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2-Methyl-1-(4-methylphenylsulfonyl)-naphtho[2,1-*b*]furan

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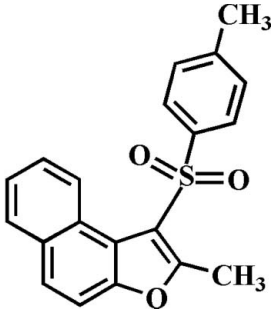
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.113; data-to-parameter ratio = 18.1.

In the title compound, $C_{20}H_{16}O_3S$, the 4-methylphenyl ring makes a dihedral angle of 83.07 (3)° with the mean plane [r.m.s. deviation = 0.020 (1) Å] of the naphthofuran fragment. In the crystal, molecules are linked by weak C—H···O and C—H···π interactions.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2008, 2012).



Experimental

Crystal data

$C_{20}H_{16}O_3S$
M_r = 336.39
Monoclinic, $P2_1/n$
a = 8.6628 (2) Å
b = 6.3669 (1) Å
c = 28.9119 (6) Å
β = 96.795 (1)°
V = 1583.44 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 173 K
0.35 × 0.32 × 0.28 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.676, T_{max} = 0.746
14714 measured reflections
3958 independent reflections
3363 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.113
S = 1.04
3958 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

Cg is the centroid of the C14–C19 4-methylphenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···O1^{i}	0.98	2.54	3.503 (2)	168
C13—H13C···O3^{ii}	0.98	2.49	3.440 (2)	164
C5—H5···Cg^{iii}	0.95	2.80	3.648 (2)	149

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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: HB6696).

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Bruker (2009). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
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supplementary materials

Acta Cryst. (2012). E**68**, o1193 [doi:10.1107/S1600536812012159]

2-Methyl-1-(4-methylphenylsulfonyl)naphtho[2,1-*b*]furan

Hong Dae Choi, Pil Ja Seo and Uk Lee

Comment

As a part of our ongoing study of 2-methylnaphtho[2,1-b]furan derivatives containing 1-phenylsulfonyl (Choi *et al.*, 2008) and 1-(4-methylphenylsulfinyl) (Choi *et al.*, 2012) substituents, we report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the naphthofuran unit is essentially planar, with a mean deviation of 0.020 (1) Å from the least-squares plane defined by the thirteen constituent atoms. The dihedral angle between the 4-methylphenyl ring and the mean plane of the naphthofuran fragment is 83.07 (3)°. The crystal packing features weak C—H···O hydrogen bonds (Fig. 2 & Table 1). In addition, weak C—H···π interactions occur (Fig. 2 & Table 1).

Experimental

77% 3-Chloroperoxybenzoic acid (448 mg, 2.0 mmol) was added in small portions to a stirred solution of 2-methyl-1-(4-methylphenylsulfanyl)naphtho [2,1-b]furan (273 mg, 0.9 mmol) in dichloromethane (40 mL) at 273 K. After being stirred at room temperature for 10h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated at reduced pressure. The residue was purified by column chromatography (benzene) to afford the title compound as a colorless solid [yield 68%, m.p. 440–441 K; R_f = 0.41 (benzene)]. Colourless blocks were prepared by slow evaporation of a solution of the title compound in acetone at room temperature.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for aryl and 0.98 Å for methyl H atoms. Uiso(H) = 1.2U_{eq}(C) for aryl and 1.5U_{eq}(C) for methyl H atoms. The positions of methyl hydrogens were optimized rotationally.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); 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* (Sheldrick, 2008).

