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5-Bromo-2,4,6-trimethyl-3-methylsulfinyl-1-benzofuran

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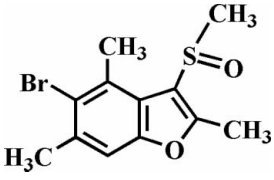
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; disorder in main residue; R factor = 0.042; wR factor = 0.112; data-to-parameter ratio = 13.8.

In the title compound, $C_{12}H_{13}BrO_2S$, there are two symmetry-independent molecules, *A* and *B*, in the asymmetric unit. The crystal studied was an inversion twin with a 0.70 (2):0.30 (2) domain ratio. The methylsulfinyl group in molecule *B* is disordered over two positions with site-occupancy factors fixed at 0.6 and 0.4. The crystal structure is stabilized by C—H···O hydrogen bonds and intermolecular C—H···π interactions. In addition, the crystal structure exhibits C—Br···π interactions, with C—Br···*Cg*1 = 3.646 (8) Å where *Cg*1 is the centroid of the furan ring.

Related literature

For the crystal structures of similar 3-methylsulfinyl-1-benzofuran compounds, see: Choi *et al.* (2007*a*,*b*).



Experimental

Crystal data

$C_{12}H_{13}BrO_2S$
M_r = 301.19
Monoclinic, *Cc*
a = 28.128 (4) Å
b = 11.013 (1) Å
c = 8.052 (1) Å
β = 102.290 (2)°
V = 2437.1 (5) Å^3
Z = 8
Mo *K*α radiation
μ = 3.53 mm^{-1}
T = 298 (2) K
0.40 × 0.40 × 0.30 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1999)
T_{min} = 0.269, T_{max} = 0.345
7147 measured reflections
4360 independent reflections
3737 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.112
S = 1.09
4360 reflections
317 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.90 e Å^{-3}
Absolute structure: Flack (1983), 1736 Friedel pairs
Flack parameter: 0.70 (2)

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

*Cg*2 is the centroid of the C2–C7 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*A*···O2^{i}	0.96	2.34	3.298 (9)	179
C12—H12*C*···O4*B*^{ii}	0.96	2.42	3.168 (12)	134
C24*A*—H24*A*···O4*A*^{iii}	0.96	2.35	3.199 (13)	148
C9—H9*A*···*Cg*2^{iii}	0.96	3.30	3.943 (9)	124

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

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2545).

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supplementary materials

Acta Cryst. (2008). E**64**, o2117 [**doi:10.1107/S1600536808032467**]

5-Bromo-2,4,6-trimethyl-3-methylsulfinyl-1-benzofuran

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Comment

This work is related to our previous communications on the synthesis and structure of 3-methylsulfinyl-1-benzofuran analogues, *viz*. 5-bromo-2-methyl-3-methylsulfinyl-1-benzofuran (Choi *et al.*, 2007*a*) and 2,5-dimethyl-3-methylsulfinyl-1-benzofuran (Choi *et al.*, 2007*b*). Here we report the crystal structure of the title compound, 5-bromo-2,4,6-trimethyl-3-methylsulfinyl-1-benzofuran which crystallizes with two unique molecules, A & B in the asymmetric unit (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.012 (6) Å for A and 0.0022 (7) Å for B, respectively, from the least-squares plane defined by the nine constituent atoms. The crystal studied was an inversion twin with a 0.70 (2):0.30 (2) domain ratio. The O4=S2—C24H_3 group in molecule B is disordered over two positions with site-occupancy factors fixed at 0.4 (for atoms labelled A) and 0.6 (for atoms labelled B) in Fig. 1. The crystal structure is stabilized by three intermolecular C—H···O hydrogen bonds (Fig. 2 and Table 1; symmetry codes as in Fig. 2). The molecular packing (Fig. 2) is further stabilized by a intermolecular C—H···π interactions, with a C9—H9A···*Cg*2^{iii} separation of 3.30 Å (Fig. 2 and Table 1; *Cg*2 is the centroid of the C2—C7 benzene ring, symmetry code as in Fig. 2). In addition, the molecular packing exhibits an intermolecular C—Br···π interaction between the Br atom and the furan ring, with a C16—Br2···*Cg*1^{iv} separation of 3.646 (8) Å (Fig. 2; *Cg*1 is the centroid of C13—C14/C19/O3/C20 furan ring, symmetry code as in Fig. 2).

