Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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Received 24 June 2005
Accepted 6 July 2005
Online 13 July 2005

Key indicators

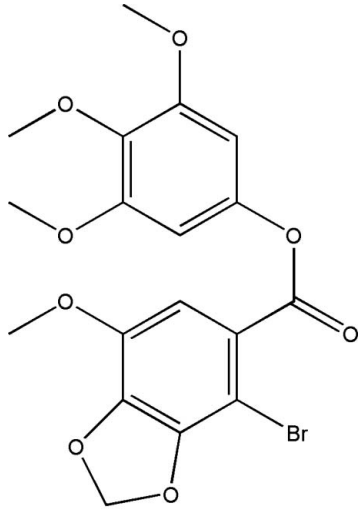
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.100
Data-to-parameter ratio = 14.8

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

The title compound, $C_{18}H_{17}BrO_8$, was synthesized in an anhydrous medium at 293 K. The dihedral angle between the two benzene rings is 38.8 (2)°.

Comment

The title compound, (I), is a key intermediate of biphenyl derivatives, which may act to moderate liver ailments, and thus be effective in the treatment of actute and chronic hepatitis (Song & Xiao, 1982).



(I)

Its molecular structure of (I) is illustrated in Fig. 1. The bond lengths and angles are unremarkable. The dihedral angle between the five-membered ring and the fused benzene ring is

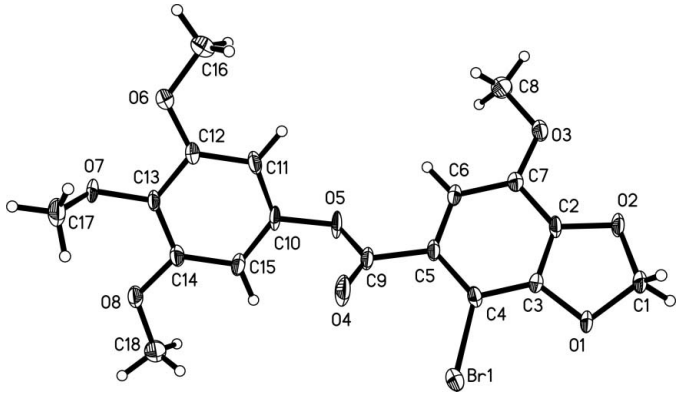


Figure 1
View of the title molecule, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms are represented by circles of arbitrary size.

3.3 (1)°, and the angle between the two benzene rings is 38.8 (2)°. Intermolecular C—H···O hydrogen bonds help to stabilize the crystal structure (Fig. 2 and Table 1).

Experimental

The title compound, (I), was prepared according to a literature procedure (Bringmann *et al.*, 2003). The reaction was initiated by the addition of one molar equivalent of 3,4,5-trimethoxyphenol, 4-bromo-7-methoxy-1,3-benzodioxole-5-carboxylic acid, dicyclohexylcarbodiimide and 0.2 molar equivalents of 4-(dimethylamino)pyridine to dichloromethane. The mixture was stirred at room temperature for 12 h. A white powder (m.p. 461 K) resulted (yield 90%) and single crystals suitable for X-ray analysis were obtained by slow evaporation of a dichloromethane solution. IR (KBr, ν cm^{-1}): 1739, 1608, 1502, 1431, 1338, 1172, 1135, 1033, 942; ^{1}H NMR ($CDCl_3$): δ 7.449 (*s*, 1H), 6.473 (*s*, 2H), 6.176 (*s*, 2H), 3.967 (*s*, 3H), 3.863 (*s*, 6H), 3.851 (s, 3H).

Crystal data

$C_{18}H_{17}BrO_8$
M_r = 441.23
Monoclinic, $P2_1/n$
a = 8.6563 (16) Å
b = 7.6330 (12) Å
c = 26.927 (4) Å
β = 95.403 (9)°
V = 1771.2 (5) Å^3
Z = 4
D_x = 1.655 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2497 reflections
θ = 2.6–26.1°
μ = 2.37 mm^{-1}
T = 294 (2) K
Block, colorless
0.54 × 0.40 × 0.32 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.288, T_{max} = 0.469
9627 measured reflections
3679 independent reflections
2307 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 26.8°
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 7$
$l = -34 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.100
S = 1.00
3679 reflections
249 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.0516P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0369 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O7^{i}	0.97	2.42	3.278 (4)	147
C8—H8A···O4^{ii}	0.96	2.45	3.244 (4)	140

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

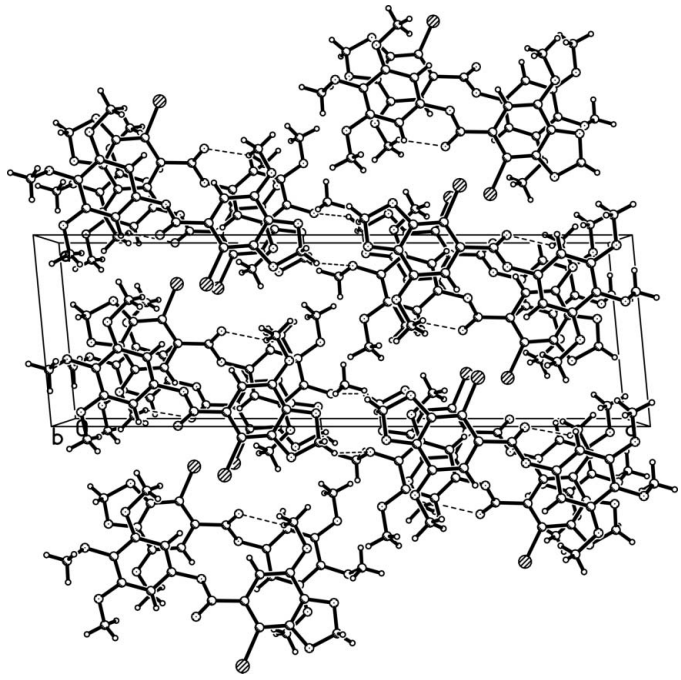


Figure 2
A view of the crystal structure, viewed approximately along the *b* axis. Dashed lines indicate hydrogen bonds.

All H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.97 Å. U_{iso}(H) values were set equal to xU_{eq}(carrier atom), where x = 1.5 for methyl H atoms and 1.2 for all others.

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: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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

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