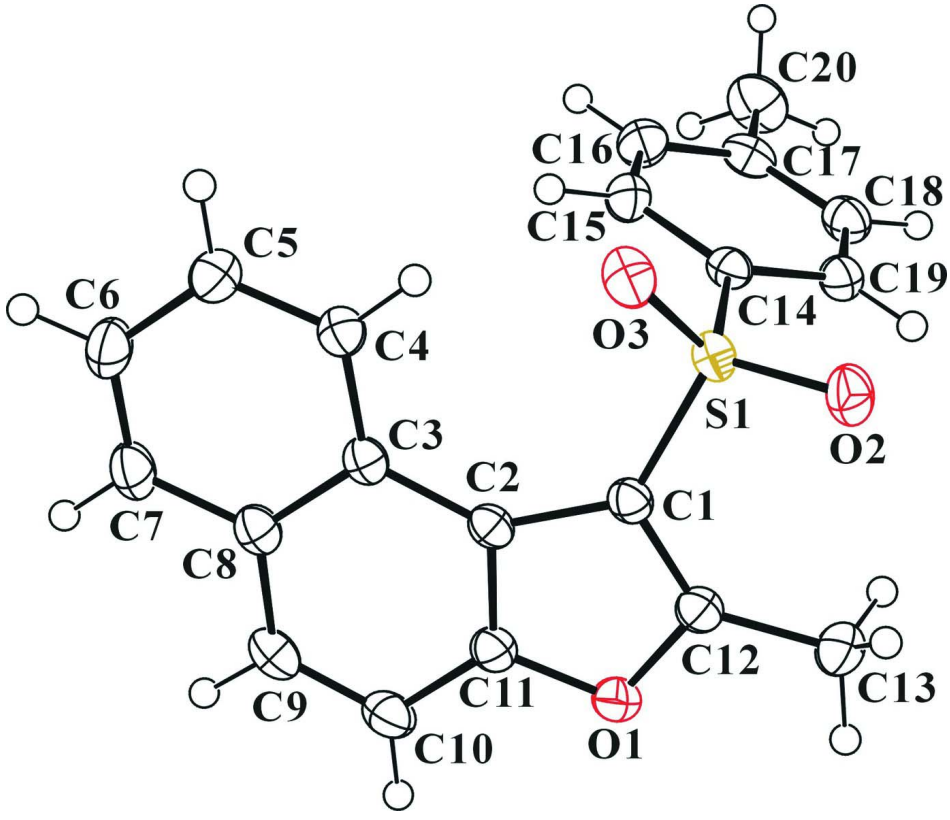

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

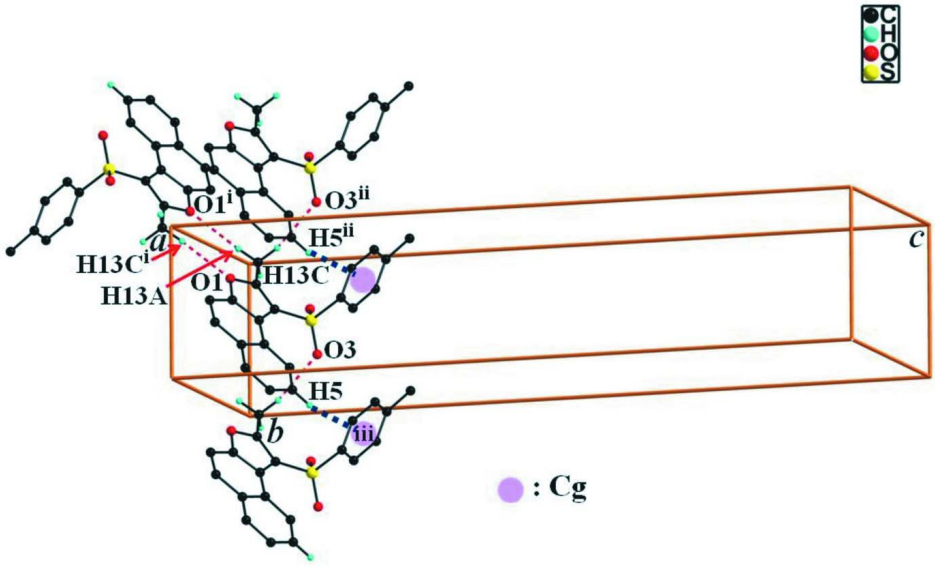

Figure 2

A view of the C—H···O and C—H···π interactions (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity. [Symmetry codes: (i) -x + 1, - y, - z; (ii) x, y - 1, z; (iii) x, y + 1, z.]

