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3-(2-Methoxyphenyl)-1H-2-benzopyran-1-one

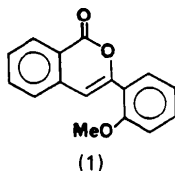
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Abstract. $C_{16}H_{12}O_3$, $M_r = 252.3$, orthorhombic, $Pna2_1$, $a = 6.633$ (3), $b = 13.367$ (2), $c = 14.056$ (2) Å, $V = 1246.3$ (9) Å³, $Z = 4$, $D_x = 1.344$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 7.17$ cm⁻¹, $F(000) = 528$, $T = 296$ K, $R = 0.027$ for 2337 observations (of 2508 unique data). The average deviation from planarity is 0.013 (1) Å with a maximum of 0.028 (1) Å for the fused-rings system, and 0.003 (1) Å with a maximum of 0.005 (1) Å for the methoxyphenyl ring. The dihedral angle between the two systems is 4.7 (3)°. The methoxyphenyl ring is pushed away from the vinyl proton and towards the endocyclic O atom; the bond angles are 130.13 (9) and 110.13 (8)°, respectively.

Experimental. Colorless crystals of (1), m.p. 398–399 K, were isolated by two successive recrystallizations in methanol from the crude product obtained from the coupling of 2-iodoanisole and copper(I) methyl (2-benzoate)ethynide in pyridine at reflux temperature (Stephens & Castro, 1963). Crystal size



0.10 × 0.25 × 0.68 mm, space group from systematic absences $0kl$ with $k+l$ odd, $h0l$ with h odd and successful refinement of a noncentrosymmetric model, cell dimensions from setting angles of 25 reflections having $20 < \theta < 25^\circ$. Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω - 2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 90 s, scan rates varied 0.61–3.30° min⁻¹. One quadrant of data having $2 < \theta < 75^\circ$, $0 \leq h \leq 8$, $0 \leq k \leq 16$, $-17 \leq l \leq 17$ measured. Data corrected for background, Lorentz, polarization and absorption effects. Absorption correc-

tions were based on ψ scans, with a minimum relative transmission coefficient of 89.86%. Standard reflections 200 and 020 displayed no decline in intensity during the experiment. Equivalent data merged, $R_{\text{int}} = 0.016$, to yield 2508 unique data, 2337 observed with $I > 3\sigma(I)$. Structure solved by direct methods, using a data set previously obtained from a lower-quality crystal with Mo $K\alpha$ radiation, employing *MULTAN82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The structure was refined using the Cu $K\alpha$ data by full-matrix least squares based upon F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using *Enraf–Nonius SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located by ΔF and refined isotropically. One reflection (171) was given zero weight on the basis of extremely poor agreement with F_c and with F_o for the 171 reflection. An attempt to determine the direction of the polar axis by refinement of the structure inverted through the origin was inconclusive. Final $R = 0.027$ (0.031 for all data), $wR = 0.035$, $S = 1.918$ for 220 variables. Maximum shift 0.03 σ in the final cycle, max. residual density 0.24 e Å⁻³, min. -0.17 e Å⁻³, extinction coefficient $g = 5.2(2) \times 10^{-6}$ where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c . Coordinates are given in Table 1; bond distances, angles and torsion angles are given in Table 2.† The molecule is illustrated in Fig. 1, and a packing diagram is shown in Fig. 2.

Related literature. Structure of the dimethyl derivative of lateropyrone: Bushnell, Li & Poulton (1984). The title compound shows the same distorted bond angles

† Tables of H-atom coordinates, distances and angles involving H atoms, anisotropic thermal parameters, structure-factor amplitudes and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51686 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation: $\frac{1}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33}]$.

