

Table 1. Fractional positional parameters and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^2$) with e.s.d.'s in parentheses

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
C(10)	-0.0264 (2)	0.5384 (2)	0.2673 (2)	3.85 (6)
C(9)	-0.0570 (2)	0.3455 (2)	0.2038 (3)	4.23 (6)
O(1)	0.0623 (1)	0.2387 (2)	0.1403 (2)	5.45 (5)
C(4)	0.1357 (2)	0.6262 (2)	0.2646 (2)	3.75 (6)
C(5)	-0.1560 (2)	0.6354 (3)	0.3287 (3)	4.80 (7)
C(3)	0.2482 (2)	0.5166 (2)	0.1974 (3)	4.79 (7)
C(8)	-0.2076 (2)	0.2508 (3)	0.1973 (3)	5.24 (8)
C(7)	-0.3311 (2)	0.3510 (3)	0.2567 (3)	5.90 (8)
O(21)	0.3136 (2)	0.2130 (2)	0.0663 (3)	8.11 (8)
C(41)	0.1696 (2)	0.8341 (2)	0.3348 (3)	4.45 (7)
C(6)	-0.3066 (2)	0.5428 (3)	0.3228 (3)	5.74 (8)
C(2)	0.2167 (2)	0.3170 (3)	0.1298 (3)	5.36 (8)
C(42)	0.3415 (2)	0.9132 (3)	0.3299 (4)	4.97 (9)

Table 2. Bond lengths (\AA) and bond angles ($^\circ$) with e.s.d.'s in parentheses

C(10)—C(9)	1.398 (2)	C(10)—C(4)	1.459 (2)
C(10)—C(5)	1.407 (2)	C(9)—O(1)	1.377 (2)
C(9)—C(8)	1.390 (2)	O(1)—C(2)	1.376 (2)
C(4)—C(3)	1.343 (2)	C(4)—C(41)	1.507 (2)
C(5)—C(6)	1.385 (3)	C(3)—C(2)	1.446 (2)
C(8)—C(7)	1.377 (3)	C(7)—C(6)	1.389 (3)
O(21)—C(2)	1.207 (2)	C(41)—C(42)	1.520 (3)
C(9)—C(10)—C(4)	118.7 (2)	C(9)—C(10)—C(5)	117.1 (2)
C(4)—C(10)—C(5)	124.2 (2)	C(10)—C(9)—O(1)	121.3 (1)
C(10)—C(9)—C(8)	122.4 (2)	O(1)—C(9)—C(8)	116.3 (2)
C(9)—O(1)—C(2)	121.6 (1)	C(10)—C(4)—C(3)	117.9 (2)
C(10)—C(4)—C(41)	119.0 (2)	C(3)—C(4)—C(41)	123.1 (2)
C(10)—C(5)—C(6)	121.1 (2)	C(4)—C(3)—C(2)	123.5 (2)
C(9)—C(8)—C(7)	118.7 (2)	C(8)—C(7)—C(6)	120.9 (2)
C(4)—C(41)—C(42)	115.3 (2)	C(5)—C(6)—C(7)	119.8 (2)
O(1)—C(2)—C(3)	117.1 (2)	O(1)—C(2)—O(21)	117.1 (2)
C(3)—C(2)—O(21)	125.9 (2)		

Table 1 presents fractional positional and thermal parameters and Table 2 lists bond distances and angles.*

Related literature. The structure of the title compound has been determined in the course of studies on the behaviour of 3-bromocoumarin derivatives reacting with organometallic compounds (Alberola, Calvo, González-Ortega, Vicente, García-Granda & van der Maelen, 1991). The complete molecule is essentially planar with a slight deviation for the ethyl group.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, distances and angles involving H atoms, least-squares-planes data and principal torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55017 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 3-Benzyloxy-5-(benzyloxymethyl)-5H-furan-2-one