Experimental

3-Chloroperoxybenzoic acid, 77% (197 mg, 0.88 mmol) was added in small portions to a stirred solution of 3-methylsulfanyl-2-phenyl-1-benzofuran (228 mg, 0.8 mmol) in dichloromethane (30 ml) at 273 K. After stirring for 3 h at room temperature, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated under vacuum. The residue was purified by column chromatography (ethyl acetate) to afford the title compound as a colorless solid [yield 79%, m.p. 400–401 K; R_f = 0.61 (ethyl acetate)]. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in acetone at room temperature. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.51 (s, 3H), 2.68 (s, 3H), 2.83 (s, 3H), 2.95 (s, 3H), 7.21 (s, 1H); EI—MS 302 [*M*+2], 300 [M^+].

Refinement

All H atoms were geometrically positioned and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms, 0.96 Å for methyl H atoms, respectively, and with U_{iso}(H) = 1.2Ueq(C) for aromatic H atoms and 1.5Ueq(C) for methyl H atoms.

The crystal studied was an inversion twin with a 0.70 (2):0.30 (2) domain ratio. The O4=S2—C24H_3 group in molecule B is disordered over two positions with site-occupancy factors fixed at 0.40 (for atoms labelled A) and 0.60 (for atoms labelled B) in the final refinement. Atomic and anisotropic displacement parameters of C24A and C24B were restrained using the commands EXYZ and EADP.

Figures

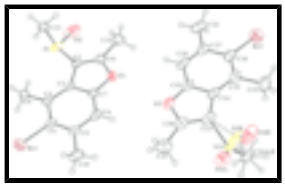


Fig. 1. The asymmetric unit of the title compound, showing displacement ellipsoids drawn at the 30% probability level. Bonds to atoms of the major disorder component of the disordered methylsulfinyl group are drawn as double dashed lines.

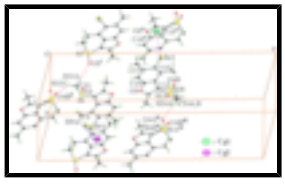


Fig. 2. C—H···O, C—H···π and C—Br···π interactions (dotted lines) in the title compound. *Cg* denotes a ring centroid. [Symmetry code: (i) *x*, -*y*, *z* - 1/2; (ii) *x* - 1/2, -*y* + 1/2, *z* + 1/2; (iii) *x*, -*y* + 1, *z* + 1/2; (iv) *x*, -*y*, *z* - 1/2.]

5-Bromo-2,4,6-trimethyl-3-methylsulfinyl-1-benzofuran

Crystal data

$C_{12}H_{13}BrO_2S$	F_{000} = 1216
M_r = 301.19	D_x = 1.642 Mg m^{-3}
Monoclinic, *Cc*	Melting point = 401–400 K
Hall symbol: C -2yc	Mo *K*α radiation λ = 0.71073 Å
a = 28.128 (4) Å	Cell parameters from 3249 reflections
b = 11.013 (1) Å	θ = 2.9–26.1º
c = 8.052 (1) Å	μ = 3.53 mm^{-1}
β = 102.290 (2)º	*T* = 298 (2) K
V = 2437.1 (5) Å^3	Block, colorless
Z = 8	0.40 × 0.40 × 0.30 mm

Data collection

Bruker SMART CCD diffractometer	4360 independent reflections
Radiation source: fine-focus sealed tube	3737 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
Detector resolution: 10.0 pixels mm^{-1}	θ_{max} = 27.0º
T = 298(2) K	θ_{min} = 1.5º
φ and ω scans	*h* = −35→28
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	*k* = −14→12
T_{min} = 0.269, T_{max} = 0.345	*l* = −9→10
7147 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.112$

$S = 1.09$

4360 reflections

317 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 6.0617P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.45$ e Å^{-3}

