

2-Iodobenzenesulfonyl chloride

Stuart D. Robertson,
Alexandra M. Z. Slawin and
J. Derek Woollins*

Department of Chemistry, University of St
Andrews, St Andrews KY16 9ST, Scotland

Correspondence e-mail: jdw3@st-and.ac.uk

Key indicators

Single-crystal X-ray study

$T = 93$ K

Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å

R factor = 0.016

wR factor = 0.033

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>.

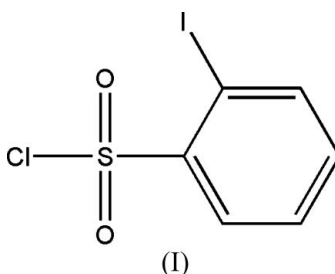
In the molecule of 2-iodobenzenesulfonyl chloride, $\text{C}_6\text{H}_4\text{IO}_2\text{S}^+\cdot\text{Cl}^-$, the *ortho* substitution by large atoms causes angular distortions at the ring C atoms rather than significant displacement of the substituents out of the ring plane.

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Comment

The title compound, (I) (Fig. 1), was prepared as an intermediate in the synthesis of dibenzo[*ce*][1,2]dithiine and its related oxides (Aucott *et al.*, 2004; Aucott *et al.*, 2004*a,b*; Aucott, Kilian *et al.*, 2005; Aucott, Milton *et al.*, 2005) as part of a study of conformationally restricted molecules.



Compound (I) crystallizes in the monoclinic space group $P2_1/n$. The aromatic ring is essentially planar, with atom S1 0.14 (1) Å and I1 -0.08 (1) Å from this plane. The SO_2Cl group is oriented with O2 close to the aromatic plane [0.16 (1) Å] and O1 and Cl1 lying 1.13 (1) and -1.165 (1) Å above and below this plane, respectively. The *ortho* substitution of two heavy atoms results in enlargement of angles at carbon of the aromatic ring; $\text{C}2-\text{C}1-\text{I}1 = 125.00$ (18)° and $\text{S}1-\text{C}2-\text{C}1 = 123.23$ (19)°.

Experimental

2-Iodobenzenesulfonyl chloride was prepared as previously described (Chau & Kice, 1977) and was crystallized from chloroform/hexane (1:1 *v/v*) to give well formed colourless blocks.

Crystal data

$\text{C}_6\text{H}_4\text{IO}_2\text{S}^+\cdot\text{Cl}^-$
 $M_r = 302.50$
Monoclinic, $P2_1/n$
 $a = 8.338$ (3) Å
 $b = 12.741$ (3) Å
 $c = 8.517$ (2) Å
 $\beta = 109.797$ (7)°
 $V = 851.3$ (4) Å³
 $Z = 4$

$D_x = 2.360$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 3111
reflections
 $\theta = 2.5$ – 25.4 °
 $\mu = 4.26$ mm⁻¹
 $T = 93$ (2) K
Block, colourless
 $0.10 \times 0.10 \times 0.08$ mm

Data collection

Rigaku MM007/Mercury CCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.600$, $T_{\max} = 0.710$
 4840 measured reflections

1496 independent reflections
 1457 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$
 $\theta_{\text{max}} = 25.4^\circ$
 $h = -10 \rightarrow 9$
 $k = -15 \rightarrow 12$
 $l = -8 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.016$
 $wR(F^2) = 0.033$
 $S = 1.13$
 1496 reflections
 101 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0044P)^2 + 1.4521P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.47 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.46 \text{ e } \text{\AA}^{-3}$

All H atoms were included in calculated positions ($\text{C}-\text{H} = 0.95 \text{ \AA}$) and were refined as riding atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *CrystalClear* (Rigaku/MS, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

