5-Bromo-2-phenyl-3-phenylsulfinyl-1-benzofuran

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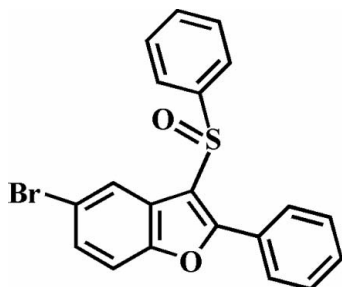Correspondence e-mail: uklee@pknu.ac.kr

In the title compound, $C_{20}H_{13}BrO_2S$, the O atom and the phenyl group of the phenylsulfinyl substituent are located on opposite sides of the plane of the benzofuran system. The S-bound phenyl ring is almost perpendicular to this plane [80.35 (8)°]. The phenyl ring in the 2-position is twisted with respect to the benzofuran plane, making a dihedral angle of 16.0 (1)°.

Related literature

For the crystal structures of similar 5-halo-2-phenyl-3-phenylsulfinyl-1-benzofuran derivatives, see: Choi *et al.* (2009*a,b*). For the pharmacological activity of benzofuran compounds, see: Howlett *et al.* (1999); Twyman & Allsop (1999). For natural products involving a benzofuran ring system, see: Akgul & Anil (2003); Reuss & König (2004).

Experimental

Crystal data

$C_{20}H_{13}BrO_2S$	γ = 69.526 (1)°
M_r = 397.27	V = 831.76 (2) Å^3
Triclinic, $P\bar{1}$	Z = 2
a = 8.2670 (1) Å	Mo $K\alpha$ radiation
b = 9.5233 (2) Å	μ = 2.61 mm^{-1}
c = 11.8663 (2) Å	T = 293 K
α = 72.187 (1)°	0.35 × 0.22 × 0.11 mm
β = 80.772 (1)°	

Data collection

Bruker SMART CCD diffractometer	13542 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)	3275 independent reflections
T_{min} = 0.462, T_{max} = 0.763	2716 reflections with $I > 2\sigma(I)$
	R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	217 parameters
$wR(F^2)$ = 0.095	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.51 e Å^{-3}
3275 reflections	$\Delta\rho_{min}$ = −0.59 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2491).

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supporting information

5-Bromo-2-phenyl-3-phenylsulfinyl-1-benzofuran

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S1. Comment

Molecules containing benzofuran moiety have received much attenttion in the field of their pharmacological properties (Howlett *et al.*, 1999; Twyman & Allsop, 1999), and these compounds are ubiquitous in nature (Akgul & Anil, 2003; Reuss & König, 2004). As a part of our ongoing studies of the effect of side chain substituents on the solid state structures of 5-halo-2-phenyl-3-phenylsulfinyl-1-benzofuran analogues (Choi *et al.*, 2009*a,b*), we present the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.010 (2) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the plane of the benzofuran unit and the plane of 2-phenyl ring is 16.0 (1)°. The phenyl ring (C15-C20) is almost perpendicular to the plane of the benzofuran unit [80.35 (8)°].

S2. Experimental

77% 3-Chloroperoxybenzoic acid (224 mg, 1.0 mmol) was added in small portions to a stirred solution of 5-bromo-2-phenyl-3-phenylsulfanyl-1-benzofuran (343 mg, 0.9 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 4h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (hexane-ethyl acetate, 2 : 1 v/v) to afford the title compound as a colorless solid [yield 73%, m.p. 417-418 K; R_f = 0.56 (hexane-ethyl acetate, 2 : 1 v/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in benzene at room temperature.