$\Delta\rho_{min} = -0.90$ e Å^{-3}

Extinction correction: none

Absolute structure: Flack (1983), 1736 Friedel pairs

Flack parameter: 0.70 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
Br1	0.21922 (3)	0.71294 (6)	0.66785 (9)	0.0659 (2)	
Br2	0.50554 (3)	−0.18594 (8)	0.33653 (9)	0.0854 (3)	
S1	0.17522 (7)	0.15294 (19)	0.6928 (2)	0.0577 (5)	
S2A	0.5460 (2)	0.4107 (6)	0.3537 (10)	0.0523 (16)	0.40
O4A	0.5400 (5)	0.4962 (11)	0.2811 (18)	0.067 (4)	0.40
C24A	0.5884 (2)	0.3483 (7)	0.5252 (9)	0.0616 (19)	0.40
H24A	0.5790	0.3678	0.6298	0.092*	0.40
H24B	0.6201	0.3813	0.5265	0.092*	0.40
H24C	0.5893	0.2617	0.5126	0.092*	0.40
S2B	0.5499 (2)	0.3607 (6)	0.3149 (7)	0.081 (2)	0.60
O4B	0.5586 (3)	0.3734 (12)	0.1963 (14)	0.099 (4)	0.60
C24B	0.5884 (2)	0.3483 (7)	0.5252 (9)	0.0616 (19)	0.60
H24D	0.5945	0.4278	0.5740	0.092*	0.60
H24E	0.6187	0.3109	0.5173	0.092*	0.60
H24F	0.5724	0.2996	0.5957	0.092*	0.60
O1	0.30391 (17)	0.2256 (4)	0.5845 (6)	0.0485 (11)	
O2	0.1830 (2)	0.0201 (6)	0.7294 (9)	0.097 (3)	
O3	0.42047 (19)	0.2988 (4)	0.4257 (7)	0.0614 (14)	
C1	0.2298 (2)	0.2149 (5)	0.6484 (9)	0.0366 (15)	
C2	0.2427 (2)	0.3433 (5)	0.6439 (7)	0.0354 (12)	
C3	0.2204 (3)	0.4548 (5)	0.6687 (10)	0.0403 (12)	

C4	0.2487 (2)	0.5563 (5)	0.6445 (8)	0.0413 (14)
C5	0.2937 (3)	0.5551 (5)	0.6073 (8)	0.0462 (16)
C6	0.3146 (2)	0.4447 (6)	0.5866 (9)	0.0467 (15)
H6	0.3451	0.4398	0.5603	0.056*
C7	0.2887 (2)	0.3417 (5)	0.6061 (8)	0.0412 (14)
C8	0.2665 (3)	0.1512 (6)	0.6131 (9)	0.0485 (17)
C9	0.1720 (2)	0.4603 (6)	0.7129 (10)	0.0507 (17)
H9A	0.1729	0.5172	0.8038	0.076*
H9B	0.1635	0.3814	0.7483	0.076*
H9C	0.1481	0.4858	0.6154	0.076*
C10	0.3211 (3)	0.6695 (7)	0.5822 (11)	0.075 (3)
H10A	0.3019	0.7171	0.4926	0.112*
H10B	0.3514	0.6482	0.5526	0.112*
H10C	0.3275	0.7158	0.6856	0.112*
C11	0.2774 (3)	0.0181 (6)	0.5924 (12)	0.071 (3)
H11A	0.2569	−0.0305	0.6468	0.107*
H11B	0.3109	0.0022	0.6436	0.107*
H11C	0.2715	−0.0017	0.4737	0.107*
C12	0.1395 (2)	0.1660 (6)	0.4814 (9)	0.0512 (15)
H12A	0.1524	0.1130	0.4072	0.077*
H12B	0.1405	0.2483	0.4431	0.077*
H12C	0.1065	0.1437	0.4806	0.077*
C13	0.4940 (3)	0.3120 (8)	0.3690 (11)	0.064 (2)
C14	0.4821 (3)	0.1834 (6)	0.3652 (8)	0.0507 (17)
C15	0.5031 (3)	0.0711 (7)	0.3397 (11)	0.0552 (16)
C16	0.4782 (3)	−0.0334 (7)	0.3580 (10)	0.0565 (19)
C17	0.4301 (3)	−0.0306 (7)	0.4013 (10)	0.059 (2)
C18	0.4098 (3)	0.0785 (7)	0.4214 (10)	0.060 (2)
H18	0.3788	0.0834	0.4447	0.072*
C19	0.4362 (2)	0.1824 (7)	0.4064 (9)	0.0511 (17)
C20	0.4563 (3)	0.3746 (8)	0.4063 (11)	0.065 (2)
C21	0.5530 (3)	0.0662 (10)	0.2927 (12)	0.084 (3)
H21A	0.5767	0.0361	0.3877	0.126*
H21B	0.5622	0.1462	0.2639	0.126*
H21C	0.5515	0.0131	0.1971	0.126*
C22	0.4033 (3)	−0.1465 (8)	0.4158 (11)	0.079 (3)
H22A	0.3716	−0.1282	0.4355	0.118*
H22B	0.4211	−0.1936	0.5089	0.118*
H22C	0.4001	−0.1920	0.3123	0.118*
C23	0.4473 (4)	0.5046 (8)	0.4241 (14)	0.085 (3)
H23A	0.4274	0.5347	0.3204	0.128*
H23B	0.4778	0.5473	0.4477	0.128*
H23C	0.4309	0.5168	0.5158	0.128*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0783 (5)	0.0482 (3)	0.0674 (4)	0.0096 (4)	0.0070 (3)	−0.0056 (3)

