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Ethyl 2-bromomethyl-1-phenylsulfonyl-1*H*-indole-3-carboxylate

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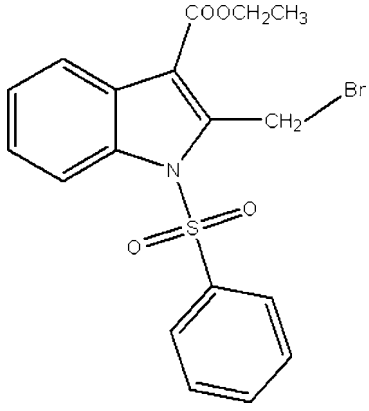
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.040; wR factor = 0.115; data-to-parameter ratio = 23.4.

In the title compound, $C_{18}H_{16}BrNO_4S$, the phenyl ring forms a dihedral angle of 83.87 (2)° with the indole ring system. The molecular structure is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by weak intermolecular C—H···O interactions.

Related literature

For related literature, see: Kusunoki *et al.* (2006); Nieto *et al.* (2005); Liu *et al.* (2007); Palani *et al.* (2006). A similar compound with a methoxycarbonyl substituent has been reported (Senthil Kumar *et al.*, 2006).



Experimental

Crystal data

$C_{18}H_{16}BrNO_4S$
M_r = 422.29
Monoclinic, $P2_1/c$
a = 8.1684 (3) Å
b = 9.4962 (4) Å
c = 23.0376 (9) Å
β = 94.147 (1)°
V = 1782.32 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.45 mm^{-1}
T = 295 (2) K
0.26 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEX II diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.527, T_{max} = 0.640
23119 measured reflections
5283 independent reflections
3029 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.115
S = 1.00
5283 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O2^{i}	0.93	2.58	3.396 (3)	147
C5—H5···O1^{ii}	0.93	2.50	3.419 (3)	171
C2—H2···O1	0.93	2.55	2.907 (3)	103
C8—H8···O2	0.93	2.27	2.852 (4)	121
C11—H11···O3	0.93	2.48	3.000 (4)	116
C15—H15*A*···O4	0.97	2.28	2.845 (3)	116
C15—H15*B*···O1	0.97	2.21	2.904 (3)	128

Symmetry codes: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x-1, y, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2442).

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supplementary materials

Acta Cryst. (2007). E**63**, o3564 [**doi:10.1107/S1600536807034800**]

Ethyl 2-bromomethyl-1-phenylsulfonyl-1*H*-indole-3-carboxylate

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Comment

The benzenesulfonamide derivatives possess significant biological activities, such as proliferation of colon adenocarcinoma cells (Kusunoki *et al.*, 2006) and antibacterial (Nieto *et al.*, 2005).

The geometric parameters in the title compound agree with the reported values of similar structures (Liu *et al.*, 2007; Palani *et al.*, 2006). The 5-membered and 6-membered rings of the indole moiety are co-planar [dihedral angle = 0.46 (2)°]. The sum of the bond angles around N1 (359.5°) indicates sp^2 hybridization.

The phenyl ring forms a dihedral angle 83.87 (2)° with the indole ring system. The torsion angles C7 - N1 - S1 - O2 and C14 - N1 - S1 - O1 [27.2 (2)° and −36.3 (2)°, respectively] indicate the *syn* conformation of the sulfonyl moiety.

The details of the hydrogen bonding are given in Table 1. The molecular structure is stabilized by weak intramolecular C - H···O interactions and the crystal packing of (I), (Fig. 2) is stabilized by weak intermolecular C - H···O interactions.

A similiar compound with methoxycarbonyl has been reported (Senthil Kumar *et al.*, 2006).

Experimental

Ethyl-1-benzenesulfonyl-2-bromomethyl indole-3-carboxylate was prepared *via* the allylic bromination of ethyl-1-benzenesulfonyl-2-methyl indole-3-carboxylate (3.93 mmol) using *N*-bromo succiniamide (3.93 mmol) in a catalytic amount of benzoyl peroxide in CCl_4 (20 ml) at reflux. The obtained compound was dissolved in hexane and ethyl acetate (9:1). Crystals were grown by slow evaporation of an ethyl acetate solution.

