Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Hong Dae Choi,[a] Pil Ja Seo,[a] Byeng Wha Son[b] and Uk Lee[b]*

[a]Department of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, and [b]Department of Chemistry, Pukyong National University, 599-1 Daeyeon 3-dong Nam-gu, Busan 608-737, Republic of Korea
Correspondence e-mail: uklee@pknu.ac.kr

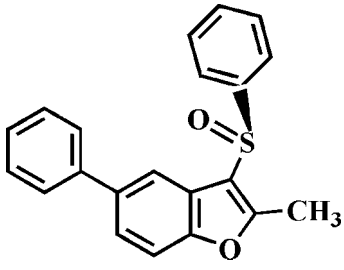
Received 21 May 2007; accepted 2 July 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.111; data-to-parameter ratio = 14.5.

The title compound, $C_{21}H_{16}O_2S$, was prepared by the oxidation of 2-methyl-5-phenyl-3-phenylsulfanyl-1-benzofuran using 3-chloroperbenzoic acid. The O atom and the phenyl group of the phenylsulfinyl substituent lie on opposite sides of the plane of the benzofuran unit. The 3-phenyl group is almost perpendicular to the plane of the benzofuran fragment [80.4 (6)°] and is tilted slightly towards it. The 5-phenyl ring is rotated out of the benzofuran plane with a dihedral angle of 21.0 (1)°. The crystal structure is stabilized by CH_2—H(methyl)$\cdots\pi$(benzofuran) interactions.

Related literature

For the crystal structures of isomers of the title compound, see: Choi, Seo, Kang *et al.* (2006); Choi, Seo, Lee *et al.* (2006).



Experimental

Crystal data

$C_{21}H_{16}O_2S$
M_r = 332.40
Monoclinic, $P2_1/n$
a = 9.8919 (5) Å
b = 9.6229 (5) Å
c = 16.9226 (9) Å
β = 91.685 (1)°
V = 1610.15 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 173 (2) K
0.65 × 0.42 × 0.35 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
8853 measured reflections
3154 independent reflections
2942 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.111
S = 1.16
3154 reflections
218 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2178).

References

Brandenburg, K. (1998). *DIAMOND*. Version 2.1. Crystal Impact GbR, Bonn, Germany.
Bruker (1997). *SMART* (Version 5.631) and *SAINT* (Version 6.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Choi, H. D., Seo, P. J., Kang, B. W., Son, B. W. & Lee, U. (2006). *Acta Cryst.* E**62**, o4796–o4797.
Choi, H. D., Seo, P. J., Lee, H. K., Son, B. W. & Lee, U. (2006). *Acta Cryst.* E**62**, o4480–o4481.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

 doi:10.1107/S1600536807032199

supplementary materials

Acta Cryst. (2007). E**63**, o3423 [**doi:10.1107/S1600536807032199**]

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

H. D. Choi, P. J. Seo, B. W. Son and U. Lee

Comment

As part of our continuing studies on the synthesis and structure of 5-phenyl-1-benzofuran analogues, the crystal structures of 3-methylsulfanyl-2,5-diphenyl-1-benzofuran (Choi, Seo, Kang *et al.*, 2006) and 2-methyl-3-methylsulfinyl-5-phenyl-1-benzofuran (Choi, Seo, Lee *et al.*, 2006) have been described to the literature. Herein we report the molecular and crystal structure of the title compound (I) (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.008 Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle in (I) formed by the plane of the benzofuran ring and the plane of 5-phenyl ring is 21.0 (1)° and phenyl ring(C15—C20) with 80.4 (6)° lies toward benzofuran plane. The molecular packing (Fig. 2) is stabilized by CH_2—H···π interactions between the methyl group and the benzene ring of benzofuran unit, with a C21—H21B···*Cg*1^{i} separation of 2.94 Å. (*Cg*1 is the centroid of the C3—C8 benzene ring, symmetry code as in Fig. 2).

Experimental

3-Chloroperbenzoic acid (77%, 291 mg, 1.30 mmol) was added in small portions to a stirred solution of 2-methyl-5-phenyl-3-phenylsulfanyl-1-benzofuran (379 mg, 1.20 mmol) in dichloromethane (30 ml) at 273 K. After being stirred at room temperature for 1hr, the mixture was washed with saturated sodium bicarbonate solution and the organic layer separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (hexane-ethyl acetate, 1:1 *v*/*v*) to afford the title compound as a colorless solid [yield 78%, m.p. 425–426 K; R_f = 0.51 (hexane-ethyl acetate, 1:1 *v*/*v*)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of title compound (I) in acetone at room temperature.