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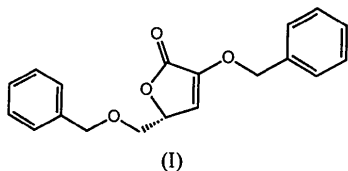
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Abstract. $C_{19}H_{18}O_4$, $M_r = 310.35$, monoclinic, $P2_1/c$, $a = 9.123$ (3), $b = 15.727$ (3), $c = 11.916$ (3) \AA , $\beta = 106.53$ (2) $^\circ$, $V = 1639.1$ (7) \AA^3 , $Z = 4$, $D_x = 1.258$ g cm^{-3} , Mo $K\alpha$ radiation (graphite monochro-

mated), $\lambda = 0.71069$ \AA , $\mu = 0.819$ cm^{-1} , $F(000) = 656$, $T = 295$ (1) K, 2269 unique reflections, 1445 with $I > 2\sigma(I)$, final $R = 0.069$ ($wR = 0.065$). The side chains are arranged to give the molecule an

essentially linear arrangement. The planes of the phenyl rings of the benzyloxy and the benzyloxymethyl groups make angles of 8.1 (4) and 105.2 (5) $^\circ$ respectively with the remaining atoms of these groups, which are otherwise essentially planar.

Experimental. Colourless crystals of $C_{19}H_{18}O_4$ (I) were obtained from hexane/ethyl acetate solution. A crystal of dimensions $0.23 \times 0.28 \times 0.58$ mm was chosen for data collection on a Nicolet R3 diffractometer; crystals weakly diffracting with broad reflections. Lattice parameters were determined from 20 reflections with $2\theta = 25\text{--}30^\circ$. Intensities were collected for $2\theta < 46^\circ$, h 0 to 10, $k - 1$ to 17, $l - 13$ to 13, using Wyckoff ω scans, scan width 0.90° at $2.0\text{--}19.3^\circ \text{min}^{-1}$. Three standard reflections measured every 150 reflections showed negligible variation in intensity. Lorentz and polarization corrections, but no corrections for absorption were applied. Of 2748 reflections measured, 2269 were unique ($R_{\text{int}} = 0.021$) and 1455 were considered observed with $I > 2\sigma(I)$.



The structure was solved by direct methods (Sheldrick, 1986) which revealed positions for all of the non-H atoms. H atoms were placed in idealized positions ($C\text{--}H = 1.00 \text{ \AA}$) and constrained to ride on the atom to which they were bonded. Anisotropic refinement (on F) of the non-H atoms and refinement of isotropic temperature factors for the H atoms employed a full-matrix least-squares method (Sheldrick, 1976), which minimized $\sum w(|F_o| - |F_c|)^2$ where $w = [\sigma^2(F_o) + 0.00018|F_o|^2]^{-1}$. The refinement converged at $R = 0.069$, $wR = 0.065$, $S = 2.41$, for 222 parameters, $(\Delta/\sigma)_{\text{max}} = 0.001$, $(\Delta\rho)_{\text{max}} = 0.33$, $(\Delta\rho)_{\text{min}} = -0.22 \text{ e \AA}^{-3}$. Scattering factors for neutral atoms were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV) and were corrected for the real part of anomalous dispersion. Calculations performed using *SHELXS86* (Sheldrick, 1986) and *SHELX76* (Sheldrick, 1976). Atomic positional parameters are given in Table 1, and Table 2 lists bond lengths and angles.* Fig. 1

* Lists of anisotropic thermal parameters, H-atom coordinates, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55020 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU0403]

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors (\AA^2) for (I) with *e.s.d.*'s in parentheses

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	0.8131 (6)	0.6938 (3)	0.4115 (4)	0.071 (2)
C(2)	0.8795 (5)	0.7787 (3)	0.4194 (4)	0.067 (2)
C(3)	0.8422 (6)	0.8141 (3)	0.3161 (4)	0.081 (2)
C(4)	0.7532 (6)	0.7524 (3)	0.2268 (4)	0.084 (2)
C(5)	0.6017 (6)	0.7819 (3)	0.1582 (4)	0.095 (3)
C(6)	1.0219 (6)	0.8905 (3)	0.5280 (4)	0.075 (2)
C(7)	1.1105 (7)	0.9140 (3)	0.6494 (4)	0.065 (2)
C(8)	1.0352 (6)	0.9334 (3)	0.7306 (4)	0.079 (2)
C(9)	1.1154 (7)	0.9570 (3)	0.8439 (5)	0.090 (3)
C(10)	1.2703 (8)	0.9621 (3)	0.8752 (5)	0.086 (3)
C(11)	1.3475 (7)	0.9416 (4)	0.7960 (5)	0.097 (3)
C(12)	1.2660 (7)	0.9188 (3)	0.6838 (5)	0.087 (3)
C(13)	0.4116 (7)	0.7473 (4)	-0.0130 (4)	0.107 (3)
C(14)	0.3587 (6)	0.6811 (3)	-0.1059 (4)	0.069 (2)
C(15)	0.2418 (6)	0.7005 (4)	-0.2061 (5)	0.081 (2)
C(16)	0.1900 (6)	0.6426 (4)	-0.2935 (5)	0.086 (3)
C(17)	0.2546 (7)	0.5639 (4)	-0.2816 (5)	0.081 (3)
C(18)	0.3684 (6)	0.5426 (4)	-0.1840 (4)	0.080 (2)
C(19)	0.4221 (6)	0.6004 (3)	-0.0966 (4)	0.075 (2)
O(1)	0.8234 (4)	0.6404 (2)	0.4846 (3)	0.097 (2)
O(2)	0.7362 (4)	0.6788 (2)	0.2987 (3)	0.086 (2)
O(3)	0.9651 (3)	0.8060 (2)	0.5262 (2)	0.072 (1)
O(4)	0.5409 (4)	0.7193 (2)	0.0705 (3)	0.095 (2)