Refinement

H atoms were positioned geometrically and refined using riding model with C—H = 0.93 Å, U_{iso}(H) = 1.2Ueq(C) for aromatic C—H, C—H = 0.97 Å and U_{iso}(H) = 1.2Ueq(C) for CH_2 and C—H = 0.96 Å and U_{iso}(H) = 1.5Ueq(C) for CH_3.

Figures

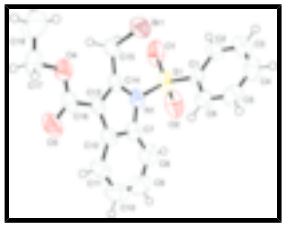

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

Fig. 2. The packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Ethyl 2-bromomethyl-1-phenylsulfonyl-1*H*-indole-3-carboxylate

Crystal data

$C_{18}H_{16}BrNO_4S$	F_{000} = 856
M_r = 422.29	D_x = 1.574 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6136 reflections
a = 8.1684 (3) Å	θ = 2.3–24.9º
b = 9.4962 (4) Å	μ = 2.45 mm^{-1}
c = 23.0376 (9) Å	T = 295 (2) K
β = 94.1470 (10)º	Needle, colourless
V = 1782.32 (12) Å^3	0.26 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker Kappa APEX II diffractometer	5283 independent reflections
Radiation source: fine-focus sealed tube	3029 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
T = 295(2) K	θ_{max} = 30.2º
ω and φ scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −11→11
T_{min} = 0.527, T_{max} = 0.640	k = −13→13
23119 measured reflections	l = −32→32