Refinement

All H atoms were geometrically located in ideal positions and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms and C—H = 0.96 Å for methyl H atoms, and with U_{iso}(H) = 1.2Ueq(C) for aromatic H atoms, and U_{iso}(H) = 1.5Ueq(C) for methyl H atoms. The highest peak in the difference map is 1.21 Å from S and the largest hole is 0.34 Å from S.

Figures

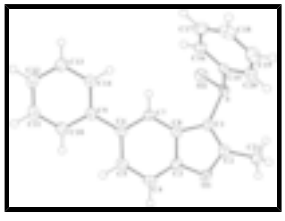


Fig. 1. The molecular structure of (I), showing displacement ellipsoides drawn at the 50% probability level.

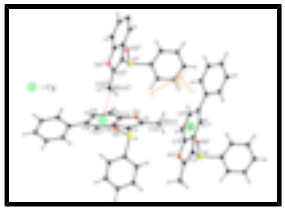


Fig. 2. CH_2—H···π interactions (dashed lines) in (I). *Cg* denotes ring centroid. [Symmetry codes: (i) 1/2 − *x*, 1/2 + *y*, 1/2 − *z*; (ii) 1/2 − *x*, *y* − 1/2, 1/2 − *z*.]

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

Crystal data

$C_{21}H_{16}O_2S$	F_{000} = 696
M_r = 332.40	D_x = 1.371 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -p_2yn	Cell parameters from 6665 reflections
a = 9.8919 (5) Å	θ = 2.4–28.3º
b = 9.6229 (5) Å	μ = 0.21 mm^{-1}
c = 16.9226 (9) Å	*T* = 173 (2) K
β = 91.6850 (10)º	Block, colorless
V = 1610.15 (14) Å^3	0.65 × 0.42 × 0.35 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	3154 independent reflections
Radiation source: fine-focus sealed tube	2942 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.030
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 26.0º
T = 173(2) K	θ_{min} = 2.4º
φ and ω scans	*h* = −12→12
Absorption correction: none	*k* = −11→10
8853 measured reflections	*l* = −17→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H-atom parameters constrained
$wR(F^2)$ = 0.111	$w = 1/[\sigma^2(F_o^2) + (0.042P)^2 + 1.3632P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.16	$(\Delta/\sigma)_{max} < 0.001$
3154 reflections	$\Delta\rho_{max}$ = 0.62 e Å^{-3}
218 parameters	$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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}
S	0.08659 (5)	0.14104 (5)	0.34971 (3)	0.02305 (15)
O1	0.45015 (14)	0.03554 (15)	0.28292 (8)	0.0268 (3)
O2	0.00179 (15)	0.02393 (16)	0.37785 (9)	0.0315 (4)
C1	0.25122 (19)	0.0754 (2)	0.33966 (11)	0.0216 (4)
C2	0.3295 (2)	0.1062 (2)	0.27759 (11)	0.0245 (4)
C3	0.4471 (2)	−0.0421 (2)	0.35150 (11)	0.0228 (4)
C4	0.5483 (2)	−0.1282 (2)	0.38024 (12)	0.0273 (4)
H4	0.6298	−0.1412	0.3528	0.033*
C5	0.5256 (2)	−0.1948 (2)	0.45110 (12)	0.0253 (4)
H5	0.5940	−0.2539	0.4729	0.030*
C6	0.40448 (19)	−0.1783 (2)	0.49227 (11)	0.0213 (4)
C7	0.30329 (19)	−0.0919 (2)	0.46039 (11)	0.0207 (4)
H7	0.2201	−0.0811	0.4864	0.025*
C8	0.32614 (19)	−0.02158 (19)	0.38991 (11)	0.0207 (4)
C9	0.38708 (19)	−0.2488 (2)	0.56998 (11)	0.0219 (4)
C10	0.4633 (2)	−0.3664 (2)	0.59166 (12)	0.0291 (5)
H10	0.5261	−0.4035	0.5558	0.035*
C11	0.4484 (2)	−0.4295 (2)	0.66456 (13)	0.0336 (5)
H11	0.5020	−0.5083	0.6783	0.040*
C12	0.3565 (2)	−0.3791 (2)	0.71746 (13)	0.0340 (5)
H12	0.3464	−0.4227	0.7673	0.041*
C13	0.2792 (2)	−0.2639 (2)	0.69661 (12)	0.0319 (5)
H13	0.2154	−0.2286	0.7323	0.038*
C14	0.2945 (2)	−0.1996 (2)	0.62387 (12)	0.0260 (4)
H14	0.2408	−0.1207	0.6106	0.031*
C15	0.11738 (19)	0.2550 (2)	0.43243 (11)	0.0223 (4)
C16	0.0866 (2)	0.2136 (2)	0.50802 (12)	0.0274 (4)
H16	0.0506	0.1238	0.5175	0.033*
C17	0.1093 (2)	0.3064 (3)	0.56983 (12)	0.0331 (5)
H17	0.0904	0.2792	0.6223	0.040*
C18	0.1594 (2)	0.4382 (2)	0.55570 (13)	0.0337 (5)