Br2	0.0915 (7)	0.0859 (5)	0.0720 (6)	0.0122 (5)	0.0022 (5)	−0.0039 (4)
S1	0.0518 (11)	0.0758 (13)	0.0445 (10)	−0.0326 (9)	0.0081 (8)	0.0092 (8)
S2A	0.026 (2)	0.063 (4)	0.063 (4)	−0.006 (2)	0.000 (2)	0.000 (3)
O4A	0.067 (8)	0.048 (6)	0.085 (10)	−0.015 (6)	0.011 (7)	0.037 (6)
C24A	0.044 (4)	0.080 (4)	0.056 (5)	−0.009 (3)	−0.001 (3)	−0.005 (3)
S2B	0.075 (3)	0.112 (5)	0.053 (3)	−0.062 (3)	0.006 (2)	0.018 (3)
O4B	0.065 (6)	0.163 (11)	0.075 (7)	−0.021 (6)	0.027 (5)	0.026 (7)
C24B	0.044 (4)	0.080 (4)	0.056 (5)	−0.009 (3)	−0.001 (3)	−0.005 (3)
O1	0.040 (3)	0.041 (2)	0.066 (3)	0.0064 (17)	0.013 (2)	0.0089 (19)
O2	0.091 (5)	0.082 (4)	0.103 (5)	−0.044 (4)	−0.015 (4)	0.058 (4)
O3	0.038 (3)	0.068 (3)	0.075 (4)	−0.005 (2)	0.005 (2)	0.004 (2)
C1	0.035 (4)	0.040 (3)	0.036 (3)	−0.013 (2)	0.009 (3)	0.006 (2)
C2	0.024 (3)	0.046 (3)	0.033 (3)	−0.008 (2)	0.000 (2)	0.002 (2)
C3	0.029 (3)	0.055 (3)	0.034 (3)	−0.006 (3)	0.000 (2)	−0.001 (3)
C4	0.041 (4)	0.045 (3)	0.036 (3)	−0.009 (3)	0.005 (3)	0.000 (2)
C5	0.049 (4)	0.044 (3)	0.043 (4)	−0.013 (3)	0.003 (3)	−0.002 (2)
C6	0.030 (3)	0.059 (4)	0.052 (4)	−0.014 (3)	0.010 (3)	0.004 (3)
C7	0.037 (3)	0.041 (3)	0.044 (3)	−0.005 (2)	0.006 (3)	0.004 (2)
C8	0.051 (4)	0.043 (4)	0.045 (4)	−0.006 (3)	−0.003 (3)	0.003 (3)
C9	0.041 (4)	0.058 (4)	0.055 (4)	0.000 (3)	0.015 (3)	−0.008 (3)
C10	0.069 (6)	0.061 (4)	0.090 (6)	−0.035 (4)	0.010 (5)	0.016 (4)
C11	0.078 (6)	0.045 (4)	0.082 (6)	−0.005 (4)	−0.002 (5)	0.007 (4)
C12	0.038 (4)	0.058 (3)	0.056 (4)	−0.012 (3)	0.008 (3)	−0.007 (3)
C13	0.043 (5)	0.084 (5)	0.058 (5)	−0.020 (3)	−0.004 (4)	0.016 (4)
C14	0.042 (4)	0.069 (4)	0.040 (4)	−0.021 (3)	0.004 (3)	0.009 (3)
C15	0.043 (4)	0.087 (5)	0.035 (3)	−0.005 (4)	0.008 (3)	0.008 (4)
C16	0.046 (4)	0.070 (4)	0.046 (4)	−0.001 (3)	−0.005 (3)	0.003 (3)
C17	0.047 (5)	0.066 (4)	0.061 (5)	−0.017 (3)	0.003 (4)	0.011 (3)
C18	0.038 (4)	0.072 (5)	0.067 (5)	−0.022 (3)	0.007 (3)	0.009 (3)
C19	0.029 (3)	0.070 (4)	0.051 (4)	−0.015 (3)	0.001 (3)	0.007 (3)
C20	0.047 (4)	0.081 (5)	0.066 (5)	−0.014 (4)	0.006 (4)	0.006 (4)
C21	0.055 (5)	0.132 (9)	0.069 (6)	−0.004 (5)	0.023 (4)	−0.006 (5)
C22	0.073 (6)	0.089 (6)	0.068 (5)	−0.039 (5)	0.004 (4)	0.014 (4)
C23	0.096 (7)	0.070 (6)	0.089 (7)	−0.019 (5)	0.018 (6)	−0.001 (5)