2-Methyl-1-(4-methylphenylsulfonyl)naphtho[2,1-*b*]furan

Crystal data

$C_{20}H_{16}O_3S$
$M_r = 336.39$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.6628$ (2) Å
$b = 6.3669$ (1) Å
$c = 28.9119$ (6) Å
$\beta = 96.795$ (1)°
$V = 1583.44$ (6) Å^3
$Z = 4$
$F(000) = 704$
$D_x = 1.411$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5369 reflections
$\theta = 2.4$–$28.2°$
$\mu = 0.22$ mm^{-1}
$T = 173$ K
Block, colourless
0.35 × 0.32 × 0.28 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: rotating anode
Graphite multilayer monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.676$, $T_{max} = 0.746$
14714 measured reflections
3958 independent reflections
3363 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.4°$
$h = -11 \rightarrow 11$
$k = -8 \rightarrow 8$
$l = -38 \rightarrow 36$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.113$
$S = 1.04$
3958 reflections
219 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0592P)^2 + 0.6896P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ 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}
S1	0.27965 (4)	0.47230 (6)	0.122492 (12)	0.02209 (11)
O1	0.53486 (13)	0.23737 (18)	0.03339 (4)	0.0289 (3)
O2	0.14170 (12)	0.35873 (19)	0.10517 (4)	0.0307 (3)
O3	0.26753 (12)	0.69453 (18)	0.12988 (4)	0.0298 (3)

C1	0.41907 (16)	0.4236 (2)	0.08492 (5)	0.0219 (3)
C2	0.56531 (16)	0.5295 (2)	0.07891 (5)	0.0213 (3)
C3	0.65037 (16)	0.7128 (2)	0.09593 (5)	0.0218 (3)
C4	0.60027 (17)	0.8614 (2)	0.12726 (5)	0.0247 (3)
H4	0.5033	0.8415	0.1389	0.030*
C5	0.68864 (19)	1.0335 (3)	0.14119 (6)	0.0295 (3)
H5	0.6519	1.1313	0.1622	0.035*
C6	0.83300 (19)	1.0672 (3)	0.12478 (6)	0.0332 (4)
H6	0.8936	1.1870	0.1345	0.040*
C7	0.88506 (18)	0.9260 (3)	0.09476 (6)	0.0316 (4)
H7	0.9831	0.9485	0.0840	0.038*
C8	0.79719 (16)	0.7470 (2)	0.07925 (5)	0.0254 (3)
C9	0.85486 (18)	0.6061 (3)	0.04712 (6)	0.0305 (3)
H9	0.9540	0.6316	0.0373	0.037*
C10	0.77213 (19)	0.4364 (3)	0.03018 (6)	0.0308 (3)
H10	0.8093	0.3442	0.0082	0.037*
C11	0.62874 (18)	0.4049 (2)	0.04693 (5)	0.0251 (3)
C12	0.40872 (18)	0.2496 (2)	0.05693 (5)	0.0260 (3)
C13	0.2966 (2)	0.0750 (3)	0.04686 (6)	0.0349 (4)
H13A	0.3285	−0.0119	0.0217	0.052*
H13B	0.1927	0.1325	0.0374	0.052*
H13C	0.2943	−0.0110	0.0749	0.052*
C14	0.35605 (16)	0.3550 (2)	0.17546 (5)	0.0218 (3)
C15	0.46746 (18)	0.4594 (3)	0.20557 (5)	0.0279 (3)
H15	0.4992	0.5975	0.1985	0.033*
C16	0.53175 (19)	0.3600 (3)	0.24596 (5)	0.0320 (4)
H16	0.6078	0.4310	0.2666	0.038*
C17	0.48649 (18)	0.1574 (3)	0.25668 (5)	0.0298 (3)
C18	0.37223 (19)	0.0579 (3)	0.22656 (6)	0.0291 (3)
H18	0.3380	−0.0784	0.2340	0.035*
C19	0.30745 (17)	0.1542 (2)	0.18583 (5)	0.0253 (3)
H19	0.2308	0.0838	0.1652	0.030*
C20	0.5594 (2)	0.0488 (3)	0.30016 (6)	0.0441 (5)
H20A	0.5242	0.1161	0.3275	0.066*
H20B	0.6729	0.0591	0.3020	0.066*
H20C	0.5287	−0.0994	0.2992	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01901 (17)	0.0225 (2)	0.02499 (19)	−0.00027 (13)	0.00374 (13)	0.00294 (13)
O1	0.0367 (6)	0.0263 (6)	0.0254 (5)	−0.0045 (5)	0.0102 (4)	−0.0036 (4)
O2	0.0212 (5)	0.0360 (7)	0.0345 (6)	−0.0048 (5)	0.0009 (4)	0.0022 (5)
O3	0.0270 (5)	0.0233 (6)	0.0403 (6)	0.0038 (4)	0.0093 (5)	0.0044 (5)
C1	0.0227 (6)	0.0237 (7)	0.0195 (6)	−0.0006 (5)	0.0028 (5)	0.0026 (5)
C2	0.0217 (6)	0.0232 (7)	0.0193 (6)	0.0012 (5)	0.0031 (5)	0.0034 (5)
C3	0.0210 (6)	0.0231 (7)	0.0210 (6)	0.0007 (5)	0.0014 (5)	0.0047 (5)
C4	0.0240 (7)	0.0236 (7)	0.0266 (7)	0.0005 (6)	0.0037 (5)	0.0011 (6)
C5	0.0308 (8)	0.0261 (8)	0.0313 (8)	−0.0006 (6)	0.0030 (6)	−0.0022 (6)
C6	0.0299 (8)	0.0312 (9)	0.0375 (9)	−0.0099 (7)	0.0004 (6)	−0.0011 (7)

