1196 (4)	2.9 (1)
C3	0.6807 (6)	0.3298 (5)	0.6017 (5)	5.7 (2)
C4	0.8439 (7)	0.2726 (7)	0.6266 (5)	8.8 (2)
C5	0.5944 (6)	0.1815 (5)	0.4879 (5)	6.6 (2)

C6	0.7247 (7)	0.0684 (5)	0.4574 (6)	8.4 (2)
C11	0.7541 (5)	0.5274 (4)	0.4101 (4)	4.2 (1)
C12	0.6955 (5)	0.4011 (4)	0.4035 (4)	4.0 (1)
C13	0.6772 (5)	0.3772 (4)	0.3037 (4)	3.8 (1)
C14	0.7136 (4)	0.4712 (4)	0.2146 (4)	2.7 (1)
C15	0.7696 (4)	0.5973 (4)	0.2162 (4)	2.5 (1)
C16	0.7844 (5)	0.6190 (4)	0.3190 (4)	3.4 (1)
C21	0.7351 (5)	0.5168 (4)	0.0224 (4)	3.4 (1)
C22	0.7178 (5)	0.4660 (4)	−0.0686 (4)	5.1 (1)
C23	0.7610 (6)	0.5395 (4)	−0.1661 (4)	5.1 (1)
C24	0.8232 (5)	0.6625 (4)	−0.1748 (4)	3.8 (1)
C25	0.8397 (5)	0.7120 (4)	−0.0827 (4)	3.1 (1)
C26	0.7940 (4)	0.6424 (4)	0.0193 (4)	3.2 (1)
C31	0.7887 (4)	0.9397 (4)	0.1275 (4)	2.9 (1)
C32	0.7218 (5)	1.0746 (4)	0.1372 (4)	3.6 (1)
C33	0.5532 (5)	1.1042 (4)	0.1469 (4)	4.1 (1)
C34	0.4564 (4)	1.0016 (4)	0.1482 (4)	3.7 (1)
C35	0.5250 (4)	0.8678 (4)	0.1372 (4)	3.0 (1)
C36	0.6935 (4)	0.8383 (3)	0.1275 (4)	2.5 (1)

Table 2. *Selected geometric parameters* (Å, °)

O1—C14	1.385 (7)	C11—C16	1.358 (7)
O1—C21	1.367 (8)	C12—C13	1.369 (9)
O2—C1	1.372 (5)	C13—C14	1.363 (8)
O2—C2	1.487 (5)	C14—C15	1.400 (8)
O3—C1	1.204 (4)	C15—C16	1.390 (7)
O4—N2	1.213 (8)	C21—C22	1.37 (1)
O5—N2	1.213 (9)	C21—C26	1.395 (8)
N1—C3	1.456 (7)	C22—C23	1.350 (9)
N1—C5	1.465 (7)	C23—C24	1.374 (9)
N1—C12	1.372 (8)	C24—C25	1.379 (9)
N2—C24	1.466 (9)	C25—C26	1.385 (8)
C1—C31	1.471 (5)	C31—C32	1.376 (5)
C2—C15	1.476 (7)	C31—C36	1.371 (5)
C2—C26	1.499 (8)	C32—C33	1.379 (6)
C2—C36	1.510 (6)	C33—C34	1.386 (6)
C3—C4	1.456 (7)	C34—C35	1.372 (5)
C5—C6	1.481 (8)	C35—C36	1.378 (5)
C11—C12	1.427 (8)		
C14—O1—C21	120.9 (8)	C2—C15—C14	123.5 (7)
C1—O2—C2	111.0 (3)	C2—C15—C16	123.1 (7)
C3—N1—C5	116.3 (6)	C14—C15—C16	113.3 (7)
C3—N1—C12	123.5 (7)	C11—C16—C15	124.9 (7)
C5—N1—C12	120.0 (7)	O1—C21—C22	115.6 (7)
O4—N2—O5	125.7 (5)	O1—C21—C26	121.2 (9)
O4—N2—C24	117.7 (9)	C22—C21—C26	123.1 (4)
O5—N2—C24	116.5 (4)	C21—C22—C23	119.1 (9)
O2—C1—O3	121.0 (4)	C22—C23—C24	120.7 (8)
O2—C1—C31	107.9 (3)	N2—C24—C23	120.0 (9)
O3—C1—C31	131.1 (4)	N2—C24—C25	120.5 (9)
O2—C2—C15	109.5 (5)	C23—C24—C25	119.5 (4)
O2—C2—C26	107.3 (4)	C24—C25—C26	122.2 (9)
O2—C2—C36	102.4 (3)	C2—C26—C21	122.4 (8)
C15—C2—C26	110.9 (5)	C2—C26—C25	122.2 (7)
C15—C2—C36	113.1 (5)	C21—C26—C25	115.4 (9)
C26—C2—C36	113.1 (5)	C1—C31—C32	129.1 (4)
N1—C3—C4	113.7 (6)	C1—C31—C36	108.8 (3)
N1—C5—C6	114.3 (5)	C32—C31—C36	122.1 (4)
C12—C11—C16	119.2 (7)	C31—C32—C33	117.0 (4)
N1—C12···C11	120.2 (8)	C32—C33—C34	121.1 (4)
N1—C12—C13	122.2 (8)	C33—C34—C35	121.3 (4)
C11—C12—C13	117.5 (9)	C34—C35—C36	117.5 (4)
C12—C13—C14	120.8 (8)	C2—C36—C31	109.8 (3)
O1—C14—C13	115.7 (7)	C2—C36—C35	129.2 (3)
O1—C14—C15	120.1 (8)	C31—C36—C35	121.0 (3)
C13—C14—C15	124.3 (7)		

The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). All H atoms were found in $\Delta\rho$ maps. The structure was refined by full-matrix least-squares calculations with anisotropic displacement factors for non-H atoms and isotropic displacement factors for H atoms. Calculations were carried out on a VAX 3100 computer using *MolEN* (Fair, 1990).

This work was supported by the Key Discipline Fund of Tianjin Higher Education, People's Republic of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and least-squares-planes data have been deposited with the IUCr (Reference: AB1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Phenyl 2-Fluorobenzoate, Phenyl 4-Fluorobenzoate and Phenyl Benzoate

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Abstract

On comparing the crystal structures of the title compounds, two isomers of $C_{13}H_9FO_2$, and $C_{13}H_{10}O_2$, it becomes clear that the differences in the molecular arrangements and conformations of the fluorinated phenyl benzoates compared with phenyl benzoate (PB) depend on the position of the F atom pendent on the aromatic nucleus of PB, as an F atom at C(2) does not affect either the molecular arrangement or conformation as much as an F atom at C(4).