Table 2. Bond lengths (\AA) and bond angles ($^\circ$) for (I) with *e.s.d.*'s in parentheses

C(1)—C(2)	1.458 (7)	C(8)—C(9)	1.392 (8)
C(1)—O(1)	1.194 (6)	C(9)—C(10)	1.357 (8)
C(1)—O(2)	1.348 (6)	C(10)—C(11)	1.368 (8)
C(2)—C(3)	1.304 (7)	C(11)—C(12)	1.379 (8)
C(2)—O(3)	1.360 (6)	C(13)—C(14)	1.495 (8)
C(3)—C(4)	1.496 (7)	C(13)—O(4)	1.381 (7)
C(4)—C(5)	1.466 (8)	C(14)—C(15)	1.390 (8)
C(4)—O(2)	1.475 (6)	C(14)—C(19)	1.386 (7)
C(5)—O(4)	1.429 (7)	C(15)—C(16)	1.363 (8)
C(6)—C(7)	1.489 (7)	C(16)—C(17)	1.360 (9)
C(6)—O(3)	1.425 (6)	C(17)—C(18)	1.362 (8)
C(7)—C(8)	1.370 (7)	C(18)—C(19)	1.364 (8)
C(7)—C(12)	1.362 (8)		
C(2)—C(1)—O(1)	130.7 (5)	C(8)—C(9)—C(10)	120.1 (5)
C(2)—C(1)—O(2)	108.2 (4)	C(9)—C(10)—C(11)	119.8 (6)
O(1)—C(1)—O(2)	121.0 (5)	C(10)—C(11)—C(12)	119.3 (6)
C(1)—C(2)—C(3)	109.8 (4)	C(7)—C(12)—C(11)	122.3 (5)
C(1)—C(2)—O(3)	117.8 (4)	C(14)—C(13)—O(4)	109.9 (5)
C(3)—C(2)—O(3)	132.4 (5)	C(13)—C(14)—C(15)	119.4 (5)
C(2)—C(3)—C(4)	109.7 (4)	C(13)—C(14)—C(19)	122.6 (5)
C(3)—C(4)—C(5)	115.3 (4)	C(15)—C(14)—C(19)	118.0 (5)
C(3)—C(4)—O(2)	102.8 (4)	C(14)—C(15)—C(16)	121.6 (5)
C(5)—C(4)—O(2)	109.5 (4)	C(15)—C(16)—C(17)	118.8 (6)
C(4)—C(5)—O(4)	107.4 (4)	C(16)—C(17)—C(18)	121.1 (6)
C(7)—C(6)—O(3)	109.9 (4)	C(17)—C(18)—C(19)	120.5 (5)
C(6)—C(7)—C(8)	119.9 (5)	C(14)—C(19)—C(18)	119.9 (5)
C(6)—C(7)—C(12)	122.5 (5)	C(1)—O(2)—C(4)	109.4 (4)
C(8)—C(7)—C(12)	117.6 (5)	C(2)—O(3)—C(6)	114.7 (4)
C(7)—C(8)—C(9)	120.9 (5)	C(5)—O(4)—C(13)	112.4 (4)

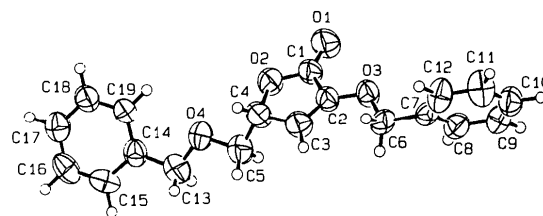


Fig. 1. The structure of $C_{19}H_{18}O_4$ (50% probability thermal ellipsoids).