References

- Aucott, S. M., Kilian, P., Milton, H. L., Robertson, S. D., Slawin, A. M. Z. & Woollins, J. D. (2005). *Inorg. Chem.* **44**, 2710–2718.
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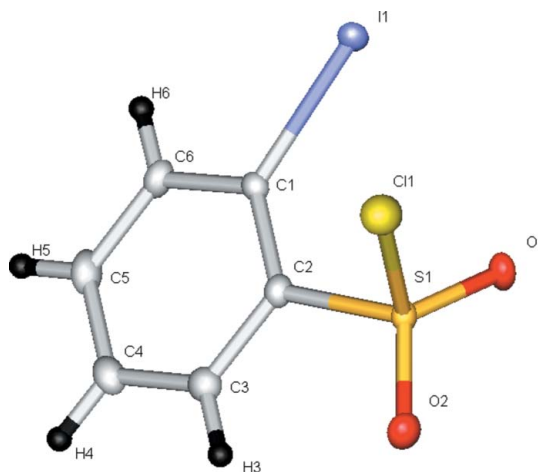


Figure 1

The structure of (I), with displacement ellipsoids drawn at the 50% probability level.

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supporting information

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Crystal data

$C_6H_4IO_2S^+Cl^-$

$M_r = 302.50$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 8.338\ (3)\ \text{\AA}$

$b = 12.741\ (3)\ \text{\AA}$

$c = 8.517\ (2)\ \text{\AA}$

$\beta = 109.797\ (7)^\circ$

$V = 851.3\ (4)\ \text{\AA}^3$

$Z = 4$

$F(000) = 568$

$D_x = 2.360\ \text{Mg m}^{-3}$

Melting point: 52 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3111 reflections

$\theta = 2.5\text{--}25.4^\circ$

$\mu = 4.27\ \text{mm}^{-1}$

$T = 93\ \text{K}$

Block, colourless

$0.10 \times 0.10 \times 0.08\ \text{mm}$

Data collection

CCD

diffractometer

Radiation source: rotating anode

Confocal monochromator

Detector resolution: $0.83\ \text{pixels mm}^{-1}$

dtprofit.ref scans

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

$T_{\min} = 0.600$, $T_{\max} = 0.710$

4840 measured reflections

1496 independent reflections

1457 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.013$

$\theta_{\max} = 25.4^\circ$, $\theta_{\min} = 3.0^\circ$

$h = -10 \rightarrow 9$

$k = -15 \rightarrow 12$

$l = -8 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.016$

$wR(F^2) = 0.033$

$S = 1.13$

1496 reflections

101 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0044P)^2 + 1.4521P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.47\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.46\ \text{e \AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.42383 (2)	0.287007 (12)	0.57459 (2)	0.01844 (6)
Cl1	0.02906 (7)	0.14086 (5)	0.52384 (8)	0.02264 (14)
S1	0.19229 (7)	0.11930 (5)	0.76112 (7)	0.01317 (13)
O1	0.2205 (2)	0.22062 (13)	0.8354 (2)	0.0182 (4)
O2	0.1205 (2)	0.03818 (14)	0.8311 (2)	0.0202 (4)
C1	0.4827 (3)	0.13418 (19)	0.6674 (3)	0.0139 (5)
C2	0.3799 (3)	0.07205 (19)	0.7313 (3)	0.0136 (5)
C3	0.4261 (3)	-0.0310 (2)	0.7824 (3)	0.0166 (5)
H3A	0.3538	-0.0732	0.8218	0.020*
C4	0.5777 (3)	-0.0718 (2)	0.7755 (3)	0.0193 (5)
H4A	0.6104	-0.1416	0.8113	0.023*
C5	0.6816 (3)	-0.0095 (2)	0.7156 (3)	0.0195 (5)
H5A	0.7863	-0.0369	0.7120	0.023*
C6	0.6340 (3)	0.0915 (2)	0.6613 (3)	0.0172 (5)
H6A	0.7056	0.1324	0.6192	0.021*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02291 (10)	0.01213 (9)	0.02630 (10)	-0.00095 (6)	0.01621 (7)	0.00174 (6)
Cl1	0.0135 (3)	0.0286 (3)	0.0237 (3)	0.0021 (3)	0.0034 (2)	0.0029 (3)
S1	0.0122 (3)	0.0123 (3)	0.0173 (3)	-0.0002 (2)	0.0079 (2)	-0.0014 (2)
O1	0.0191 (9)	0.0130 (9)	0.0281 (10)	-0.0027 (7)	0.0153 (8)	-0.0062 (7)
O2	0.0192 (9)	0.0195 (9)	0.0272 (10)	-0.0004 (8)	0.0147 (8)	-0.0002 (8)
C1	0.0145 (12)	0.0118 (12)	0.0161 (11)	-0.0011 (9)	0.0061 (9)	-0.0020 (10)
C2	0.0110 (12)	0.0159 (12)	0.0143 (11)	-0.0007 (9)	0.0046 (9)	-0.0035 (10)
C3	0.0178 (12)	0.0168 (13)	0.0151 (12)	-0.0021 (10)	0.0054 (10)	-0.0007 (10)
C4	0.0213 (13)	0.0153 (13)	0.0186 (12)	0.0057 (10)	0.0031 (10)	-0.0006 (10)
C5	0.0128 (12)	0.0230 (14)	0.0213 (13)	0.0035 (10)	0.0041 (10)	-0.0062 (11)
C6	0.0134 (12)	0.0217 (14)	0.0188 (12)	-0.0033 (10)	0.0086 (10)	-0.0064 (10)