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H-atom parameters constrained
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.431P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.002
5283 reflections	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
226 parameters	$\Delta\rho_{min}$ = −0.51 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2387 (3)	0.2446 (3)	0.68219 (9)	0.0461 (5)
C2	0.2765 (3)	0.3652 (3)	0.71316 (11)	0.0589 (6)
H2	0.3853	0.3907	0.7222	0.071*
C3	0.1510 (4)	0.4474 (3)	0.73057 (13)	0.0747 (8)
H3	0.1748	0.5289	0.7519	0.090*
C4	−0.0089 (4)	0.4103 (3)	0.71671 (13)	0.0739 (8)
H4	−0.0932	0.4660	0.7292	0.089*
C5	−0.0456 (3)	0.2918 (3)	0.68459 (13)	0.0699 (8)
H5	−0.1546	0.2690	0.6743	0.084*
C6	0.0785 (3)	0.2060 (3)	0.66739 (12)	0.0590 (6)
H6	0.0545	0.1241	0.6463	0.071*
C7	0.2583 (3)	−0.0606 (3)	0.59266 (11)	0.0548 (6)
C8	0.2107 (4)	−0.1621 (3)	0.63136 (14)	0.0756 (8)
H8	0.2394	−0.1554	0.6711	0.091*
C9	0.1188 (4)	−0.2737 (4)	0.60822 (19)	0.0861 (10)
H9	0.0865	−0.3439	0.6331	0.103*
C10	0.0738 (4)	−0.2844 (3)	0.55022 (18)	0.0802 (9)
H10	0.0090	−0.3598	0.5367	0.096*
C11	0.1223 (3)	−0.1862 (3)	0.51146 (14)	0.0658 (7)
H11	0.0935	−0.1955	0.4718	0.079*
C12	0.2166 (3)	−0.0711 (2)	0.53288 (11)	0.0518 (6)
C13	0.2853 (3)	0.0485 (2)	0.50541 (10)	0.0472 (5)
C14	0.3655 (3)	0.1301 (3)	0.54708 (10)	0.0467 (5)
C15	0.4574 (3)	0.2627 (3)	0.53971 (12)	0.0589 (6)
H15A	0.5022	0.2631	0.5019	0.071*
H15B	0.5481	0.2683	0.5692	0.071*
C16	0.2679 (3)	0.0703 (3)	0.44194 (11)	0.0565 (6)
C17	0.3131 (4)	0.2231 (4)	0.36344 (11)	0.0755 (9)
H17A	0.2003	0.2113	0.3479	0.091*
H17B	0.3820	0.1598	0.3429	0.091*
C18	0.3658 (5)	0.3693 (5)	0.35624 (15)	0.1081 (13)
H18A	0.3578	0.3931	0.3156	0.162*
H18B	0.4774	0.3796	0.3717	0.162*
H18C	0.2966	0.4309	0.3767	0.162*
N1	0.3515 (2)	0.0648 (2)	0.60141 (8)	0.0529 (5)
O1	0.5447 (2)	0.2186 (3)	0.66281 (9)	0.0900 (8)
O2	0.4074 (3)	0.0190 (3)	0.70652 (8)	0.0897 (7)
O3	0.2063 (3)	−0.0149 (2)	0.40830 (9)	0.0834 (6)
O4	0.3275 (2)	0.1922 (2)	0.42551 (7)	0.0658 (5)
S1	0.40267 (8)	0.13478 (9)	0.66717 (3)	0.0608 (2)
Br1	0.31355 (4)	0.42658 (3)	0.546316 (14)	0.07439 (13)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0396 (11)	0.0612 (14)	0.0374 (11)	0.0051 (10)	0.0012 (9)	−0.0013 (10)
C2	0.0543 (14)	0.0657 (15)	0.0552 (14)	−0.0019 (13)	−0.0056 (12)	−0.0089 (13)
C3	0.094 (2)	0.0675 (18)	0.0618 (17)	0.0152 (16)	−0.0029 (16)	−0.0147 (14)
C4	0.073 (2)	0.081 (2)	0.0690 (18)	0.0306 (16)	0.0164 (15)	0.0056 (16)
C5	0.0412 (13)	0.084 (2)	0.085 (2)	0.0091 (13)	0.0078 (13)	0.0100 (17)
C6	0.0432 (13)	0.0621 (15)	0.0710 (16)	−0.0007 (11)	−0.0006 (12)	−0.0062 (13)
C7	0.0471 (13)	0.0575 (14)	0.0597 (15)	0.0160 (11)	0.0036 (11)	−0.0021 (12)
C8	0.0763 (19)	0.076 (2)	0.0755 (19)	0.0153 (16)	0.0082 (15)	0.0103 (16)
C9	0.077 (2)	0.0643 (19)	0.119 (3)	0.0090 (16)	0.020 (2)	0.023 (2)
C10	0.0606 (17)	0.0526 (16)	0.127 (3)	0.0066 (13)	0.0039 (18)	−0.0018 (18)
C11	0.0529 (15)	0.0556 (15)	0.087 (2)	0.0102 (12)	−0.0040 (14)	−0.0149 (14)
C12	0.0408 (12)	0.0511 (13)	0.0630 (15)	0.0150 (10)	0.0009 (11)	−0.0110 (11)
C13	0.0395 (11)	0.0520 (13)	0.0498 (13)	0.0120 (9)	0.0006 (10)	−0.0119 (10)
C14	0.0369 (11)	0.0558 (13)	0.0474 (13)	0.0120 (10)	0.0032 (9)	−0.0105 (10)
C15	0.0478 (13)	0.0648 (15)	0.0641 (15)	0.0025 (12)	0.0034 (11)	−0.0143 (13)
C16	0.0504 (14)	0.0670 (16)	0.0521 (14)	0.0143 (12)	0.0035 (11)	−0.0147 (13)
C17	0.0757 (19)	0.106 (2)	0.0452 (15)	0.0131 (17)	0.0034 (13)	−0.0012 (15)
C18	0.134 (4)	0.125 (3)	0.064 (2)	−0.018 (3)	−0.002 (2)	0.023 (2)
N1	0.0481 (11)	0.0638 (12)	0.0461 (11)	0.0088 (9)	−0.0015 (9)	−0.0099 (9)
O1	0.0357 (9)	0.160 (2)	0.0733 (12)	−0.0026 (12)	−0.0043 (8)	−0.0447 (14)
O2	0.1038 (16)	0.1094 (17)	0.0528 (11)	0.0539 (14)	−0.0144 (11)	−0.0001 (11)
O3	0.1037 (16)	0.0862 (14)	0.0584 (12)	−0.0081 (13)	−0.0063 (11)	−0.0274 (11)
O4	0.0751 (12)	0.0783 (13)	0.0436 (9)	−0.0006 (10)	0.0008 (8)	−0.0060 (9)
S1	0.0430 (3)	0.0916 (5)	0.0460 (3)	0.0190 (3)	−0.0092 (3)	−0.0135 (3)
Br1	0.0816 (2)	0.05670 (18)	0.0855 (2)	0.00656 (14)	0.01067 (16)	−0.00998 (14)

