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2,3-Diiodo-1-(phenylsulfonyl)-1*H*-indole

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.009 Å
R factor = 0.032
wR factor = 0.082
Data-to-parameter ratio = 13.3

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

In the title compound, $C_{14}H_9I_2NO_2S$, the two I atoms lie essentially in the plane of the indole ring and there is some iodine–iodine repulsion.

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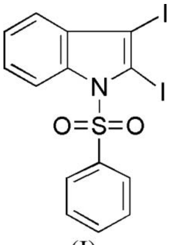
Comment

In connection with our interest in the chemistry of halogenated indoles (Roy & Gribble, 2006; Liu & Gribble, 2002*a*,*b*; Saulnier & Gribble, 1982), we have synthesized the title compound, (I), and report its crystal structure here (Fig. 1).


(I)

The observed parameters for (I) are generally comparable to the reported values for other 1-(phenylsulfonyl)indoles (Beddoes *et al.*, 1986; Schollmeyer *et al.*, 1995; Yokum & Fronczek, 1997; Govindasamy *et al.*, 1998; Sankaranarayanan *et al.*, 2000; Sonar *et al.*, 2004; Palani *et al.*, 2006*b*). The sum of the angles around the indole N atom is 351.8°, indicating significant pyramidalization of the nitrogen. The two C—I bond lengths are 2.060 (4) and 2.062 (4) Å. The I1—C2—C3—I2 torsion angle is 5.1 (6)°, indicating some repulsion between the two iodines, as is also seen in 1,8-diiodonaphthalene (Bock *et al.*, 1998). Accordingly, the I2—C3—C8—C9 and I1—C2—C3—C8 torsion angles are 173.6 (3) and −179.2 (3)°, respectively, which differ from those reported for 2,3-dimethyl-1-(phenylsulfonyl)indole [178.2 (2) and 178.6 (2)°, respectively; Palani *et al.*, 2006*b*]. The iodine···iodine separation is 3.08 (4) Å, compared to 3.51 (1)Å in 1,8-diiodonaphthalene (Bock *et al.*, 1998). As might be expected, the C2–C3 indole double bond length of 1.368 (6) Å is elongated relative to the C2–C3 bond length of 1.349 (11) Å in 2,3-diphenyl-1-(phenylsulfonyl)indole (Liu *et al.*, 2007) and of 1.341 (3) Å in 2,3-dimethyl-1-(phenylsulfonyl)indole (Palani *et al.*, 2006*b*).

Experimental

The procedure used for the preparation of (I) is that of Saulnier & Gribble (1982). A solution of lithium diisopropylamide (LDA) (2 *M*, 2.2 ml, 4.4 mmol) was added dropwise to a brown solution of 3-iodo-1-(phenylsulfonyl)-1*H*-indole (1.04 g, 2.71 mmol) in anhydrous THF (30 ml), stirring at 195 K. The reaction mixture was stirred for 55 min.

A solution of iodine (0.851 g, 3.35 mmol) in anhydrous THF (20 ml) was added *via* a cannula. The red–brown reaction mixture was allowed to gradually warm up to room temperature. It was poured on to saturated aqueous sodium thiosulfate (50 ml) at 273 K. The organic layer was extracted with dichloromethane (3 × 50 ml), washed with distilled water (2 *x* 50 ml) and brine (2 × 50 ml), and dried with anhydrous magnesium sulfate. The solvent was evaporated *in vacuo*, yielding an orange solid [0.901 g, 1.77 mmol, 65%; m.p. 431–432 K (literature m.p. 439–440 K; Saulnier & Gribble, 1982)]. Crystals suitable for X-ray determination were grown from diethyl ether–hexane (1:1).

Crystal data

$C_{14}H_9I_2NO_2S$
$M_r = 509.11$
Monoclinic, $P2_1/n$
$a = 10.4680$ (15) Å
$b = 11.9852$ (18) Å
$c = 12.4945$ (19) Å
$\beta = 109.550$ (3)°
$V = 1477.2$ (4) Å^3
$Z = 4$
$D_x = 2.289$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 4.40$ mm^{-1}
$T = 100$ K
Prism, violet
0.50 × 0.26 × 0.12 mm