	x	y	z	$B_{eq}(\text{\AA}^2)$
O(1)	-0.0470 (2)	0.11003 (8)	1.000	4.99 (2)
O(2)	0.1754 (1)	0.02678 (7)	0.91535 (6)	3.74 (2)
O(3)	0.4665 (1)	-0.11988 (7)	0.68907 (7)	4.15 (2)
C(1)	0.0003 (2)	0.08132 (9)	0.92169 (9)	3.48 (2)
C(2)	-0.1087 (2)	0.09944 (8)	0.83333 (9)	3.06 (2)
C(3)	-0.2862 (2)	0.15555 (9)	0.8351 (1)	3.71 (2)
C(4)	-0.3863 (2)	0.17497 (9)	0.7514 (1)	4.09 (3)
C(5)	-0.3100 (2)	0.1389 (1)	0.6660 (1)	4.39 (3)
C(6)	-0.1375 (2)	0.0814 (1)	0.6637 (1)	4.05 (3)
C(7)	-0.0328 (2)	0.06077 (8)	0.74808 (9)	3.17 (2)
C(8)	0.1506 (2)	0.00309 (9)	0.74936 (9)	3.33 (2)
C(9)	0.2503 (2)	-0.01244 (8)	0.83097 (9)	3.06 (2)
C(10)	0.4404 (2)	-0.06672 (8)	0.84904 (8)	3.08 (2)
C(11)	0.5221 (2)	-0.0664 (1)	0.94077 (9)	3.69 (2)
C(12)	0.6982 (2)	-0.1163 (1)	0.9626 (1)	4.20 (3)
C(13)	0.7999 (2)	-0.1675 (1)	0.8924 (1)	4.41 (3)
C(14)	0.7252 (2)	-0.16863 (9)	0.8006 (1)	4.09 (3)
C(15)	0.5474 (2)	-0.11904 (9)	0.77802 (9)	3.40 (2)
C(16)	0.5709 (3)	-0.1713 (1)	0.6148 (1)	5.09 (3)

Table 2. *Bond distances (Å), angles (°) and selected torsion angles (°)*

O(1)—C(1)	1.207 (1)	C(6)—C(7)	1.402 (1)
O(2)—C(1)	1.374 (1)	C(7)—C(8)	1.441 (1)
O(2)—C(9)	1.389 (1)	C(8)—C(9)	1.340 (1)
O(3)—C(15)	1.361 (1)	C(9)—C(10)	1.477 (1)
O(3)—C(16)	1.429 (1)	C(10)—C(11)	1.399 (1)
C(1)—C(2)	1.457 (1)	C(10)—C(15)	1.410 (1)
C(2)—C(3)	1.397 (1)	C(11)—C(12)	1.380 (2)
C(2)—C(7)	1.399 (1)	C(12)—C(13)	1.378 (2)
C(3)—C(4)	1.376 (2)	C(13)—C(14)	1.382 (2)
C(4)—C(5)	1.388 (2)	C(14)—C(15)	1.389 (1)
C(5)—C(6)	1.379 (2)		
C(1)—O(2)—C(9)	123.93 (7)	C(6)—C(7)—C(8)	122.28 (9)
C(15)—O(3)—C(16)	118.95 (9)	C(7)—C(8)—C(9)	120.68 (9)
O(1)—C(1)—O(2)	116.58 (9)	O(2)—C(9)—C(8)	119.73 (8)
O(1)—C(1)—C(2)	126.54 (9)	O(2)—C(9)—C(10)	110.13 (8)
O(2)—C(1)—C(2)	116.88 (8)	C(8)—C(9)—C(10)	130.13 (9)
C(1)—C(2)—C(3)	119.50 (9)	C(9)—C(10)—C(11)	119.22 (8)
C(1)—C(2)—C(7)	119.37 (8)	C(9)—C(10)—C(15)	123.48 (8)
C(3)—C(2)—C(7)	121.14 (9)	C(11)—C(10)—C(15)	117.30 (9)
C(2)—C(3)—C(4)	119.5 (1)	C(10)—C(11)—C(12)	122.2 (1)
C(3)—C(4)—C(5)	119.8 (1)	C(11)—C(12)—C(13)	119.6 (1)
C(4)—C(5)—C(6)	121.1 (1)	C(12)—C(13)—C(14)	119.9 (1)
C(5)—C(6)—C(7)	120.0 (1)	C(13)—C(14)—C(15)	120.8 (1)
C(2)—C(7)—C(6)	118.31 (9)	O(3)—C(15)—C(10)	117.12 (8)
C(2)—C(7)—C(8)	119.40 (9)	O(3)—C(15)—C(14)	122.73 (9)
C(10)—C(15)—C(14)	120.13 (9)		
C(9)—O(2)—C(1)—O(1)	179.46 (11)	C(5)—C(6)—C(7)—C(8)	-178.48 (12)
C(9)—O(2)—C(1)—C(2)	-1.0 (2)	C(2)—C(7)—C(8)—C(9)	-1.0 (2)
C(1)—O(2)—C(9)—C(8)	0.6 (2)	C(6)—C(7)—C(8)—C(9)	177.83 (12)
C(1)—O(2)—C(9)—C(10)	180.0 (2)	C(7)—C(8)—C(9)—O(2)	0.5 (2)
C(16)—O(3)—C(15)—C(10)	179.47 (11)	C(7)—C(8)—C(9)—C(10)	-178.74 (11)
C(16)—O(3)—C(15)—C(14)	-1.9 (2)	O(2)—C(9)—C(10)—C(11)	-3.9 (2)
O(1)—C(1)—C(2)—C(3)	0.3 (2)	O(2)—C(9)—C(10)—C(15)	176.74 (10)
O(1)—C(1)—C(2)—C(7)	179.91 (14)	C(8)—C(9)—C(10)—C(11)	175.45 (12)
O(2)—C(1)—C(2)—C(3)	-179.16 (10)	C(8)—C(9)—C(10)—C(15)	-4.0 (2)
O(2)—C(1)—C(2)—C(7)	0.5 (2)	C(9)—C(10)—C(11)—C(12)	179.44 (12)
C(1)—C(2)—C(3)—C(4)	178.25 (11)	C(15)—C(10)—C(11)—C(12)	-1.1 (2)
C(7)—C(2)—C(3)—C(4)	-1.4 (2)	C(9)—C(10)—C(15)—O(3)	-1.1 (2)
C(1)—C(2)—C(7)—C(6)	-178.38 (11)	C(9)—C(10)—C(15)—C(14)	-179.82 (13)
C(1)—C(2)—C(7)—C(8)	0.5 (2)	C(11)—C(10)—C(15)—O(3)	179.44 (11)
C(3)—C(2)—C(7)—C(6)	1.2 (2)	C(11)—C(10)—C(15)—C(14)	0.8 (2)
C(3)—C(2)—C(7)—C(8)	-179.87 (12)	C(10)—C(11)—C(12)—C(13)	0.8 (2)
C(2)—C(3)—C(4)—C(5)	-0.1 (2)	C(11)—C(12)—C(13)—C(14)	-0.1 (2)
C(3)—C(4)—C(5)—C(6)	1.8 (2)	C(12)—C(13)—C(14)—C(15)	-0.3 (2)
C(4)—C(5)—C(6)—C(7)	-1.9 (2)	C(13)—C(14)—C(15)—O(3)	-178.70 (12)
C(5)—C(6)—C(7)—C(2)	0.4 (2)	C(13)—C(14)—C(15)—C(10)	-0.1 (2)