H18	0.1739	0.5012	0.5983	0.040*
C19	0.1883 (2)	0.4783 (2)	0.47930 (14)	0.0338 (5)
H19	0.2225	0.5687	0.4696	0.041*
C20	0.1674 (2)	0.3867 (2)	0.41729 (13)	0.0281 (4)
H20	0.1871	0.4137	0.3649	0.034*
C21	0.3119 (2)	0.2026 (2)	0.20968 (12)	0.0317 (5)
H21A	0.2160	0.2266	0.2026	0.047*
H21B	0.3435	0.1577	0.1616	0.047*
H21C	0.3646	0.2873	0.2199	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0224 (3)	0.0228 (3)	0.0238 (3)	0.00043 (18)	−0.00299 (18)	0.00061 (19)
O1	0.0292 (8)	0.0299 (8)	0.0215 (7)	0.0001 (6)	0.0057 (6)	0.0012 (6)
O2	0.0272 (8)	0.0300 (8)	0.0374 (8)	−0.0067 (6)	−0.0001 (6)	−0.0012 (6)
C1	0.0244 (10)	0.0202 (9)	0.0200 (9)	−0.0009 (8)	−0.0010 (7)	−0.0011 (7)
C2	0.0288 (10)	0.0218 (10)	0.0228 (10)	−0.0014 (8)	−0.0005 (8)	−0.0024 (8)
C3	0.0258 (10)	0.0231 (10)	0.0195 (9)	−0.0022 (8)	0.0024 (7)	−0.0018 (7)
C4	0.0234 (10)	0.0305 (11)	0.0284 (10)	0.0027 (8)	0.0069 (8)	−0.0038 (9)
C5	0.0218 (10)	0.0268 (10)	0.0274 (10)	0.0043 (8)	−0.0002 (8)	−0.0029 (8)
C6	0.0230 (9)	0.0205 (9)	0.0204 (9)	−0.0012 (7)	−0.0007 (7)	−0.0024 (7)
C7	0.0209 (9)	0.0219 (9)	0.0194 (9)	−0.0007 (7)	0.0019 (7)	−0.0021 (7)
C8	0.0219 (9)	0.0190 (9)	0.0211 (9)	−0.0007 (7)	−0.0020 (7)	−0.0046 (7)
C9	0.0199 (9)	0.0223 (9)	0.0234 (9)	−0.0023 (7)	−0.0038 (7)	−0.0011 (8)
C10	0.0273 (11)	0.0301 (11)	0.0296 (11)	0.0040 (9)	−0.0010 (8)	0.0014 (9)
C11	0.0335 (12)	0.0316 (12)	0.0353 (12)	0.0056 (9)	−0.0077 (9)	0.0068 (9)
C12	0.0382 (12)	0.0380 (12)	0.0254 (10)	−0.0054 (10)	−0.0034 (9)	0.0097 (9)
C13	0.0335 (11)	0.0373 (12)	0.0250 (10)	−0.0006 (9)	0.0036 (8)	0.0020 (9)
C14	0.0267 (10)	0.0252 (10)	0.0261 (10)	0.0016 (8)	0.0003 (8)	0.0017 (8)
C15	0.0169 (9)	0.0232 (10)	0.0267 (10)	0.0037 (7)	−0.0013 (7)	−0.0019 (8)
C16	0.0226 (10)	0.0311 (11)	0.0287 (10)	0.0008 (8)	0.0034 (8)	0.0020 (9)
C17	0.0245 (11)	0.0491 (14)	0.0257 (10)	0.0069 (10)	0.0022 (8)	−0.0011 (10)
C18	0.0256 (11)	0.0399 (13)	0.0354 (12)	0.0061 (9)	−0.0050 (9)	−0.0141 (10)
C19	0.0345 (12)	0.0260 (11)	0.0405 (12)	−0.0026 (9)	−0.0041 (9)	−0.0032 (9)
C20	0.0295 (11)	0.0254 (10)	0.0296 (11)	−0.0005 (8)	−0.0002 (8)	0.0024 (8)
C21	0.0397 (12)	0.0300 (11)	0.0254 (10)	−0.0016 (9)	0.0028 (9)	0.0045 (9)