C7	0.0241 (7)	0.0358 (9)	0.0352 (8)	−0.0076 (6)	0.0048 (6)	0.0033 (7)
C8	0.0220 (6)	0.0291 (8)	0.0249 (7)	−0.0004 (6)	0.0026 (5)	0.0041 (6)
C9	0.0257 (7)	0.0366 (9)	0.0309 (8)	0.0011 (7)	0.0108 (6)	0.0043 (7)
C10	0.0331 (8)	0.0327 (9)	0.0286 (8)	0.0022 (7)	0.0124 (6)	0.0000 (6)
C11	0.0301 (7)	0.0234 (7)	0.0225 (7)	−0.0016 (6)	0.0056 (6)	0.0014 (6)
C12	0.0316 (7)	0.0256 (8)	0.0210 (7)	−0.0037 (6)	0.0042 (5)	0.0015 (6)
C13	0.0430 (9)	0.0297 (9)	0.0326 (8)	−0.0117 (7)	0.0066 (7)	−0.0041 (7)
C14	0.0233 (6)	0.0215 (7)	0.0216 (6)	0.0020 (5)	0.0061 (5)	0.0003 (5)
C15	0.0312 (7)	0.0259 (8)	0.0268 (7)	−0.0050 (6)	0.0048 (6)	−0.0002 (6)
C16	0.0334 (8)	0.0370 (9)	0.0250 (7)	−0.0034 (7)	0.0007 (6)	−0.0018 (7)
C17	0.0316 (8)	0.0362 (9)	0.0227 (7)	0.0050 (7)	0.0080 (6)	0.0045 (6)
C18	0.0339 (8)	0.0253 (8)	0.0297 (8)	0.0003 (6)	0.0097 (6)	0.0033 (6)
C19	0.0274 (7)	0.0231 (7)	0.0262 (7)	−0.0024 (6)	0.0069 (6)	0.0000 (6)
C20	0.0469 (10)	0.0543 (12)	0.0306 (9)	0.0023 (9)	0.0021 (8)	0.0138 (8)