Geometric parameters (Å, °)

Br1—C4	1.941 (6)	C9—H9C	0.9600
Br2—C16	1.871 (8)	C10—H10A	0.9600
S1—O2	1.499 (7)	C10—H10B	0.9600
S1—C1	1.783 (6)	C10—H10C	0.9600
S1—C12	1.788 (7)	C11—H11A	0.9600
S2A—O4A	1.102 (13)	C11—H11B	0.9600
S2A—C24A	1.761 (10)	C11—H11C	0.9600
S2A—C13	1.848 (10)	C12—H12A	0.9600
C24A—H24A	0.9600	C12—H12B	0.9600
C24A—H24B	0.9600	C12—H12C	0.9600
C24A—H24C	0.9600	C13—C20	1.352 (14)
S2B—O4B	1.044 (11)	C13—C14	1.454 (10)

S2B—C13	1.801 (10)	C14—C19	1.401 (10)
O1—C7	1.372 (7)	C14—C15	1.405 (11)
O1—C8	1.392 (9)	C15—C16	1.372 (10)
O3—C20	1.344 (10)	C15—C21	1.530 (12)
O3—C19	1.375 (10)	C16—C17	1.469 (12)
C1—C8	1.328 (10)	C17—C18	1.355 (11)
C1—C2	1.462 (7)	C17—C22	1.498 (10)
C2—C7	1.389 (9)	C18—C19	1.383 (9)
C2—C3	1.413 (9)	C18—H18	0.9300
C3—C4	1.411 (9)	C20—C23	1.466 (12)
C3—C9	1.479 (11)	C21—H21A	0.9600
C4—C5	1.362 (10)	C21—H21B	0.9600
C5—C6	1.375 (9)	C21—H21C	0.9600
C5—C10	1.513 (8)	C22—H22A	0.9600
C6—C7	1.375 (8)	C22—H22B	0.9600
C6—H6	0.9300	C22—H22C	0.9600
C8—C11	1.514 (10)	C23—H23A	0.9600
C9—H9A	0.9600	C23—H23B	0.9600
C9—H9B	0.9600	C23—H23C	0.9600
O2—S1—C1	108.4 (4)	S1—C12—H12A	109.5
O2—S1—C12	107.1 (4)	S1—C12—H12B	109.5
C1—S1—C12	96.4 (3)	H12A—C12—H12B	109.5
O4A—S2A—C24A	138.7 (9)	S1—C12—H12C	109.5
O4A—S2A—C13	120.3 (8)	H12A—C12—H12C	109.5
C24A—S2A—C13	97.6 (5)	H12B—C12—H12C	109.5
O4B—S2B—C13	130.4 (7)	C20—C13—C14	108.3 (7)
C7—O1—C8	105.1 (5)	C20—C13—S2B	132.0 (7)
C20—O3—C19	107.4 (7)	C14—C13—S2B	119.6 (8)
C8—C1—C2	107.2 (5)	C20—C13—S2A	112.6 (7)
C8—C1—S1	125.6 (4)	C14—C13—S2A	138.9 (8)
C2—C1—S1	127.2 (5)	C19—C14—C15	117.8 (6)
C7—C2—C3	120.3 (5)	C19—C14—C13	102.9 (7)
C7—C2—C1	104.1 (5)	C15—C14—C13	139.3 (7)
C3—C2—C1	135.7 (6)	C16—C15—C14	118.8 (7)
C4—C3—C2	112.8 (7)	C16—C15—C21	120.9 (8)
C4—C3—C9	125.2 (6)	C14—C15—C21	120.3 (7)
C2—C3—C9	122.0 (5)	C15—C16—C17	121.8 (7)
C5—C4—C3	127.0 (6)	C15—C16—Br2	120.9 (6)
C5—C4—Br1	117.8 (4)	C17—C16—Br2	117.3 (5)
C3—C4—Br1	115.2 (5)	C18—C17—C16	118.6 (6)
C4—C5—C6	118.4 (5)	C18—C17—C22	121.0 (8)
C4—C5—C10	123.1 (6)	C16—C17—C22	120.3 (8)
C6—C5—C10	118.5 (7)	C17—C18—C19	118.4 (7)
C7—C6—C5	117.7 (6)	C17—C18—H18	120.8
C7—C6—H6	121.1	C19—C18—H18	120.8
C5—C6—H6	121.1	O3—C19—C18	124.8 (7)
O1—C7—C6	124.5 (6)	O3—C19—C14	110.7 (6)