Geometric parameters (\AA , $^\circ$)

I1—C1	2.096 (2)	C3—C4	1.386 (4)
Cl1—S1	2.0368 (10)	C3—H3A	0.9500
S1—O1	1.4217 (18)	C4—C5	1.392 (4)
S1—O2	1.4222 (18)	C4—H4A	0.9500
S1—C2	1.773 (2)	C5—C6	1.379 (4)
C1—C6	1.390 (3)	C5—H5A	0.9500
C1—C2	1.405 (3)	C6—H6A	0.9500

C2—C3	1.396 (3)		
O1—S1—O2	120.22 (11)	C4—C3—C2	119.9 (2)
O1—S1—C2	110.53 (11)	C4—C3—H3A	120.0
O2—S1—C2	109.27 (11)	C2—C3—H3A	120.0
O1—S1—C11	105.86 (8)	C3—C4—C5	119.4 (2)
O2—S1—C11	106.21 (8)	C3—C4—H4A	120.3
C2—S1—C11	103.26 (8)	C5—C4—H4A	120.3
C6—C1—C2	118.3 (2)	C6—C5—C4	120.7 (2)
C6—C1—I1	116.68 (18)	C6—C5—H5A	119.7
C2—C1—I1	125.00 (18)	C4—C5—H5A	119.7
C3—C2—C1	120.7 (2)	C5—C6—C1	120.9 (2)
C3—C2—S1	116.03 (18)	C5—C6—H6A	119.5
C1—C2—S1	123.23 (19)	C1—C6—H6A	119.5
C6—C1—C2—C3	-2.0 (3)	C11—S1—C2—C1	68.7 (2)
I1—C1—C2—C3	176.66 (17)	C1—C2—C3—C4	2.2 (4)
C6—C1—C2—S1	175.12 (18)	S1—C2—C3—C4	-175.14 (19)
I1—C1—C2—S1	-6.2 (3)	C2—C3—C4—C5	-0.8 (4)
O1—S1—C2—C3	133.11 (19)	C3—C4—C5—C6	-0.8 (4)
O2—S1—C2—C3	-1.3 (2)	C4—C5—C6—C1	1.0 (4)
C11—S1—C2—C3	-114.05 (18)	C2—C1—C6—C5	0.4 (4)
O1—S1—C2—C1	-44.1 (2)	I1—C1—C6—C5	-178.36 (18)
O2—S1—C2—C1	-178.58 (19)		
