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3-(4-Methoxystyryl)-2*H*-1,4-benzoxazin-2-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.140
Data-to-parameter ratio = 16.4

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

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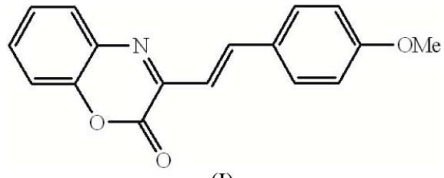
The title compound, $C_{17}H_{13}NO_3$, a coumarin analog prepared from (*E*)-methyl 4-(4-methoxyphenyl)-2-oxobut-3-enoate and 2-aminophenol, has a planar conformation. Aromatic π-stacking interactions and C—H···O hydrogen bonds stabilize the crystal structure.

Comment

Coumarins and their derivatives are widely distributed in the plant kingdom and have attracted considerable attention because of their varied pharmaceutical activities; these include inhibition of platelet aggregation (Cravotto *et al.*, 2001), antibacterial activity (Kayser & Kolodziej, 1997), anticancer activity (Wang *et al.*, 2002), inhibition of steroid 5-reductase (Fan *et al.*, 2001) and inhibition of HIV-1 protease (Kirkiacharian *et al.*, 2002). It is interesting to synthesize coumarin analogs since they may yield a new chemical class of pharmaceutical agents, with new modes of action and lacking resistance to currently used chemicals. Against this background, the title compound, (I), has been synthesized and its crystal structure determined.



(I)

The structure of (I) is shown in Fig. 1, with the atomic numbering scheme. All atoms, with the exception of methyl H atoms, are essentially coplanar, with an r.m.s. deviation of 0.060 Å. A packing diagram of the crystal structure of (I) (Fig. 2) shows that aromatic π-stacking interactions and C—H···O hydrogen bonds (Table 2) stabilize the crystal structure. The

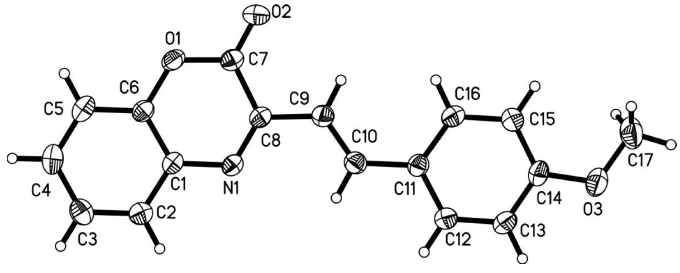


Figure 1
View of the molecule, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by circles of arbitrary size.

distance between the planes of the heterocyclic ring and ring C1–C6 at $(1-x, 1-y, 1-z)$ is 3.523 (1) Å.

Experimental

A mixture of (*E*)-methyl 4-(4-methoxyphenyl)-2-oxobut-3-enoate (0.2 mmol) and 2-aminophenol (0.2 mmol) in boiling trifluoroacetic acid (2 ml) under atmospheric nitrogen was stirred for 1 d; the trifluoroacetic acid was then distilled out for future use. The residue was diluted with dichloromethane (30 ml), washed with saturated aqueous sodium bicarbonate (5 ml) and then water (5 ml). It was then dried over anhydrous sodium sulfate, filtered, evaporated under reduced pressure, and isolated by flash chromatography on silica gel (200–300 mesh) in 91% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution (m.p. 431–433 K). FT–IR (KBr, ν cm^{-1}): 1735, 1600, 1511, 1260, 1169, 1079, 754; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.05 (*d, J* = 16.11 Hz, 1H), 7.71 (*dd*, *J* = 9.33, 1.59 Hz, 1H), 7.50 (*d, J* = 8.72 Hz, 2H), 7.41–7.31 (*m*, 3H), 7.24 (*dd*, *J* = 8.04, 1.35 Hz, 1H), 6.89 (*d, J* = 8.72 Hz, 2H), 3.82 (*s*, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 161.18, 149.69, 145.89, 140.04, 132.05, 130.09, 129.80, 128.70, 128.61, 125.59, 118.76, 116.23, 114.38, 55.39; analysis calculated for $C_{17}H_{13}NO_3$: C 73.11, H 4.69, N 5.02%; found: C 73.19, H 4.63, N 5.00%.

Crystal data

$C_{17}H_{13}NO_3$
M_r = 279.28
Orthorhombic, *Pbca*
a = 11.857 (2) Å
b = 7.1867 (14) Å
c = 32.253 (7) Å
V = 2748.5 (10) Å^3
Z = 8
D_x = 1.350 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 19445 reflections
θ = 1.3–27.5°
μ = 0.09 mm^{-1}
T = 293 (2) K
Platelet, yellow
0.49 × 0.45 × 0.04 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.955, T_{max} = 0.997
19445 measured reflections
3131 independent reflections
1471 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 27.5°
$h = -14 \rightarrow 15$
$k = -8 \rightarrow 9$
$l = -41 \rightarrow 40$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.140
S = 0.83
3131 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0395P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.368 (2)	N1—C8	1.291 (3)
O1—C6	1.378 (2)	N1—C1	1.395 (3)
O2—C7	1.194 (3)	C9—C10	1.325 (3)
C10—C9—C8	123.7 (2)	C9—C10—C11	127.2 (2)
C7—C8—C9—C10	−179.2 (2)	C8—C9—C10—C11	179.8 (2)

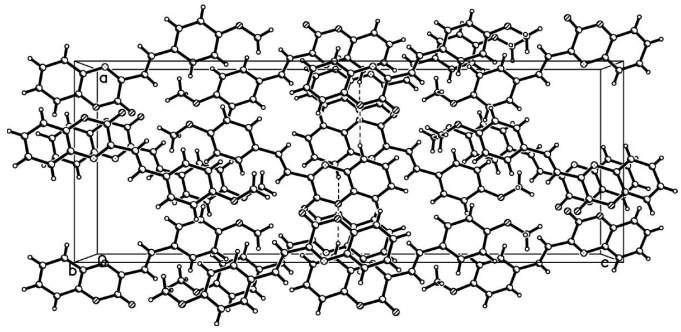


Figure 2
The molecular packing, viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O1^{i}	0.93	2.50	3.397 (3)	163

Symmetry code: (i) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$.

The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *RAPID-AUTO* (Rigaku, 2000); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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