Structure of 4-Ethylcoumarin

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Abstract. 4-Ethyl-2*H*-1-benzopyran-2-one, $C_{11}H_{10}O_2$, $M_r = 174.20$, triclinic, $P\bar{1}$, $a = 8.854\,(2)$, $b = 7.677\,(2)$, $c = 7.061\,(2)$ Å, $\alpha = 107.91\,(3)$, $\beta = 77.25\,(3)$, $\gamma = 101.40\,(2)°$, $V = 441.0\,(2)$ Å^3, $Z = 2$, $D_x = 1.31$ Mg m^{-3}, Mo $K\alpha$ radiation (graphite-crystal monochromator), $\lambda = 0.71073$ Å, $\mu = 0.084$ mm^{-1}, $F(000) = 184$, $T = 293$ K, final conventional $R = 0.044$ for 1196 observed reflections and 158 variables. The complete molecule is essentially planar with a slight deviation for the ethyl group. The torsion angles C(3)—C(4)—C(41)—C(42) and C(10)—C(4)—C(41)—C(42) are $-1.5\,(3)$ and $179.0\,(2)°$, respectively.

Experimental. Data were collected with a colourless crystal, $0.33 \times 0.20 \times 0.10$ mm, using an Enraf–Nonius CAD-4 single-crystal diffractometer with Mo $K\alpha$ radiation (graphite-crystal monochromated). Unit-cell dimensions were determined from the angular settings of 25 reflections with $0 < \theta < 15°$. Space group $P\bar{1}$ was derived from the structure determination. 5106 reflections were measured ($h - 12\rightarrow12$, $k - 10\rightarrow10$, $l - 9\rightarrow9$; $0 < \theta < 30°$), using ω–2θ scan technique with a variable scan speed and a maximum scan time of 60 s per reflection. Intensity was checked by monitoring three standard reflections every 60 min. Final drift corrections were between 0.97 and 1.35. On all reflections, profile analysis was performed (Lehmann & Larsen, 1974; Grant & Gabe, 1978); empirical absorption correction was applied using ψ scans (North, Phillips & Mathews, 1968), correction factors ranging from 0.69 to 0.99. Friedel pairs were averaged, $R_{int} = (I - \langle I\rangle)/\Sigma I = 0.022$; 2553 reflections were unique and 1196 observed with $I > 3\sigma(I)$. Lorentz and polarization corrections were applied and data reduced to $|F_o|$ values. The structure was solved by direct methods using the program *SHELXS*86 (Sheldrick, 1985) and

Fourier synthesis. Isotropic least-squares refinement, using *SHELX*76 (Sheldrick, 1976), converged to $R = 0.108$. At this stage an additional empirical absorption correction was applied (Walker & Stuart, 1983), resulting in a further decrease of R to 0.086. The maximum and minimum absorption correction factors were 1.18 and 0.82, respectively. Further anisotropic refinements followed by a difference Fourier synthesis allowed location of all the H atoms.

Positional parameters and anisotropic thermal parameters of the non-H atoms were refined. All H atoms were refined isotropically with individual thermal parameters. The final conventional agreement factors were $R = 0.044$ and $wR = 0.047$ for the 1196 observed reflections and 158 variables. The function minimized was $\Sigma w(F_o - F_c)^2$, $w = 1/[\sigma^2(F_o) + 0.00040F_o^2]$ with $\sigma(F_o)$ from counting statistics. The maximum shift to e.s.d. ratio in the last full-matrix least-squares cycle was less than 0.02. The final difference Fourier map showed no peaks higher than 0.15 or deeper than -0.21 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Fig. 1. was made with *PLUTO* (Motherwell, 1976). Geometrical calculations were made with *PARST* (Nardelli, 1983). All calculations were made on a MicroVAX 3300 computer at the Scientific Computer Center of the University of Oviedo.
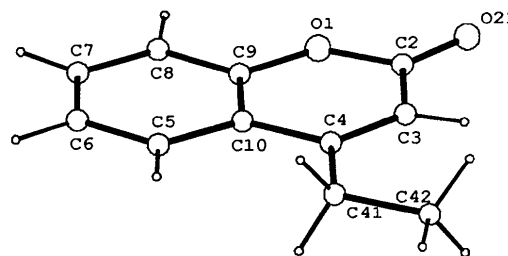

Fig. 1. *PLUTO* (Motherwell, 1976) diagram showing a view of the molecule and the atomic numbering scheme.

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

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	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* (Å) *and bond angles* (°) *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.

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* 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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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) Å, $\beta = 106.53$ (2)°, $V = 1639.1$ (7) Å^3, $Z = 4$, $D_x = 1.258$ g cm^{-3}, Mo $K\alpha$ radiation (graphite monochromated), $\lambda = 0.71069$ Å, $\mu = 0.819$ 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