Geometric parameters (Å, º)

S1—O2	1.4354 (11)	C9—H9	0.9500
S1—O3	1.4366 (12)	C10—C11	1.400 (2)
S1—C1	1.7450 (15)	C10—H10	0.9500
S1—C14	1.7611 (15)	C12—C13	1.483 (2)
O1—C12	1.3562 (18)	C13—H13A	0.9800
O1—C11	1.3700 (18)	C13—H13B	0.9800
C1—C12	1.369 (2)	C13—H13C	0.9800
C1—C2	1.4632 (19)	C14—C19	1.390 (2)
C2—C11	1.381 (2)	C14—C15	1.390 (2)
C2—C3	1.436 (2)	C15—C16	1.386 (2)
C3—C4	1.413 (2)	C15—H15	0.9500
C3—C8	1.4293 (19)	C16—C17	1.394 (2)
C4—C5	1.370 (2)	C16—H16	0.9500
C4—H4	0.9500	C17—C18	1.391 (2)
C5—C6	1.405 (2)	C17—C20	1.506 (2)
C5—H5	0.9500	C18—C19	1.386 (2)
C6—C7	1.363 (2)	C18—H18	0.9500
C6—H6	0.9500	C19—H19	0.9500
C7—C8	1.414 (2)	C20—H20A	0.9800
C7—H7	0.9500	C20—H20B	0.9800
C8—C9	1.424 (2)	C20—H20C	0.9800
C9—C10	1.355 (2)		

O2—S1—O3	118.39 (7)	O1—C11—C2	111.64 (13)
O2—S1—C1	107.60 (7)	O1—C11—C10	122.26 (14)
O3—S1—C1	109.60 (7)	C2—C11—C10	126.08 (15)
O2—S1—C14	107.55 (7)	O1—C12—C1	110.07 (13)
O3—S1—C14	108.36 (7)	O1—C12—C13	113.98 (13)
C1—S1—C14	104.45 (7)	C1—C12—C13	135.95 (15)
C12—O1—C11	107.34 (11)	C12—C13—H13A	109.5
C12—C1—C2	107.44 (13)	C12—C13—H13B	109.5
C12—C1—S1	120.67 (11)	H13A—C13—H13B	109.5
C2—C1—S1	131.67 (11)	C12—C13—H13C	109.5

C11—C2—C3	117.89 (13)	H13A—C13—H13C	109.5
C11—C2—C1	103.51 (13)	H13B—C13—H13C	109.5
C3—C2—C1	138.60 (13)	C19—C14—C15	120.70 (14)
C4—C3—C8	117.86 (13)	C19—C14—S1	119.04 (11)
C4—C3—C2	125.43 (13)	C15—C14—S1	120.22 (12)
C8—C3—C2	116.70 (13)	C16—C15—C14	119.28 (15)
C5—C4—C3	121.38 (14)	C16—C15—H15	120.4
C5—C4—H4	119.3	C14—C15—H15	120.4
C3—C4—H4	119.3	C15—C16—C17	120.97 (15)
C4—C5—C6	120.75 (15)	C15—C16—H16	119.5
C4—C5—H5	119.6	C17—C16—H16	119.5
C6—C5—H5	119.6	C18—C17—C16	118.70 (14)
C7—C6—C5	119.30 (15)	C18—C17—C20	120.71 (16)
C7—C6—H6	120.4	C16—C17—C20	120.59 (16)
C5—C6—H6	120.4	C19—C18—C17	121.13 (15)
C6—C7—C8	121.82 (15)	C19—C18—H18	119.4
C6—C7—H7	119.1	C17—C18—H18	119.4
C8—C7—H7	119.1	C18—C19—C14	119.19 (14)
C7—C8—C9	119.84 (14)	C18—C19—H19	120.4
C7—C8—C3	118.89 (14)	C14—C19—H19	120.4
C9—C8—C3	121.26 (14)	C17—C20—H20A	109.5
C10—C9—C8	121.78 (14)	C17—C20—H20B	109.5
C10—C9—H9	119.1	H20A—C20—H20B	109.5
C8—C9—H9	119.1	C17—C20—H20C	109.5
C9—C10—C11	116.24 (15)	H20A—C20—H20C	109.5
C9—C10—H10	121.9	H20B—C20—H20C	109.5
C11—C10—H10	121.9		