Geometric parameters (Å, °)

S—O2	1.491 (2)	C11—C12	1.383 (3)
S—C1	1.760 (2)	C11—H11	0.9500
S—C15	1.797 (2)	C12—C13	1.386 (3)
O1—C2	1.374 (2)	C12—H12	0.9500
O1—C3	1.381 (2)	C13—C14	1.390 (3)
C1—C2	1.356 (3)	C13—H13	0.9500
C1—C8	1.451 (3)	C14—H14	0.9500
C2—C21	1.483 (3)	C15—C16	1.382 (3)
C3—C4	1.377 (3)	C15—C20	1.387 (3)

C3—C8	1.392 (3)	C16—C17	1.388 (3)
C4—C5	1.384 (3)	C16—H16	0.9500
C4—H4	0.9500	C17—C18	1.385 (3)
C5—C6	1.412 (3)	C17—H17	0.9500
C5—H5	0.9500	C18—C19	1.387 (3)
C6—C7	1.397 (3)	C18—H18	0.9500
C6—C9	1.494 (3)	C19—C20	1.381 (3)
C7—C8	1.395 (3)	C19—H19	0.9500
C7—H7	0.9500	C20—H20	0.9500
C9—C14	1.394 (3)	C21—H21A	0.9800
C9—C10	1.403 (3)	C21—H21B	0.9800
C10—C11	1.387 (3)	C21—H21C	0.9800
C10—H10	0.9500		
O2—S—C1	106.89 (9)	C12—C11—H11	119.6
O2—S—C15	107.25 (9)	C10—C11—H11	119.6
C1—S—C15	99.10 (9)	C11—C12—C13	118.9 (2)
C2—O1—C3	106.39 (15)	C11—C12—H12	120.5
C2—C1—C8	107.53 (17)	C13—C12—H12	120.5
C2—C1—S	123.21 (15)	C12—C13—C14	120.5 (2)
C8—C1—S	129.21 (15)	C12—C13—H13	119.7
C1—C2—O1	110.83 (17)	C14—C13—H13	119.7
C1—C2—C21	132.87 (19)	C13—C14—C9	121.25 (19)
O1—C2—C21	116.25 (17)	C13—C14—H14	119.4
C4—C3—O1	126.03 (17)	C9—C14—H14	119.4
C4—C3—C8	123.11 (18)	C16—C15—C20	121.57 (19)
O1—C3—C8	110.86 (17)	C16—C15—S	120.54 (16)
C3—C4—C5	116.71 (18)	C20—C15—S	117.84 (15)
C3—C4—H4	121.6	C15—C16—C17	118.5 (2)
C5—C4—H4	121.6	C15—C16—H16	120.8
C4—C5—C6	122.48 (18)	C17—C16—H16	120.8
C4—C5—H5	118.8	C18—C17—C16	120.6 (2)
C6—C5—H5	118.8	C18—C17—H17	119.7
C7—C6—C5	119.01 (17)	C16—C17—H17	119.7
C7—C6—C9	120.69 (17)	C17—C18—C19	120.0 (2)
C5—C6—C9	120.27 (17)	C17—C18—H18	120.0
C8—C7—C6	119.09 (17)	C19—C18—H18	120.0
C8—C7—H7	120.5	C20—C19—C18	120.0 (2)
C6—C7—H7	120.5	C20—C19—H19	120.0
C3—C8—C7	119.57 (18)	C18—C19—H19	120.0
C3—C8—C1	104.39 (17)	C19—C20—C15	119.3 (2)
C7—C8—C1	136.04 (18)	C19—C20—H20	120.4
C14—C9—C10	117.47 (18)	C15—C20—H20	120.4
C14—C9—C6	121.08 (17)	C2—C21—H21A	109.5
C10—C9—C6	121.45 (18)	C2—C21—H21B	109.5
C11—C10—C9	121.0 (2)	H21A—C21—H21B	109.5
C11—C10—H10	119.5	C2—C21—H21C	109.5
C9—C10—H10	119.5	H21A—C21—H21C	109.5
C12—C11—C10	120.8 (2)	H21B—C21—H21C	109.5