O1—C7—C2	111.7 (5)	C18—C19—C14	124.5 (8)
C6—C7—C2	123.7 (6)	O3—C20—C13	110.6 (7)

C1—C8—O1	112.0 (5)	O3—C20—C23	116.5 (8)
C1—C8—C11	136.1 (7)	C13—C20—C23	132.8 (8)
O1—C8—C11	111.9 (7)	C15—C21—H21A	109.5
C3—C9—H9A	109.5	C15—C21—H21B	109.5
C3—C9—H9B	109.5	H21A—C21—H21B	109.5
H9A—C9—H9B	109.5	C15—C21—H21C	109.5
C3—C9—H9C	109.5	H21A—C21—H21C	109.5
H9A—C9—H9C	109.5	H21B—C21—H21C	109.5
H9B—C9—H9C	109.5	C17—C22—H22A	109.5
C5—C10—H10A	109.5	C17—C22—H22B	109.5
C5—C10—H10B	109.5	H22A—C22—H22B	109.5
H10A—C10—H10B	109.5	C17—C22—H22C	109.5
C5—C10—H10C	109.5	H22A—C22—H22C	109.5
H10A—C10—H10C	109.5	H22B—C22—H22C	109.5
H10B—C10—H10C	109.5	C20—C23—H23A	109.5
C8—C11—H11A	109.5	C20—C23—H23B	109.5
C8—C11—H11B	109.5	H23A—C23—H23B	109.5
H11A—C11—H11B	109.5	C20—C23—H23C	109.5
C8—C11—H11C	109.5	H23A—C23—H23C	109.5
H11A—C11—H11C	109.5	H23B—C23—H23C	109.5
H11B—C11—H11C	109.5		
O2—S1—C1—C8	14.9 (8)	O4A—S2A—C13—C14	139.6 (14)
C12—S1—C1—C8	−95.6 (7)	C24A—S2A—C13—C14	−57.7 (11)
O2—S1—C1—C2	−165.0 (6)	O4A—S2A—C13—S2B	115.7 (17)
C12—S1—C1—C2	84.5 (6)	C24A—S2A—C13—S2B	−81.5 (10)
C8—C1—C2—C7	−0.7 (7)	C20—C13—C14—C19	−1.2 (9)
S1—C1—C2—C7	179.2 (5)	S2B—C13—C14—C19	−177.5 (6)
C8—C1—C2—C3	179.8 (8)	S2A—C13—C14—C19	173.1 (9)
S1—C1—C2—C3	−0.3 (11)	C20—C13—C14—C15	−178.4 (9)
C7—C2—C3—C4	2.2 (9)	S2B—C13—C14—C15	5.3 (14)
C1—C2—C3—C4	−178.3 (7)	S2A—C13—C14—C15	−4.2 (18)
C7—C2—C3—C9	−178.6 (7)	C19—C14—C15—C16	−0.7 (11)
C1—C2—C3—C9	0.9 (12)	C13—C14—C15—C16	176.2 (9)
C2—C3—C4—C5	−2.3 (11)	C19—C14—C15—C21	179.4 (7)
C9—C3—C4—C5	178.5 (7)	C13—C14—C15—C21	−3.6 (15)
C2—C3—C4—Br1	177.7 (5)	C14—C15—C16—C17	0.7 (11)
C9—C3—C4—Br1	−1.5 (10)	C21—C15—C16—C17	−179.4 (8)
C3—C4—C5—C6	1.5 (11)	C14—C15—C16—Br2	−176.9 (6)
Br1—C4—C5—C6	−178.6 (4)	C21—C15—C16—Br2	3.0 (10)
C3—C4—C5—C10	179.5 (7)	C15—C16—C17—C18	0.9 (11)
Br1—C4—C5—C10	−0.5 (9)	Br2—C16—C17—C18	178.6 (5)
C4—C5—C6—C7	−0.5 (9)	C15—C16—C17—C22	178.4 (7)
C10—C5—C6—C7	−178.6 (6)	Br2—C16—C17—C22	−3.9 (10)
C8—O1—C7—C6	−178.8 (6)	C16—C17—C18—C19	−2.4 (11)
C8—O1—C7—C2	−1.2 (7)	C22—C17—C18—C19	−179.9 (7)
C5—C6—C7—O1	177.9 (6)	C20—O3—C19—C18	178.4 (7)
C5—C6—C7—C2	0.6 (10)	C20—O3—C19—C14	−3.1 (8)
C3—C2—C7—O1	−179.2 (6)	C17—C18—C19—O3	−179.2 (7)
C1—C2—C7—O1	1.2 (7)	C17—C18—C19—C14	2.6 (11)