(Johnson, 1976) shows the structure of (I) and the atom-numbering scheme used.

Related literature. The structure of (I) was determined during the course of developing efficient synthetic approaches to all stereoisomers of 2-(hydroxymethyl)pyrrolidine-3,4-diols, which have important biological properties, especially in the stimulation of the immune response (Meng & Hesse, 1991).

Professor M. Hesse and Mr Q. Meng are thanked for providing the crystals.

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Structure of 3,4-Diethylcanthine-5,6-dione

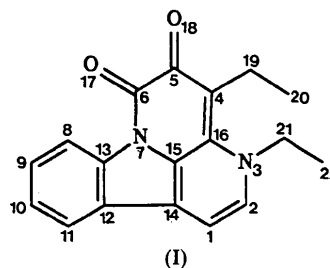
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Abstract. 3,4-Diethylindolo[3,2,1-*de*][1,5]naphthyridine-5,6-dione, C₁₈H₁₆N₂O₂, *M_r* = 292.34, triclinic, *P* $\bar{1}$, *a* = 8.4118 (2), *b* = 11.9934 (4), *c* = 7.9901 (4) Å, α = 83.493 (3), β = 114.946 (3), γ = 101.598 (3)°, *V* = 715.61 (5) Å³, *Z* = 2, *D_x* = 1.356 g cm⁻³, $\lambda(\text{Cu } K\alpha)$ = 1.54178 Å, μ = 6.8 cm⁻¹, *F*(000) = 308, *T* = 293 K, *R* = 0.039 and *wR* = 0.045 for 2690 unique observed reflections. The canthine-5,6-dione skeleton is planar (r.m.s. = 0.031 Å and χ^2 = 1.06 for 16 atoms). The side chains are oriented so that the terminal C—C bonds of the ethyl groups are perpendicular to the plane of the canthine-5,6-dione with one group above and the other below the plane [the torsion angles are $\tau_1 = \text{C}(5)\text{—C}(4)\text{—C}(19)\text{—C}(20) = 89.2$ (2) and $\tau_2 = \text{C}(16)\text{—N}(3)\text{—C}(21)\text{—C}(22) = -88.6$ (2)°]. In the crystal, the molecules that are related by the center of symmetry stack in head-to-tail pairs with a distance between the planes of the aromatic rings of adjacent molecules of 3.384 Å. Owing to the lack of hydrogen-bond donors in the crystal, there are no hydrogen-bond interactions.

eters and orientation matrix were determined by least-squares refinement of the angular positions of 25 reflections with $35 < \theta < 50^\circ$. During the data collection the intensities of three reflections (116, 045, 132) were measured every 2000 s of X-ray exposure time; these reflections showed no significant variation in intensity. Intensities for 3073 reflections were collected [$h - 10 \rightarrow 10$, $k 0 \rightarrow 15$, $l - 10 \rightarrow 10$, maximum $(\sin\theta/\lambda) = 0.6265 \text{ \AA}^{-1}$], of which 2928 reflections were unique (*R*_{int} = 0.044) and 2690 reflections with $I \geq 2.5\sigma(I)$ were regarded as observed and included in the refinement. Lorentz and polarization corrections were applied and absorption correction was not necessary.



The structure was solved by direct methods with *SHELXS86* (Sheldrick, 1986) and refined on *F* by full-matrix least-squares techniques with *XRAY76* (Stewart, 1976). Most of the H atoms were located in difference Fourier syntheses and the remaining H atoms were placed in calculated idealized positions. The parameters varied in the final refinement cycles consisted of the coordinates of all the atoms, the

Experimental. The title compound was provided by Dr Janos Fischer of Gedeon Richter Ltd, Budapest, Hungary. A single crystal of dimensions 0.36 × 0.37 × 0.14 mm was obtained by slow evaporation from dichloromethane. X-ray intensity data were collected at room temperature on an Enraf–Nonius CAD-4F automated diffractometer with Ni-filtered Cu *K*α radiation, using an $\omega/2\theta$ scan mode. The cell param-

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