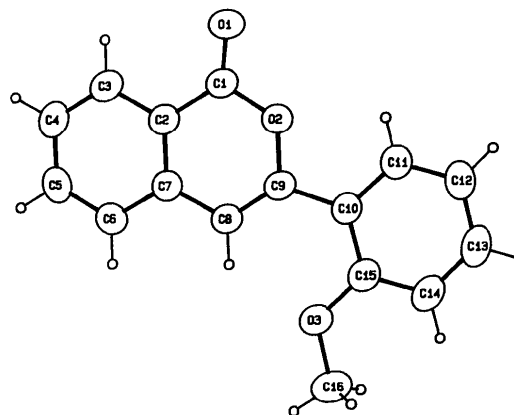


Fig. 1. Numbering scheme, and thermal ellipsoids drawn at the 40% probability level. H atoms have arbitrary radius.

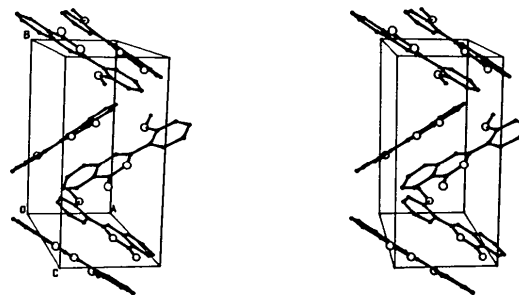


Fig. 2. Stereopair of the unit cell, viewed slightly oblique to the c axis, illustrating the polar nature of the molecular packing. O atoms are represented by larger circles and H atoms are omitted.

that we observed between the benzopyrone system and its substituent. Structure of lateropyrone diacetate: Gorst-Allman, Van Rooyen, Wnuk, Golinski & Chelkowski (1986).

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