

5-(4-Bromophenyl)-2-methyl-3-methylsulfanyl-1-benzofuran

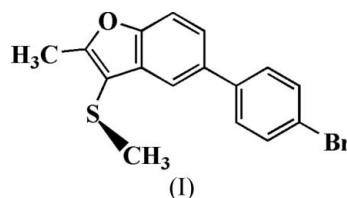
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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.029
 wR factor = 0.073
Data-to-parameter ratio = 17.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{16}\text{H}_{13}\text{BrOS}$, was prepared by the Lewis acid-catalysed reaction of 4'-bromo-1,1'-biphenyl-4-ol with α -chloro- α -(methylsulfanyl)acetone. The crystal structure is stabilized by a $\text{C}-\text{H}\cdots\pi$ interaction involving a CH group of the 4-bromophenyl ring and the furan ring.Received 10 July 2006
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Comment

1-Benzofurans are present in numerous examples of synthetic substances and natural products; derivatives containing such ring systems have attracted considerable interest because of their various pharmacological properties (Ward, 1997; Howlett *et al.*, 1999). The synthetic chemistry and reactivity of 1-benzofurans have been also extensively studied and discussed elsewhere (Cagniant & Cagniant, 1975). With our continued interest in the syntheses and structures of 1-benzofuran derivatives (Choi *et al.*, 2003, 2004, 2006), we report here the crystal structure of the title compound, (I), which was obtained directly by the one-pot reaction of 4'-bromo-1,1'-biphenyl-4-ol with α -chloro- α -(methylsulfanyl)acetone in the presence of zinc chloride.In (I), bond lengths and angles (Table 1) are as expected for this type of compound (Choi *et al.*, 2006). The dihedral angle formed by the 4-bromophenyl and benzofuran ring system is $37.62(4)^\circ$ (Fig. 1). As a result of this twisting, no efficient π - π stacking interaction is observed in the crystal structure. The packing of (I) is stabilized by a $\text{C}-\text{H}\cdots\pi$ interaction involving a CH group of the 4-bromophenyl ring and the furan ring of an adjacent molecule (Table 2).

Experimental

The title compound, (I), was prepared following a published method for 2-arylbenzo[*b*]furan derivatives (Choi *et al.*, 1999). Zinc chloride (341 mg, 2.5 mmol) was added at room temperature to a stirred solution of 4'-bromo-1,1'-biphenyl-4-ol (623 mg, 2.5 mmol) and α -chloro- α -(methylsulfanyl)acetone (346 mg, 2.5 mmol) in dichloromethane (30 ml) and stirred for 40 min. The mixture was quenched with water and the organic layer separated, dried over magnesium sulfate, filtered and concentrated *in vacuo*. The residue was purified by column chromatography (CCl_4) to afford (I) as a pale-yellow solid.

Crystals suitable for X-ray analysis were grown by slow evaporation of a tetrahydrofuran solution [yield 53%, m.p. 376–377 K; $R_F = 0.61$ (CCl_4)].

Crystal data

$\text{C}_{16}\text{H}_{13}\text{BrOS}$ $Z = 4$
 $M_r = 333.23$ $D_x = 1.576 \text{ Mg m}^{-3}$
 Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation
 $a = 11.4244 (8) \text{ \AA}$ $\mu = 3.06 \text{ mm}^{-1}$
 $b = 7.8195 (5) \text{ \AA}$ $T = 120 (2) \text{ K}$
 $c = 15.934 (1) \text{ \AA}$ Block, colourless
 $\beta = 99.441 (1)^\circ$ $0.60 \times 0.35 \times 0.35 \text{ mm}$
 $V = 1404.15 (16) \text{ \AA}^3$

Data collection

Bruker SMART CCD 8283 measured reflections
 diffractometer 3058 independent reflections
 φ and ω scans 2559 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{\text{int}} = 0.036$
 (SADABS; Sheldrick, 1999) $\theta_{\text{max}} = 27.0^\circ$
 $T_{\text{min}} = 0.193$, $T_{\text{max}} = 0.348$

Refinement

Refinement on F^2 H-atom parameters constrained
 $R[F^2 > 2\sigma(F^2)] = 0.029$ $w = 1/[\sigma^2(F_o^2) + (0.0414P)^2]$
 $wR(F^2) = 0.073$ where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.06$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 3058 reflections $\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$
 174 parameters $\Delta\rho_{\text{min}} = -0.62 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

Br—C1	1.898 (2)	O—C10	1.377 (2)
S—C12	1.7543 (18)	O—C11	1.387 (2)
S—C16	1.812 (2)		
C12—S—C16	100.96 (9)	C2—C1—Br	119.44 (15)
C10—O—C11	106.26 (14)	C6—C1—Br	119.74 (15)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

C_g is the centroid of the furan ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C2-H2 \cdots C_g^i$	0.93	2.83	3.604 (3)	142

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

All H atoms were positioned geometrically and refined using a riding model, with $C-H = 0.93 \text{ \AA}$ for aromatic H atoms and 0.96 \AA for methyl H atoms, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

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: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXL97.

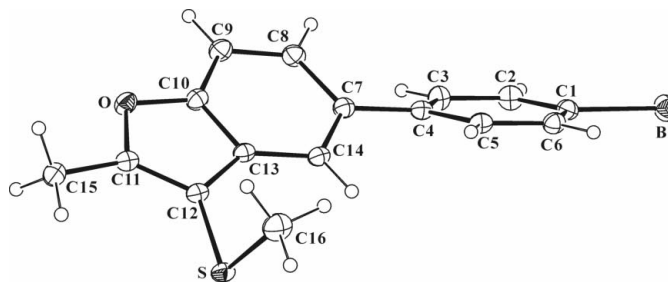


Figure 1

The molecular structure of (I), with 30% probability displacement ellipsoids.

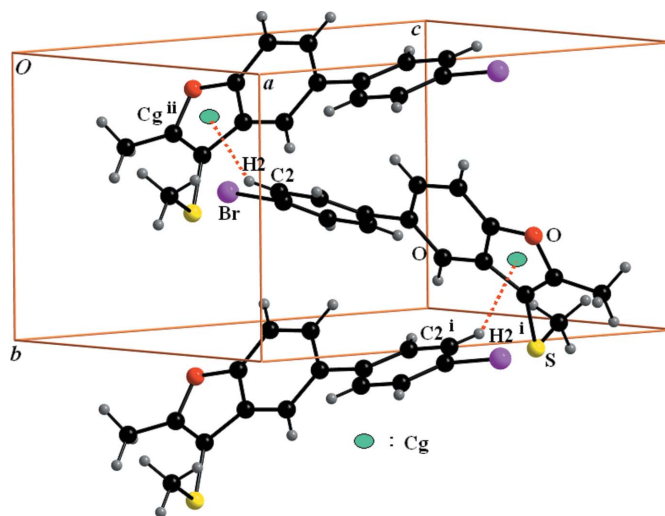


Figure 2

A partial packing diagram for (I), with $C-H \cdots \pi$ interactions shown as dotted lines. C_g is the centroid of the furan ring. [Symmetry codes: (i) $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $2 - x, y + \frac{1}{2}, \frac{1}{2} - z$.]

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