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2-Bromo-3,4,5-trimethoxybenzyl 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) = 0.004 Å
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 17.4

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

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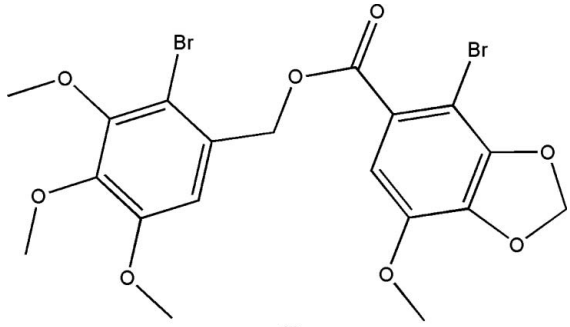
The title compound, $C_{19}H_{18}Br_2O_8$, was synthesized in an anhydrous medium at 293 K. The five-membered ring adopts an envelope conformation. The dihedral angle between the two benzene rings is 2.6 (1)°.

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 acute and chronic hepatitis (Song & Xiao, 1982).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The bond lengths and angles are unremarkable. The five-membered ring adopts an envelope conformation with atom C7 as the flap. The dihedral angle between the C1–C6/O2/O3 and C11–C16 planes is 2.6 (1)°, and the angle between the C9/C10/O4/O5 and C1–C6/O2/O3 planes is 21.8 (2)°. The C—O—C—C torsion angles, which describe the orientation of the methoxy groups with respect to the attached rings, are given in Table 1.

Weak intramolecular C—H···O and C—H···Br interactions are observed. Intermolecular C—H···O hydrogen bonds help to stabilize the crystal structure (Table 2). In addition, a short Br2···O4($1-x$, $-y$, $1-z$) contact of 3.065 (2) Å is observed.

Experimental

The title compound was prepared according to the literature procedure of Bringmann *et al.* (2003). The reaction was initiated by the addition of one molar equivalent of (2-bromo-3,4,5-trimethoxyphenyl)methanol, 4-bromo-7-methoxy-1,3-benzodioxole-5-carboxylic acid, dicyclohexylcarbodiimide and 0.2 molar equivalent of 4-(dimethylamino)pyridine to dichloromethane. The mixture was stirred at room temperature for 12 h. A white powder (m.p. 394 K) resulted (yield 88%) and single crystals of (I) suitable for X-ray

analysis were obtained by slow evaporation of a dichloromethane solution.

Crystal data

$C_{19}H_{18}Br_2O_8$	V = 1009.2 (3) Å^3
M_r = 534.15	Z = 2
Triclinic, $P\overline{1}$	D_x = 1.758 Mg m^{-3}
a = 7.9131 (13) Å	Mo $K\alpha$ radiation
b = 9.5314 (15) Å	μ = 4.06 mm^{-1}
c = 13.949 (3) Å	T = 294 (2) K
α = 77.692 (12)°	Block, colourless
β = 79.132 (17)°	0.12 × 0.10 × 0.08 mm
γ = 88.946 (18)°	

Data collection

Rigaku Saturn diffractometer	10738 measured reflections
ω scans	4595 independent reflections
Absorption correction: multi-scan (Jacobson, 1998)	3140 reflections with $I > 2\sigma(I)$
T_{min} = 0.642, T_{max} = 0.737	R_{int} = 0.035
	θ_{max} = 27.5°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.078	$\Delta\rho_{max}$ = 0.62 e Å^{-3}
S = 0.93	$\Delta\rho_{min}$ = −0.54 e Å^{-3}
4595 reflections	Extinction correction: *SHELXL97*
264 parameters	Extinction coefficient: 0.0077 (10)
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C8—O1—C1—C6	−7.4 (4)	C18—O7—C14—C15	81.7 (3)
C17—O6—C13—C12	86.8 (3)	C19—O8—C15—C16	1.1 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O5	0.93	2.37	2.713 (3)	101
C8—H8A···O2^{i}	0.96	2.59	3.533 (3)	166
C16—H16···O5	0.93	2.37	2.735 (3)	103
C17—H17B···O7^{ii}	0.96	2.57	3.287 (3)	132
C17—H17C···Br2	0.96	2.93	3.468 (3)	117

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

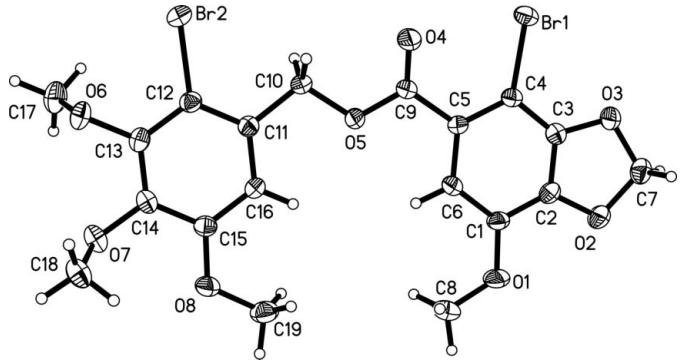


Figure 1
A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level and H atoms are represented by circles of arbitrary size.

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}(C), where x = 1.5 for methyl H atoms and 1.2 for all others.

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); 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: *CrystalStructure*.

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