Geometric parameters (Å, °)

C1—C2	1.373 (3)	C11—H11	0.9300
C1—C6	1.378 (3)	C12—C13	1.434 (3)
C1—S1	1.752 (2)	C13—C14	1.364 (3)
C2—C3	1.371 (4)	C13—C16	1.473 (3)
C2—H2	0.9300	C14—N1	1.409 (3)
C3—C4	1.368 (5)	C14—C15	1.482 (4)
C3—H3	0.9300	C15—Br1	1.962 (2)
C4—C5	1.368 (4)	C15—H15A	0.9700
C4—H4	0.9300	C15—H15B	0.9700
C5—C6	1.381 (4)	C16—O3	1.205 (3)
C5—H5	0.9300	C16—O4	1.322 (3)
C6—H6	0.9300	C17—O4	1.456 (3)
C7—C8	1.388 (4)	C17—C18	1.467 (5)
C7—C12	1.398 (4)	C17—H17A	0.9700
C7—N1	1.420 (3)	C17—H17B	0.9700
C8—C9	1.383 (5)	C18—H18A	0.9600
C8—H8	0.9300	C18—H18B	0.9600

C9—C10	1.364 (5)	C18—H18C	0.9600
C9—H9	0.9300	N1—S1	1.679 (2)
C10—C11	1.370 (4)	O1—S1	1.416 (2)
C10—H10	0.9300	O2—S1	1.424 (3)
C11—C12	1.406 (4)		
C2—C1—C6	121.6 (2)	C14—C13—C16	128.8 (2)
C2—C1—S1	116.92 (18)	C12—C13—C16	122.4 (2)
C6—C1—S1	121.26 (19)	C13—C14—N1	108.1 (2)
C3—C2—C1	118.8 (2)	C13—C14—C15	128.6 (2)
C3—C2—H2	120.6	N1—C14—C15	123.4 (2)
C1—C2—H2	120.6	C14—C15—Br1	110.73 (16)
C4—C3—C2	120.4 (3)	C14—C15—H15A	109.5
C4—C3—H3	119.8	Br1—C15—H15A	109.5
C2—C3—H3	119.8	C14—C15—H15B	109.5
C3—C4—C5	120.4 (3)	Br1—C15—H15B	109.5
C3—C4—H4	119.8	H15A—C15—H15B	108.1
C5—C4—H4	119.8	O3—C16—O4	123.3 (3)
C4—C5—C6	120.3 (3)	O3—C16—C13	123.4 (3)
C4—C5—H5	119.9	O4—C16—C13	113.3 (2)
C6—C5—H5	119.9	O4—C17—C18	107.4 (3)
C1—C6—C5	118.4 (3)	O4—C17—H17A	110.2
C1—C6—H6	120.8	C18—C17—H17A	110.2
C5—C6—H6	120.8	O4—C17—H17B	110.2
C8—C7—C12	121.6 (3)	C18—C17—H17B	110.2
C8—C7—N1	131.6 (3)	H17A—C17—H17B	108.5
C12—C7—N1	106.8 (2)	C17—C18—H18A	109.5
C9—C8—C7	117.0 (3)	C17—C18—H18B	109.5
C9—C8—H8	121.5	H18A—C18—H18B	109.5
C7—C8—H8	121.5	C17—C18—H18C	109.5
C10—C9—C8	122.4 (3)	H18A—C18—H18C	109.5
C10—C9—H9	118.8	H18B—C18—H18C	109.5
C8—C9—H9	118.8	C14—N1—C7	108.65 (19)
C9—C10—C11	121.2 (3)	C14—N1—S1	126.64 (17)
C9—C10—H10	119.4	C7—N1—S1	123.76 (18)
C11—C10—H10	119.4	C16—O4—C17	116.9 (2)