S3. Refinement

All H atoms were fixed geometrically and treated as riding with C–H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$
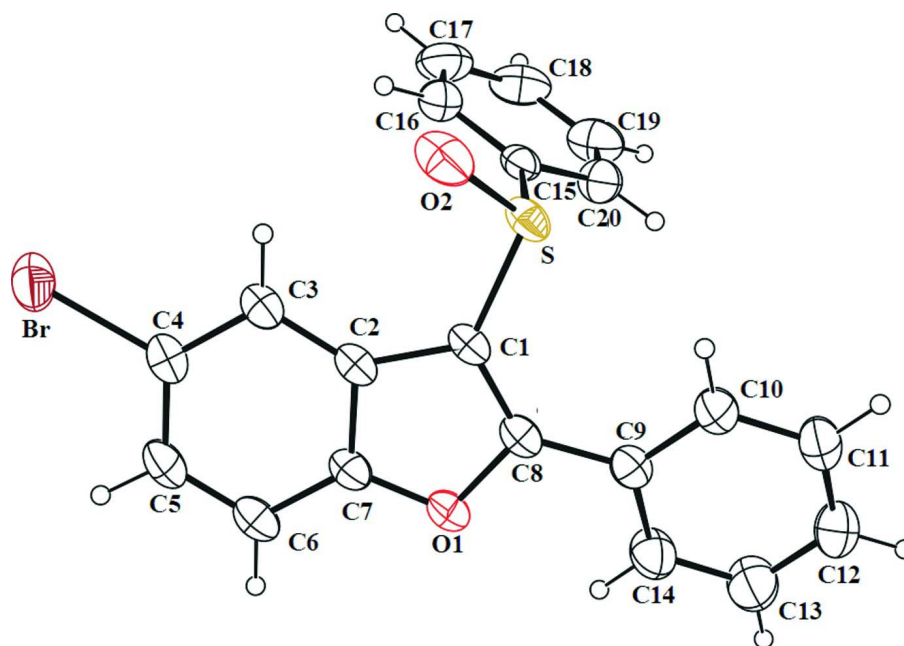
Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as small spheres of arbitrary radii.

5-Bromo-2-phenyl-3-phenylsulfinyl-1-benzofuran

Crystal data

$C_{20}H_{13}BrO_2S$	$Z = 2$
$M_r = 397.27$	$F(000) = 400$
Triclinic, $P\overline{1}$	$D_x = 1.586$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.2670$ (1) Å	Cell parameters from 5885 reflections
$b = 9.5233$ (2) Å	$\theta = 2.4$–26.0°
$c = 11.8663$ (2) Å	$\mu = 2.61$ mm^{-1}
$\alpha = 72.187$ (1)°	$T = 293$ K
$\beta = 80.772$ (1)°	Block, colorless
$\gamma = 69.526$ (1)°	$0.35 \times 0.22 \times 0.11$ mm
$V = 831.76$ (2) Å^3	

Data collection

Bruker SMART CCD diffractometer	13542 measured reflections
Radiation source: fine-focus sealed tube	3275 independent reflections
Graphite monochromator	2716 reflections with $I > 2\sigma(I)$
Detector resolution: 10.0 pixels mm^{-1}	$R_{int} = 0.030$
φ and ω scans	$\theta_{max} = 26.0°$, $\theta_{min} = 1.8°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)	$h = -10 \rightarrow 10$
	$k = -11 \rightarrow 11$
$T_{min} = 0.462$, $T_{max} = 0.763$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.095$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.3341P]$
3275 reflections	where $P = (F_o^2 + 2F_c^2)/3$
217 parameters	$(\Delta/\sigma)_{max} = 0.001$
0 restraints	$\Delta\rho_{max} = 0.51$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{min} = -0.59$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Br	1.01046 (4)	0.24078 (4)	0.38069 (3)	0.07875 (15)
S	0.60607 (8)	0.10785 (7)	0.86253 (6)	0.05063 (17)
O1	0.5011 (2)	0.56165 (18)	0.71087 (15)	0.0552 (4)
O2	0.7801 (2)	0.0093 (2)	0.8283 (2)	0.0760 (6)
C1	0.5823 (3)	0.3012 (2)	0.7751 (2)	0.0453 (5)
C2	0.6714 (3)	0.3415 (3)	0.6609 (2)	0.0470 (5)
C3	0.7888 (3)	0.2607 (3)	0.5871 (2)	0.0516 (6)
H3	0.8269	0.1525	0.6070	0.062*
C4	0.8466 (3)	0.3472 (3)	0.4831 (2)	0.0566 (6)
C5	0.7910 (4)	0.5094 (3)	0.4506 (3)	0.0655 (7)
H5	0.8338	0.5632	0.3796	0.079*
C6	0.6738 (4)	0.5895 (3)	0.5225 (3)	0.0645 (7)
H6	0.6343	0.6978	0.5017	0.077*
C7	0.6166 (3)	0.5035 (3)	0.6272 (2)	0.0518 (6)
C8	0.4830 (3)	0.4365 (3)	0.8020 (2)	0.0486 (5)
C9	0.3645 (3)	0.4760 (3)	0.9012 (2)	0.0505 (6)
C10	0.3612 (4)	0.3686 (3)	1.0094 (3)	0.0660 (7)
H10	0.4365	0.2672	1.0207	0.079*
C11	0.2474 (4)	0.4101 (4)	1.1009 (3)	0.0750 (8)
H11	0.2469	0.3363	1.1735	0.090*
C12	0.1352 (4)	0.5581 (4)	1.0866 (3)	0.0768 (9)
H12	0.0581	0.5850	1.1487	0.092*
C13	0.1375 (5)	0.6667 (4)	0.9799 (3)	0.0868 (10)
H13	0.0622	0.7680	0.9696	0.104*
C14	0.2507 (4)	0.6265 (3)	0.8877 (3)	0.0732 (8)