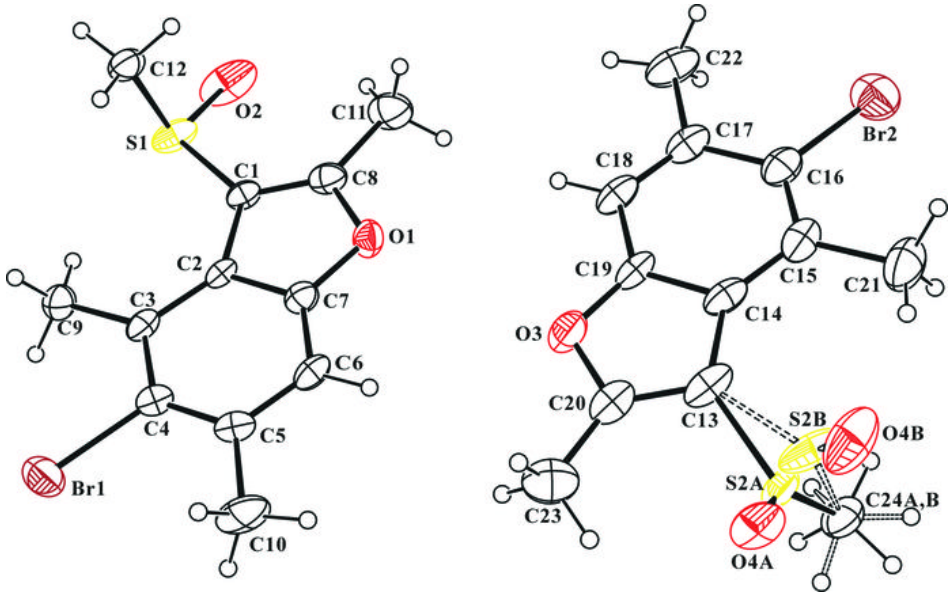
C3—C2—C7—C6	−1.5 (10)	C15—C14—C19—O3	−179.4 (6)
C1—C2—C7—C6	178.8 (6)	C13—C14—C19—O3	2.6 (8)
C2—C1—C8—O1	0.0 (8)	C15—C14—C19—C18	−1.0 (11)
S1—C1—C8—O1	−179.9 (5)	C13—C14—C19—C18	−178.9 (7)
C2—C1—C8—C11	−178.3 (8)	C19—O3—C20—C13	2.2 (9)
S1—C1—C8—C11	1.8 (13)	C19—O3—C20—C23	−179.3 (7)
C7—O1—C8—C1	0.7 (8)	C14—C13—C20—O3	−0.6 (10)
C7—O1—C8—C11	179.5 (6)	S2B—C13—C20—O3	175.1 (7)
O4B—S2B—C13—C20	−94.9 (16)	S2A—C13—C20—O3	−176.5 (6)
O4B—S2B—C13—C14	80.3 (15)	C14—C13—C20—C23	−178.7 (9)
O4B—S2B—C13—S2A	−117.4 (19)	S2B—C13—C20—C23	−3.1 (16)
O4A—S2A—C13—C20	−46.3 (13)	S2A—C13—C20—C23	5.3 (13)
C24A—S2A—C13—C20	116.4 (7)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12A···O2^{i}	0.96	2.34	3.298 (9)	179
C12—H12C···O4B^{ii}	0.96	2.42	3.168 (12)	134
C24A—H24A···O4A^{iii}	0.96	2.35	3.199 (13)	148
C9—H9A···Cg2^{iii}	0.96	3.30	3.943 (9)	124

