Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Methoxyphenyl 4-bromo-7-methoxy-1,3-benzodioxole-5-carboxylate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.046
wR factor = 0.124
Data-to-parameter ratio = 12.6

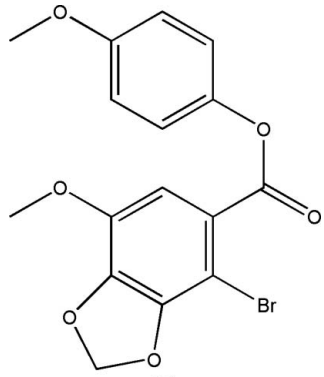
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 26 September 2005
Accepted 17 October 2005
Online 22 October 2005

The title compound, $C_{16}H_{13}BrO_6$, was synthesized in an anhydrous medium. The methylenedioxobenzoate moiety and the methoxyphenyl fragment form a dihedral angle of 65.06 (8)°.

Comment

Schizandrin-type lignans have shown a broad range of biological activities, such as antihepatotoxic, anti-HIV and anticancer activity (Chen *et al.*, 1997; Song & Xiao, 1982). The biphenyl unit of these natural products is crucial for their pharmacological activity (Chang *et al.*, 2003). The title compound, (I), is an intermediate used for the synthesis of benzocoumarin lactone by intramolecular coupling (Gray *et al.*, 2003) and the lactone can be converted to biphenyl derivatives which may have some useful biological activity.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The bond lengths and angles within the methylenedioxobenzoate moiety agree with those reported in a related structure (Ammon *et al.*, 1986). This dioxobenzoate moiety is planar, as expected, with the largest deviation being 0.0195 (2) Å for atom O2. Atom Br1, atom O3 of the methoxy fragment and atom C9 are roughly in this plane, being only 0.009 (4), 0.013 (5) and 0.093 (5) Å, respectively, away from it. Atoms O4 and O5 are only 0.13 (6) Å out of this plane. The benzene ring is twisted by 65.06 (8)° with respect to this plane.

Experimental

The title compound was prepared according to the published procedure of Gerhard *et al.* (2003). The reaction was initiated by the addition of one molar equivalent of 4-methoxyphenol and one molar equivalent of 4-bromo-7-methoxybenzo[*d*][1,3]dioxole-5-carboxylic acid to dichloromethane and subsequent stirring at room temperature for 12 h. A white powder (m.p. 399 K) resulted (yield 86%) and

single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{16}H_{13}BrO_6$
M_r = 381.17
Monoclinic, $P2_1/n$
a = 4.1749 (12) Å
b = 18.874 (5) Å
c = 19.335 (6) Å
β = 90.618 (5)°
V = 1523.5 (8) Å^3
Z = 4
D_x = 1.662 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2617 reflections
θ = 2.4–26.1°
μ = 2.73 mm^{-1}
T = 294 (2) K
Block, colourless
0.40 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.345, T_{max} = 0.580
6964 measured reflections
2653 independent reflections
1942 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 25.0°
$h = -4 \rightarrow 4$
$k = -22 \rightarrow 16$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.124
S = 1.01
2653 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0661P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

All H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.97 Å and with U_{iso}(H)= 1.2U_{eq}(CH_2 or C_{phenyl}), or 1.5U_{eq}(CH_3).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

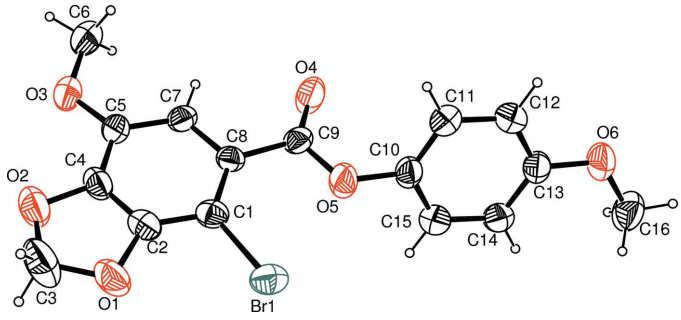


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size.

This research was supported by the National Natural Science Foundation of China (grant No. 200342005).

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