Data collection

Bruker-Nonius Kappa Apex2 diffractometer
φ and ω scans
Absorption correction: multi-scan *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997)
$T_{min} = 0.250$, $T_{max} = 0.590$
9300 measured reflections
3407 independent reflections
2422 reflections with $I > 3.0\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 28.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.082$
$S = 0.95$
2422 reflections
182 parameters
H-atom parameters constrained
$w = [1 - (F_o - F_c)^2/36\sigma^2(F)]^2/[37.8T_0(x) + 38.0T_1(x) \cdots + 29.3T_2(x)]$ where T_i are Chebychev polynomials and $x = F_c/F_{max}$ (Prince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -1.53$ e Å^{-3}
Extinction correction: Larson (1970), Equation 22
Extinction coefficient: 6.2 (16)

H atoms were included in the riding-model approximation, with C—H = 1.00 Å and $U_{iso}(H) = 1.17–1.22U_{eq}(C)$. Changes in illuminated volume were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997).

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-NT* (Bruker, 2006); data reduction: *SAINT-NT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

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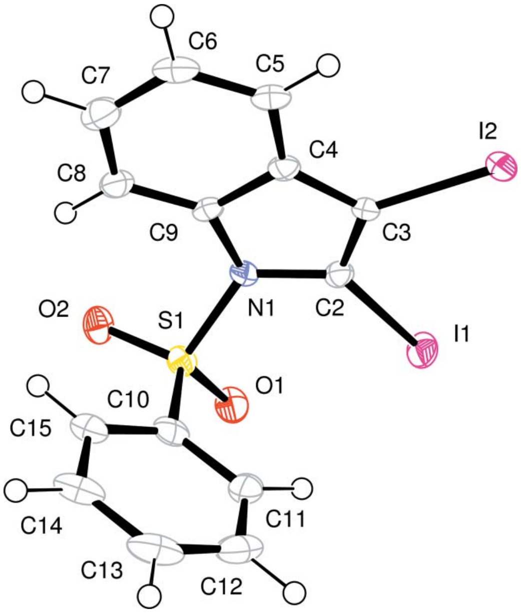

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radius.

References

Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Beddoes, R. L., Dalton, L., Joule, J. A., Mills, O. S., Street, J. D. & Watt, C. I. F. (1986). *J. Chem. Soc. Perkin Trans. 2*, pp. 787–797.

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.

Bock, H., Sievert, M. & Havlas, Z. (1998). *Chem. Eur. J.* **4**, 677–685.

Bruker (2006). *APEX2* (Version 2) and *SAINT-NT* (Version 6). Bruker AXS Inc., Madison, Wisconsin, USA.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Görbitz, C. H. (1999). *Acta Cryst.* B**55**, 1090–1098.

Govindasamy, L., Velmurugan, D., Ravikumar, K. & Mohanakrishnan, A. K. (1998). *Acta Cryst.* C**54**, 277–279.

Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall and C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

Liu, Y. & Gribble, G. W. (2002*a*). *Tetrahedron Lett.* **43**, 7135–7137.

Liu, Y. & Gribble, G. W. (2002*b*). *J. Nat. Prod.* **65**, 748–749.

Liu, Y., Gribble, G. W. & Jasinski, J. P. (2007). *Acta Cryst.* E**63**, o738–o740.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Palani, K., Ponnuswamy, M. N., Jaisankar, P., Srinivasan, P. C. & Nethaji, M. (2006*a*). *Acta Cryst.* E**62**, o440–o442.

Palani, K., Ponnuswamy, M. N., Jaisankar, P., Srinivasan, P. C. & Nethaji, M. (2006*b*). *Acta Cryst.* E**62**, o85–o87.

Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials*. New York: Springer-Verlag.

Roy, S. & Gribble, G. W. (2006). *Synth. Commun.* **36**, 3487–3492.

Sankaranarayanan, R., Velmurugan, D., Shanmuga Sundara Raj, S., Fun, H.-K., Babu, G. & Perumal, P. T. (2000). *Acta Cryst.* C**56**, 475–476.

Saulnier, M. G. & Gribble, G. W. (1982). *J. Org. Chem.* **47**, 757–761.

Schollmeyer, D., Fischer, G. & Pindur, U. (1995). *Acta Cryst.* C**51**, 2572–2575.

Sonar, V. N., Parkin, S. & Crooks, P. A. (2004). *Acta Cryst.* C**60**, o659–o661.

Watkin, D. (1994). *Acta Cryst.* A**50**, 411–437.

Yokum, T. S. & Fronczek, F. R. (1997). *Acta Cryst.* C**53**, 362–363.

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