O2—S—C1—C2	137.76 (17)	C2—C1—C8—C7	−179.6 (2)
C15—S—C1—C2	−110.99 (18)	S—C1—C8—C7	−2.3 (3)
O2—S—C1—C8	−39.2 (2)	C7—C6—C9—C14	−20.5 (3)
C15—S—C1—C8	72.04 (19)	C5—C6—C9—C14	157.58 (19)
C8—C1—C2—O1	0.5 (2)	C7—C6—C9—C10	159.74 (19)
S—C1—C2—O1	−177.00 (13)	C5—C6—C9—C10	−22.2 (3)
C8—C1—C2—C21	−176.5 (2)	C14—C9—C10—C11	−1.1 (3)
S—C1—C2—C21	5.9 (3)	C6—C9—C10—C11	178.67 (19)
C3—O1—C2—C1	−0.3 (2)	C9—C10—C11—C12	0.9 (3)
C3—O1—C2—C21	177.31 (17)	C10—C11—C12—C13	−0.2 (3)
C2—O1—C3—C4	−179.52 (19)	C11—C12—C13—C14	−0.4 (3)
C2—O1—C3—C8	−0.1 (2)	C12—C13—C14—C9	0.1 (3)
O1—C3—C4—C5	178.85 (18)	C10—C9—C14—C13	0.6 (3)
C8—C3—C4—C5	−0.5 (3)	C6—C9—C14—C13	−179.17 (18)
C3—C4—C5—C6	0.9 (3)	O2—S—C15—C16	11.79 (18)
C4—C5—C6—C7	0.2 (3)	C1—S—C15—C16	−99.18 (17)
C4—C5—C6—C9	−177.89 (18)	O2—S—C15—C20	−165.60 (15)
C5—C6—C7—C8	−1.7 (3)	C1—S—C15—C20	83.43 (16)
C9—C6—C7—C8	176.44 (17)	C20—C15—C16—C17	−1.3 (3)
C4—C3—C8—C7	−0.9 (3)	S—C15—C16—C17	−178.60 (15)
O1—C3—C8—C7	179.63 (16)	C15—C16—C17—C18	1.2 (3)
C4—C3—C8—C1	179.85 (19)	C16—C17—C18—C19	−0.5 (3)
O1—C3—C8—C1	0.4 (2)	C17—C18—C19—C20	−0.1 (3)
C6—C7—C8—C3	2.0 (3)	C18—C19—C20—C15	0.0 (3)
C6—C7—C8—C1	−179.1 (2)	C16—C15—C20—C19	0.7 (3)
C2—C1—C8—C3	−0.6 (2)	S—C15—C20—C19	178.06 (16)
S—C1—C8—C3	176.78 (15)		