H14	0.2511	0.7011	0.8156	0.088*
C15	0.4515 (3)	0.0742 (2)	0.7931 (2)	0.0455 (5)
C16	0.5017 (4)	0.0001 (3)	0.7038 (3)	0.0647 (7)
H16	0.6170	−0.0307	0.6762	0.078*
C17	0.3767 (6)	−0.0275 (4)	0.6558 (3)	0.0833 (10)
H17	0.4082	−0.0778	0.5959	0.100*
C18	0.2085 (5)	0.0188 (4)	0.6965 (4)	0.0836 (10)
H18	0.1254	0.0015	0.6629	0.100*
C19	0.1600 (4)	0.0901 (4)	0.7856 (4)	0.0791 (10)
H19	0.0445	0.1200	0.8129	0.095*
C20	0.2804 (3)	0.1180 (3)	0.8351 (3)	0.0592 (6)
H20	0.2474	0.1659	0.8964	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0790 (2)	0.0939 (3)	0.0630 (2)	−0.03538 (18)	0.01864 (16)	−0.02325 (17)
S	0.0434 (3)	0.0442 (3)	0.0520 (4)	−0.0148 (2)	−0.0066 (3)	0.0070 (2)
O1	0.0669 (11)	0.0419 (8)	0.0506 (10)	−0.0212 (8)	−0.0008 (8)	−0.0010 (7)
O2	0.0417 (10)	0.0554 (10)	0.1019 (16)	−0.0060 (8)	−0.0054 (10)	0.0088 (10)
C1	0.0446 (12)	0.0424 (11)	0.0450 (13)	−0.0191 (9)	−0.0066 (10)	0.0017 (9)
C2	0.0452 (12)	0.0475 (12)	0.0462 (13)	−0.0220 (10)	−0.0059 (10)	0.0002 (10)
C3	0.0468 (13)	0.0515 (13)	0.0524 (14)	−0.0201 (10)	−0.0030 (11)	−0.0034 (11)
C4	0.0540 (14)	0.0659 (15)	0.0496 (15)	−0.0267 (12)	0.0013 (11)	−0.0085 (12)
C5	0.0774 (19)	0.0670 (17)	0.0487 (15)	−0.0372 (15)	0.0034 (13)	0.0020 (13)
C6	0.0787 (19)	0.0507 (14)	0.0568 (16)	−0.0289 (13)	−0.0025 (14)	0.0045 (12)
C7	0.0548 (14)	0.0484 (12)	0.0489 (14)	−0.0222 (11)	−0.0026 (11)	−0.0022 (10)
C8	0.0516 (13)	0.0473 (12)	0.0438 (13)	−0.0214 (10)	−0.0071 (10)	0.0008 (10)
C9	0.0501 (13)	0.0527 (13)	0.0490 (14)	−0.0208 (11)	−0.0042 (11)	−0.0085 (11)
C10	0.0787 (19)	0.0550 (15)	0.0548 (16)	−0.0188 (13)	0.0048 (14)	−0.0090 (12)
C11	0.088 (2)	0.0731 (19)	0.0537 (17)	−0.0271 (17)	0.0110 (15)	−0.0100 (14)
C12	0.0691 (19)	0.088 (2)	0.070 (2)	−0.0255 (17)	0.0152 (15)	−0.0269 (17)
C13	0.077 (2)	0.0691 (19)	0.089 (3)	−0.0030 (16)	0.0095 (18)	−0.0164 (18)
C14	0.0709 (19)	0.0611 (16)	0.0660 (19)	−0.0098 (14)	0.0026 (15)	−0.0036 (14)
C15	0.0445 (12)	0.0335 (10)	0.0491 (13)	−0.0121 (9)	−0.0008 (10)	0.0006 (9)
C16	0.0687 (17)	0.0482 (13)	0.0679 (18)	−0.0157 (12)	0.0089 (14)	−0.0125 (12)
C17	0.127 (3)	0.0537 (16)	0.078 (2)	−0.0311 (19)	−0.018 (2)	−0.0209 (15)
C18	0.090 (3)	0.0569 (17)	0.112 (3)	−0.0276 (17)	−0.037 (2)	−0.0142 (18)
C19	0.0525 (17)	0.0681 (18)	0.116 (3)	−0.0219 (14)	−0.0125 (17)	−0.0174 (19)
C20	0.0474 (14)	0.0579 (14)	0.0708 (18)	−0.0168 (11)	0.0004 (12)	−0.0174 (13)