O2—S1—C1—C12	−21.68 (14)	C12—O1—C11—C10	178.08 (15)
O3—S1—C1—C12	−151.66 (12)	C3—C2—C11—O1	−179.34 (12)
C14—S1—C1—C12	92.43 (13)	C1—C2—C11—O1	0.22 (16)
O2—S1—C1—C2	164.56 (13)	C3—C2—C11—C10	2.0 (2)
O3—S1—C1—C2	34.58 (16)	C1—C2—C11—C10	−178.40 (15)
C14—S1—C1—C2	−81.34 (15)	C9—C10—C11—O1	−178.71 (15)
C12—C1—C2—C11	0.24 (16)	C9—C10—C11—C2	−0.2 (2)
S1—C1—C2—C11	174.62 (12)	C11—O1—C12—C1	0.76 (16)
C12—C1—C2—C3	179.65 (17)	C11—O1—C12—C13	−178.63 (13)
S1—C1—C2—C3	−6.0 (3)	C2—C1—C12—O1	−0.62 (17)
C11—C2—C3—C4	177.50 (14)	S1—C1—C12—O1	−175.75 (10)
C1—C2—C3—C4	−1.8 (3)	C2—C1—C12—C13	178.57 (17)
C11—C2—C3—C8	−2.0 (2)	S1—C1—C12—C13	3.4 (3)
C1—C2—C3—C8	178.65 (16)	O2—S1—C14—C19	17.39 (14)
C8—C3—C4—C5	0.3 (2)	O3—S1—C14—C19	146.47 (11)
C2—C3—C4—C5	−179.20 (14)	C1—S1—C14—C19	−96.75 (12)
C3—C4—C5—C6	−0.3 (2)	O2—S1—C14—C15	−164.89 (12)
C4—C5—C6—C7	−0.1 (3)	O3—S1—C14—C15	−35.81 (14)
C5—C6—C7—C8	0.6 (3)	C1—S1—C14—C15	80.97 (13)
C6—C7—C8—C9	178.71 (15)	C19—C14—C15—C16	0.9 (2)
C6—C7—C8—C3	−0.6 (2)	S1—C14—C15—C16	−176.78 (12)

C4—C3—C8—C7	0.1 (2)	C14—C15—C16—C17	0.1 (2)
C2—C3—C8—C7	179.66 (13)	C15—C16—C17—C18	−1.6 (2)
C4—C3—C8—C9	−179.15 (14)	C15—C16—C17—C20	178.72 (16)
C2—C3—C8—C9	0.4 (2)	C16—C17—C18—C19	2.1 (2)
C7—C8—C9—C10	−177.80 (16)	C20—C17—C18—C19	−178.24 (15)
C3—C8—C9—C10	1.5 (2)	C17—C18—C19—C14	−1.1 (2)
C8—C9—C10—C11	−1.5 (2)	C15—C14—C19—C18	−0.4 (2)
C12—O1—C11—C2	−0.60 (17)	S1—C14—C19—C18	177.28 (11)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C14–C19 4-methylphenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13*A*···O1^{i}	0.98	2.54	3.503 (2)	168
C13—H13*C*···O3^{ii}	0.98	2.49	3.440 (2)	164
C5—H5···*Cg*^{iii}	0.95	2.80	3.648 (2)	149

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