C10—C11—C12	118.6 (3)	O1—S1—O2	119.62 (14)
C10—C11—H11	120.7	O1—S1—N1	108.05 (12)
C12—C11—H11	120.7	O2—S1—N1	105.23 (13)
C7—C12—C11	119.3 (3)	O1—S1—C1	108.76 (13)
C7—C12—C13	107.6 (2)	O2—S1—C1	108.65 (12)
C11—C12—C13	133.1 (2)	N1—S1—C1	105.66 (10)
C14—C13—C12	108.8 (2)		
C6—C1—C2—C3	1.2 (4)	N1—C14—C15—Br1	−89.9 (2)
S1—C1—C2—C3	−174.0 (2)	C14—C13—C16—O3	173.3 (2)
C1—C2—C3—C4	−0.7 (4)	C12—C13—C16—O3	−5.8 (4)
C2—C3—C4—C5	−0.9 (5)	C14—C13—C16—O4	−6.3 (3)
C3—C4—C5—C6	2.0 (5)	C12—C13—C16—O4	174.6 (2)
C2—C1—C6—C5	−0.1 (4)	C13—C14—N1—C7	−0.5 (2)

S1—C1—C6—C5	174.9 (2)	C15—C14—N1—C7	−179.0 (2)
C4—C5—C6—C1	−1.5 (4)	C13—C14—N1—S1	−169.61 (16)
C12—C7—C8—C9	−0.6 (4)	C15—C14—N1—S1	11.9 (3)
N1—C7—C8—C9	−179.9 (3)	C8—C7—N1—C14	179.6 (3)
C7—C8—C9—C10	−0.8 (5)	C12—C7—N1—C14	0.2 (2)
C8—C9—C10—C11	2.0 (5)	C8—C7—N1—S1	−10.9 (4)
C9—C10—C11—C12	−1.7 (4)	C12—C7—N1—S1	169.74 (16)
C8—C7—C12—C11	0.9 (3)	O3—C16—O4—C17	1.3 (4)
N1—C7—C12—C11	−179.7 (2)	C13—C16—O4—C17	−179.1 (2)
C8—C7—C12—C13	−179.4 (2)	C18—C17—O4—C16	172.4 (3)
N1—C7—C12—C13	0.1 (2)	C14—N1—S1—O1	−36.3 (2)
C10—C11—C12—C7	0.3 (3)	C7—N1—S1—O1	156.06 (19)
C10—C11—C12—C13	−179.5 (2)	C14—N1—S1—O2	−165.21 (19)
C7—C12—C13—C14	−0.4 (2)	C7—N1—S1—O2	27.2 (2)
C11—C12—C13—C14	179.3 (2)	C14—N1—S1—C1	79.9 (2)
C7—C12—C13—C16	178.8 (2)	C7—N1—S1—C1	−87.67 (19)
C11—C12—C13—C16	−1.4 (4)	C2—C1—S1—O1	−29.9 (2)
C12—C13—C14—N1	0.5 (2)	C6—C1—S1—O1	154.9 (2)
C16—C13—C14—N1	−178.7 (2)	C2—C1—S1—O2	101.9 (2)
C12—C13—C14—C15	179.0 (2)	C6—C1—S1—O2	−73.4 (2)
C16—C13—C14—C15	−0.2 (4)	C2—C1—S1—N1	−145.64 (19)
C13—C14—C15—Br1	91.9 (3)	C6—C1—S1—N1	39.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O2^{i}	0.93	2.58	3.396 (3)	147
C5—H5···O1^{ii}	0.93	2.50	3.419 (3)	171
C2—H2···O1	0.93	2.55	2.907 (3)	103
C8—H8···O2	0.93	2.27	2.852 (4)	121
C11—H11···O3	0.93	2.48	3.000 (4)	116
C15—H15A···O4	0.97	2.28	2.845 (3)	116
C15—H15B···O1	0.97	2.21	2.904 (3)	128