Geometric parameters (Å, °)

Br—C4	1.899 (3)	C10—C11	1.377 (4)
S—O2	1.489 (2)	C10—H10	0.9300
S—C1	1.775 (2)	C11—C12	1.366 (5)
S—C15	1.789 (2)	C11—H11	0.9300
O1—C7	1.366 (3)	C12—C13	1.371 (5)

O1—C8	1.377 (3)	C12—H12	0.9300
C1—C8	1.365 (4)	C13—C14	1.377 (5)
C1—C2	1.444 (3)	C13—H13	0.9300
C2—C3	1.385 (4)	C14—H14	0.9300
C2—C7	1.391 (3)	C15—C16	1.379 (4)
C3—C4	1.376 (3)	C15—C20	1.383 (3)
C3—H3	0.9300	C16—C17	1.390 (5)
C4—C5	1.393 (4)	C16—H16	0.9300
C5—C6	1.366 (4)	C17—C18	1.359 (5)
C5—H5	0.9300	C17—H17	0.9300
C6—C7	1.378 (4)	C18—C19	1.361 (5)
C6—H6	0.9300	C18—H18	0.9300
C8—C9	1.459 (4)	C19—C20	1.370 (4)
C9—C10	1.379 (4)	C19—H19	0.9300
C9—C14	1.389 (4)	C20—H20	0.9300
O2—S—C1	106.63 (11)	C11—C10—H10	119.7
O2—S—C15	106.87 (13)	C9—C10—H10	119.7
C1—S—C15	97.41 (10)	C12—C11—C10	120.9 (3)
C7—O1—C8	107.23 (18)	C12—C11—H11	119.6
C8—C1—C2	107.74 (19)	C10—C11—H11	119.6
C8—C1—S	127.74 (19)	C11—C12—C13	119.3 (3)
C2—C1—S	124.50 (18)	C11—C12—H12	120.3
C3—C2—C7	119.5 (2)	C13—C12—H12	120.3
C3—C2—C1	136.0 (2)	C12—C13—C14	120.3 (3)
C7—C2—C1	104.5 (2)	C12—C13—H13	119.9
C4—C3—C2	117.2 (2)	C14—C13—H13	119.9
C4—C3—H3	121.4	C13—C14—C9	120.8 (3)
C2—C3—H3	121.4	C13—C14—H14	119.6
C3—C4—C5	122.7 (3)	C9—C14—H14	119.6
C3—C4—Br	118.6 (2)	C16—C15—C20	120.5 (3)
C5—C4—Br	118.8 (2)	C16—C15—S	121.3 (2)
C6—C5—C4	120.3 (2)	C20—C15—S	118.1 (2)
C6—C5—H5	119.9	C15—C16—C17	118.8 (3)
C4—C5—H5	119.9	C15—C16—H16	120.6
C5—C6—C7	117.3 (2)	C17—C16—H16	120.6
C5—C6—H6	121.4	C18—C17—C16	120.1 (3)
C7—C6—H6	121.4	C18—C17—H17	120.0
O1—C7—C6	126.1 (2)	C16—C17—H17	120.0
O1—C7—C2	110.8 (2)	C17—C18—C19	120.9 (3)
C6—C7—C2	123.0 (3)	C17—C18—H18	119.5
C1—C8—O1	109.7 (2)	C19—C18—H18	119.5
C1—C8—C9	135.1 (2)	C18—C19—C20	120.3 (3)
O1—C8—C9	115.3 (2)	C18—C19—H19	119.8
C10—C9—C14	118.1 (3)	C20—C19—H19	119.8
C10—C9—C8	122.2 (2)	C19—C20—C15	119.3 (3)
C14—C9—C8	119.6 (2)	C19—C20—H20	120.3
C11—C10—C9	120.6 (3)	C15—C20—H20	120.3

