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3-Chloro-1-phenylsulfonyl-1*H*-indole-2-carbaldehyde

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In the title molecule, $C_{15}H_{10}ClNO_3S$, the phenyl ring and the mean plane of the indole system make a dihedral angle of 78.3 (1)°. The crystal packing is mainly stabilized by weak intermolecular C—H···O interactions.

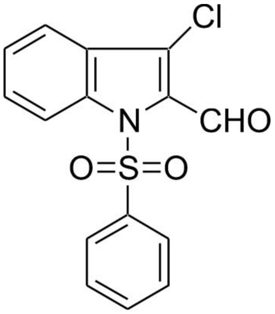
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.055
wR factor = 0.144
Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Indoles form an integral part of many natural products. They possess a number of active sites, allowing the generation of molecular diversity in a variety of chemical reactions (Farhanullah *et al.*, 2004). In continuation of our structural study of indole derivatives, we present here the crystal structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths and angles in (I) (Table 1) show normal values (Govindasamy *et al.*, 1998). The angles O1—S1—O2 and N1—S1—C10 confirm the distorted tetrahedral geometry around atom S1, which may be attributed to the Thorpe–Ingold effect (Bassindale, 1984). The phenyl ring and the mean plane of the indole system make a dihedral angle of 78.3 (1)°.

In the crystal structure, the relatively short distance between the centroids Cg of neighbouring pyrrole rings, $Cg\cdots Cg^{i}$ = 3.549 (3) Å [symmetry code: (i) $-x$, $1-y$, $-z$], indicates a possible π–π stacking interaction, which contributes to the packing stabilization (Fig. 2) along with intermolecular C—H···O hydrogen bonds (Table 2).

Experimental

To a solution of 1-phenylsulfonyl-2-bromomethyl-3-chloroindole (5 mmol) in dry chloroform (60 ml), bis(tetra-*n*-butylammonium) dichromate (5.0 mmol) was added. The resulting solution was refluxed for 8 h and the reaction mixture was then filtered through Celite (10 g) to eliminate inorganic components and unreacted tetrabutylammonium salts. The Celite was washed with diethyl ether (75 ml). Evaporation of the combined organic solvents followed by chromatographic purification using hexane and ethyl acetate (7:3)

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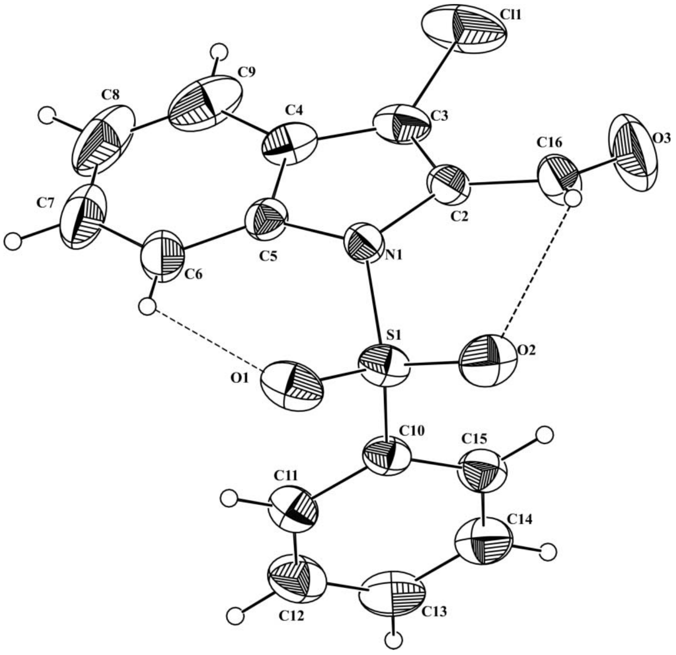

Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

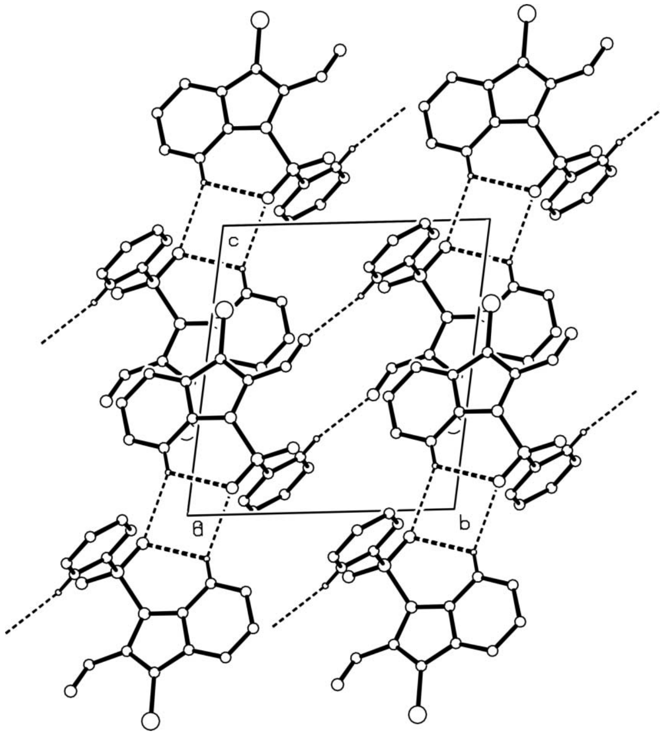

Figure 2
The molecular packing of (I), viewed down the *a* axis. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

afforded the indole-2-aldehyde as a pure crystalline solid in 84% yield (m.p. 427 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 1677, 1357, 1176; ^{1}H NMR (400 MHz, CDCl$_3$, δ, p.p.m.): 7.26–7.79 (*m*, 8H, Ar—H), 8.25–8.27 (*d*, 1H, J = 8.0 Hz, indole-7H), 10.49 (*s*, 1H, CHO); MS (*m*/*z*, %): 321 (*M*+2, 7.3), 319 (*M*+, 14.7), 178 (20.1), 150 (17.8), 140 (29.7), 122 (17.4), 114 (16.8), 84 (100), 51 (45.4).

Crystal data

$C_{15}H_{10}ClNO_3S$
$M_r = 319.75$
Triclinic, $P\bar{1}$
$a = 8.048$ (2) Å
$b = 9.568$ (3) Å
$c = 9.931$ (3) Å
$\alpha = 79.433$ (4)°
$\beta = 82.145$ (4)°
$\gamma = 69.839$ (4)°
$V = 703.5$ (4) Å^3
$Z = 2$
$D_x = 1.510$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2805 reflections
$\theta = 2.1$–27.1°
$\mu = 0.43$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.26 × 0.20 × 0.19 mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.902$, $T_{max} = 0.922$
5482 measured reflections
2805 independent reflections
2240 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 27.1°$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.144$
$S = 1.03$
2805 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0754P)^2 + 0.2825P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C2—N1	1.425 (3)	O1—S1	1.416 (2)
C5—N1	1.414 (3)	O2—S1	1.419 (2)
N1—S1	1.684 (2)		
C5—C4—C9	119.4 (3)	O2—S1—N1	105.61 (12)
C6—C5—C4	121.6 (3)	O1—S1—C10	109.76 (13)
O1—S1—O2	119.87 (14)	O2—S1—C10	109.54 (13)
O1—S1—N1	105.92 (13)	N1—S1—C10	105.00 (10)
Cl1—C3—C4—C5	−174.54 (19)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1	0.93	2.41	2.991 (8)	120
C16—H16···O2	0.93	2.34	2.803 (4)	111
C6—H6···O1^{i}	0.93	2.54	3.333 (4)	143
C15—H15···O3^{ii}	0.93	2.58	3.257 (5)	130

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

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H = 0.93 or 0.96 Å and U_{iso}(H) = 1.2–1.5U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

ZORTEP (Zsolnai, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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