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+3/2; (ii) *x*−1, *y*, *z*.

Fig. 1

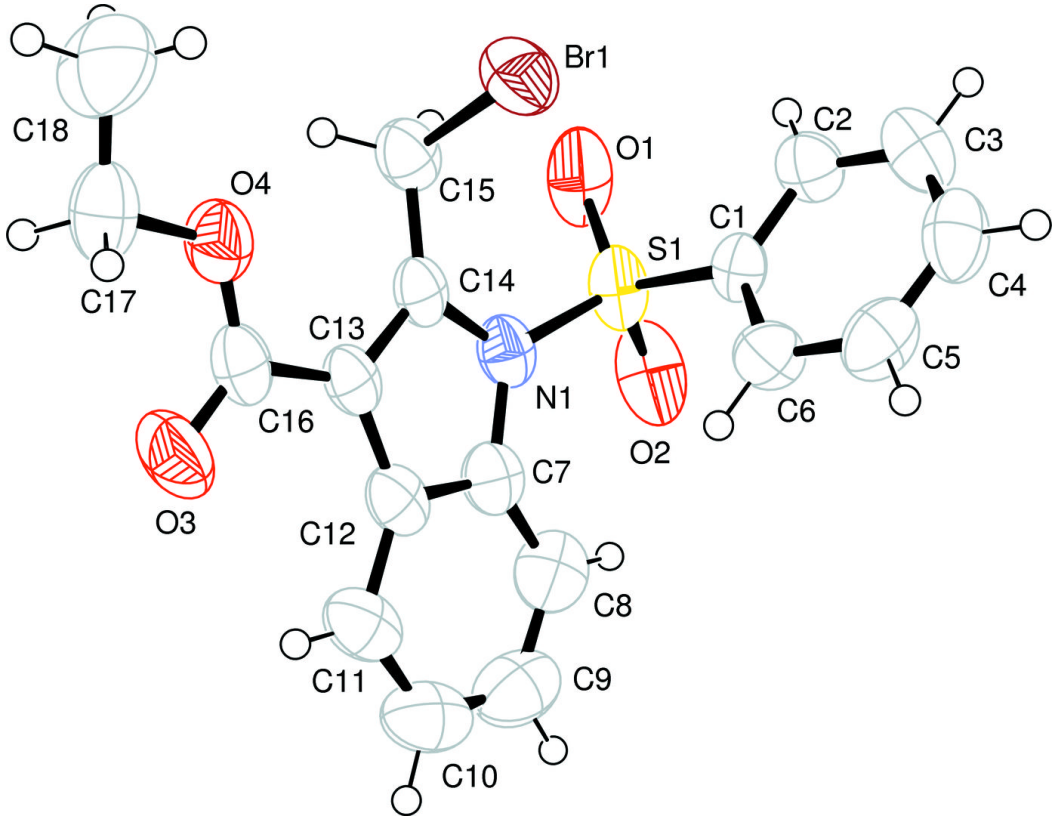
Br1
C18
O4
C15
O1
C2
C3
C1
S1
C14
C4
C17
C13
N1
C5
C16
C6
O2
O3
C7
C12
C8
C11
C9
C10

Fig. 2