O2—S—C1—C8	−152.9 (2)	C7—O1—C8—C1	−1.4 (3)
C15—S—C1—C8	96.9 (2)	C7—O1—C8—C9	179.5 (2)
O2—S—C1—C2	25.7 (2)	C1—C8—C9—C10	16.4 (5)
C15—S—C1—C2	−84.4 (2)	O1—C8—C9—C10	−164.9 (2)
C8—C1—C2—C3	179.1 (3)	C1—C8—C9—C14	−163.9 (3)
S—C1—C2—C3	0.2 (4)	O1—C8—C9—C14	14.8 (4)
C8—C1—C2—C7	0.1 (3)	C14—C9—C10—C11	0.3 (5)
S—C1—C2—C7	−178.80 (18)	C8—C9—C10—C11	−180.0 (3)
C7—C2—C3—C4	0.5 (4)	C9—C10—C11—C12	0.1 (5)
C1—C2—C3—C4	−178.4 (3)	C10—C11—C12—C13	−0.6 (6)
C2—C3—C4—C5	−0.4 (4)	C11—C12—C13—C14	0.6 (6)
C2—C3—C4—Br	179.02 (18)	C12—C13—C14—C9	−0.1 (6)
C3—C4—C5—C6	−0.3 (4)	C10—C9—C14—C13	−0.3 (5)
Br—C4—C5—C6	−179.7 (2)	C8—C9—C14—C13	179.9 (3)
C4—C5—C6—C7	0.8 (4)	O2—S—C15—C16	−14.1 (2)
C8—O1—C7—C6	−178.7 (3)	C1—S—C15—C16	95.8 (2)
C8—O1—C7—C2	1.5 (3)	O2—S—C15—C20	162.89 (18)
C5—C6—C7—O1	179.6 (3)	C1—S—C15—C20	−87.2 (2)
C5—C6—C7—C2	−0.7 (4)	C20—C15—C16—C17	0.9 (4)
C3—C2—C7—O1	179.8 (2)	S—C15—C16—C17	177.9 (2)
C1—C2—C7—O1	−1.0 (3)	C15—C16—C17—C18	0.3 (4)
C3—C2—C7—C6	0.0 (4)	C16—C17—C18—C19	−1.2 (5)
C1—C2—C7—C6	179.2 (3)	C17—C18—C19—C20	0.7 (5)
C2—C1—C8—O1	0.9 (3)	C18—C19—C20—C15	0.5 (5)
S—C1—C8—O1	179.67 (17)	C16—C15—C20—C19	−1.3 (4)
C2—C1—C8—C9	179.6 (3)	S—C15—C20—C19	−178.4 (2)
S—C1—C8—C9	−1.6 (4)		