Fig. 1

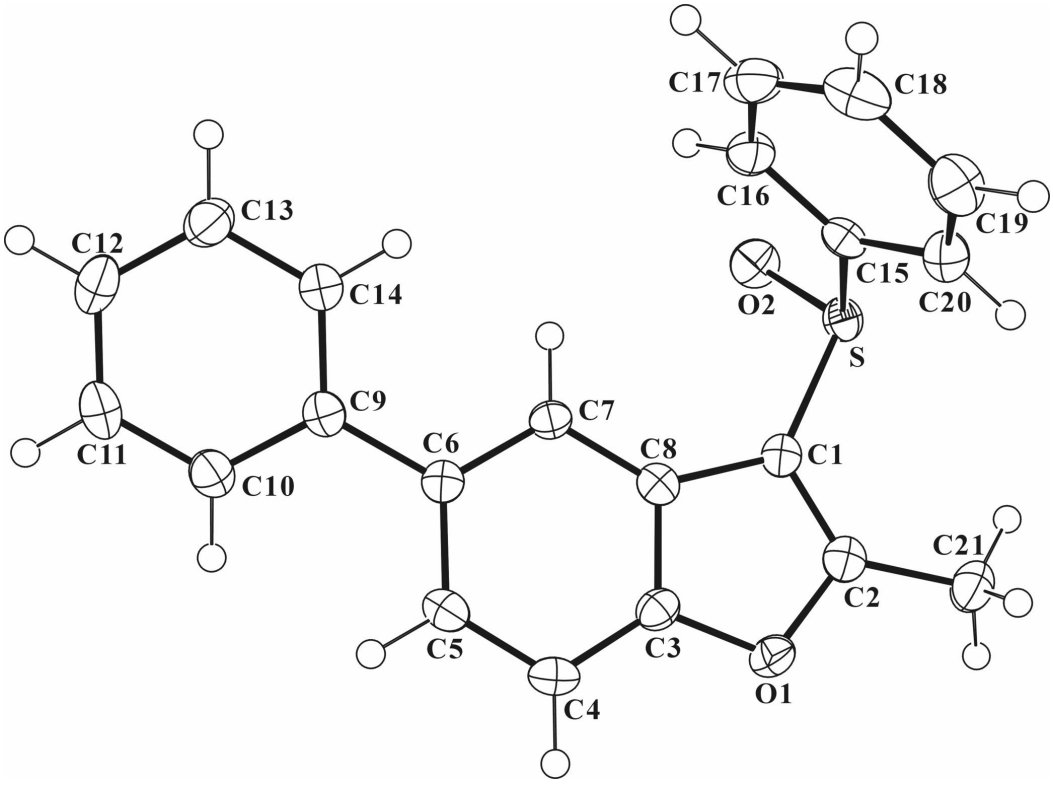
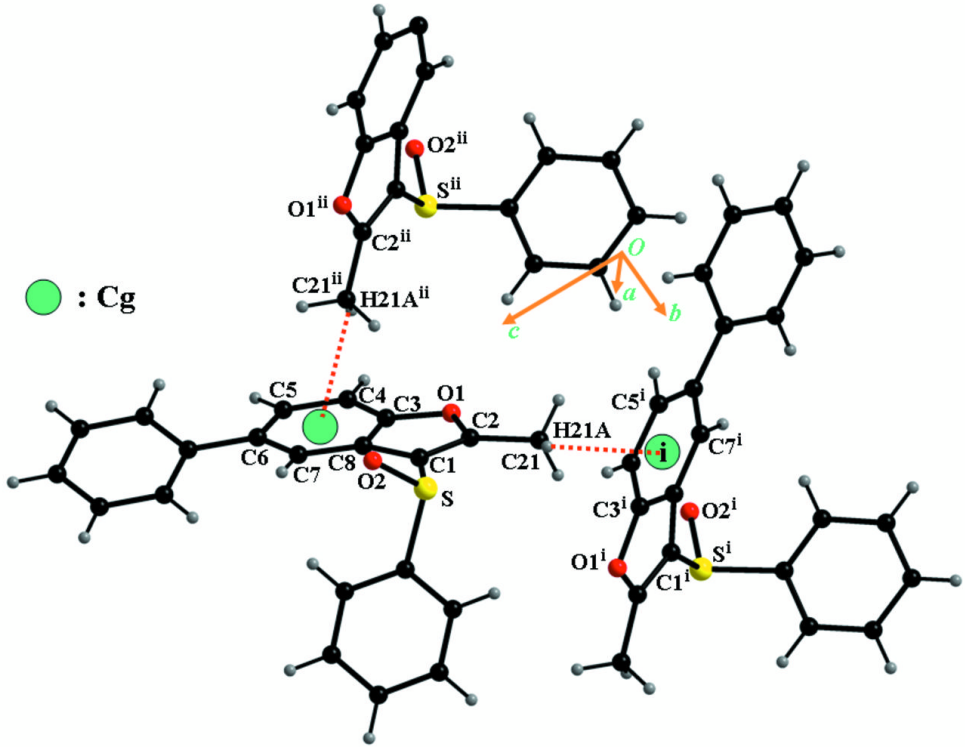

C17
C18
C16
C19
C13
C12
C15
C14
O2
C20
S
C9
C7
C11
C6
C8
C1
C10
C21
C2
C5
C3
O1
C4

Fig. 2


: Cg
O2ii
Sii
O1ii
C2ii
C21ii
H21Aii
O
a
b
c
C5
C4
C3
O1
C2
H21A
C6
C7
C8
O2
C1
C21
S
C5i
C7i
i
C3i
O2i
O1i
Si
C1i