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3-(2-Bromophenyl)isocoumarin

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.006 Å
R factor = 0.038
wR factor = 0.094
Data-to-parameter ratio = 15.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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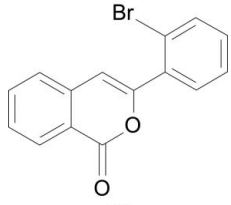
The molecular structure of the title compound, $C_{15}H_9BrO_2$, consists of two essentially planar units, benzopyran-1-one and 2-bromophenyl, which are inclined at 51.42 (12)° with respect to one another. The structure is stabilized by two weak intermolecular C—H···O interactions.

Comment

More than two hundred isocoumarins and 3,4-dihydroisocoumarins have been isolated, predominantly from a variety of fungi, lichens and bacteria, and to a lesser extent from higher plants, insects and marine organisms, and the number of known isocoumarins is still increasing dramatically (Barry, 1964; Napolitano, 1997). Isocoumarins are useful intermediates (Hauser & Baghdanor, 1988; Mali & Babu, 1998) in the synthesis of a variety of natural products. We have reported the syntheses of a number of naturally occurring and synthetic isocoumarins (Saeed, 2003*a*,*b*, 2004*a*,*b*; Saeed & Ehsan, 2005). 3-Halophenylisocoumarins are not known in nature and are expected to display a number of bioactivities. The title compound, (I), was prepared in order to investigate its bioactivity systematically. In this paper, the structure of (I) is described.



(I)

The structure of (I) consists of an essentially planar benzopyran-1-one unit, the maximum deviation of any atom

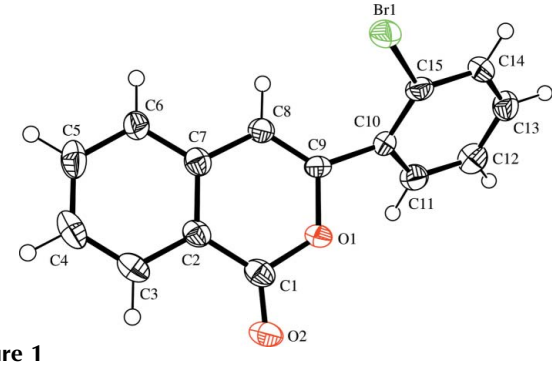


Figure 1
ORTEPII (Johnson, 1976) drawing of (I), with displacement ellipsoids plotted at the 50% probability level. H atoms are drawn as small spheres.

from its plane being 0.025 (4) Å for C1, and a 2-bromophenyl unit for which Br1 is 0.046 (8) Å out of the plane of the benzene ring (Fig. 1); the mean planes of the two units are inclined at 51.42 (12)° with respect to one another. The molecular dimensions in (I) agree with the corresponding dimensions reported for 3,4-dihydroisocoumarins and isocoumarins included in the Cambridge Structural Database (Version 5.27, 2005 Release; Allen, 2002). The structure is stabilized by two weak intermolecular C—H···O interactions (Fig. 2 and Table 1). There are no significant $\pi-\pi$ stacking interactions.

Experimental

A stirred mixture of homophthalic acid (0.5 g, 2.77 mmol) and 2-bromobenzoyl chloride (2.12 g, 9.7 mmol) was heated in an oil bath at 473 K for 4 h. Thin-layer chromatography of the residue (petroleum ether–ethyl acetate 8:3) followed by recrystallization from MeOH–H_2O (4:1) gave the title isocoumarin (0.7 g, 2.35 mmol, 85%) as light-yellow needles (m.p. 391–393 K). ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 6.90 (*s*, 1H, H-4), 7.43 (*m*, 1H, H-5), 7.47 (*m*, 1H, H-7), 7.49 (*m*, 1H, H-4′), 7.51 (*m*, 1H, H-6′), 7.69 (*dt*, J = 7.2, 1.8 Hz, 1H, H-6); 7.81 (*dt*, J = 7.8, 1.7 Hz, 1H, H-5′).

Crystal data

$C_{15}H_9BrO_2$
M_r = 301.13
Orthorhombic, $Pna2_1$
a = 12.595 (6) Å
b = 12.128 (9) Å
c = 7.699 (6) Å
V = 1176.0 (14) Å^3
Z = 4
D_x = 1.701 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.48 mm^{-1}
T = 173 (2) K
Block, colourless
0.16 × 0.14 × 0.10 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.594, T_{max} = 0.708
7105 measured reflections
2492 independent reflections
2096 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.094
S = 1.04
2492 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.803P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Absolute structure: Flack (1983), 1073 Fridels
Flack parameter: 0.536 (17)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	0.95	2.55	3.439 (6)	155
C6—H6···O2^{ii}	0.95	2.45	3.242 (7)	140

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, z-\frac{1}{2}$.

The crystal is inversion twinned with roughly equal components. The Friedel pairs (1073) were not merged during the refinement. H atoms were located in difference Fourier syntheses and were included in the refinement at geometrically idealized positions, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C).

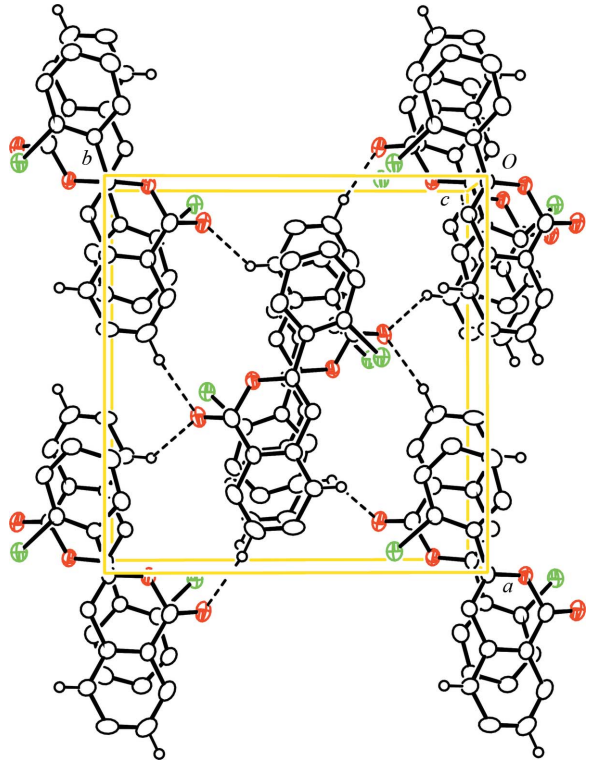


Figure 2
ORTEPII (Johnson, 1976) drawing of the packing of (I), showing weak intermolecular C—H···O hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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