Symmetry codes: (i) *x*, −*y*, *z*−1/2; (ii) *x*−1/2, −*y*+1/2, *z*+1/2; (iii) *x*, −*y*+1, *z*+1/2.

Fig. 1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O2
S1
C11
C1
C8
O1
C2
C3
C7
C9
C6
C4
C5
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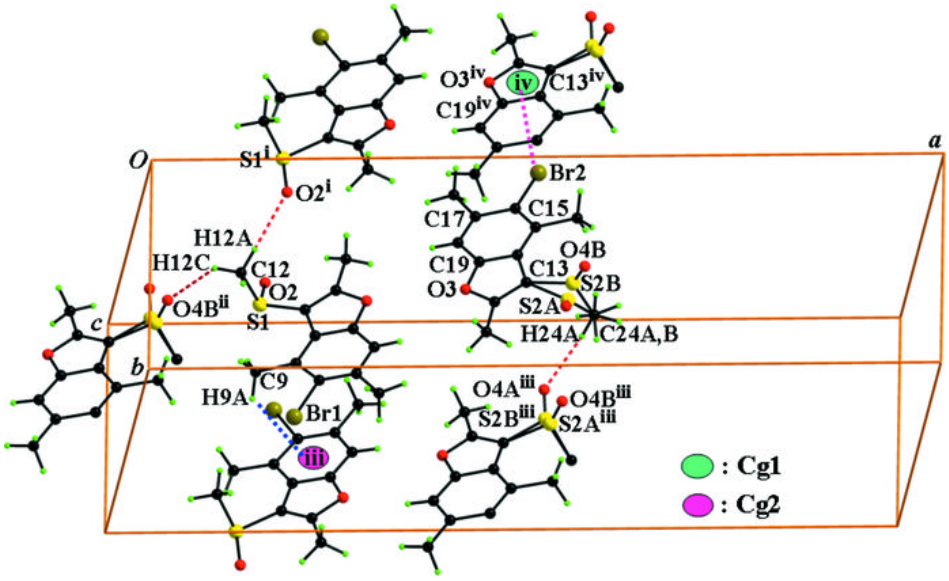
C10
C22
C17
Br2
C18
C16
C19
C15
C21
C14
O3
C20
C13
S2B
O4B
S2A
C24A,B
C23
O4A

Fig. 2


O
a
c
b
S1i
O2i
H12A
H12C
C12
O2
O4Bii
S1
C9
H9A
Br1
iii
O3iv
iv
C13iv
C19iv
Br2
C17
C15
C19
O4B
C13
O3
S2B
S2A
H24A
C24A,B
O4Aiii
O4Biii
S2Biii
S2Aiii
: Cg1
: Cg2