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(2*SR*,4*RS*)-(4-Iodo-5,5-dimethyl-2-hexylsulfonyl)benzene

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From a hexane solution, the title compound, $C_{14}H_{21}IO_2S$, crystallizes in the centrosymmetric space group *Pbca* with one molecule in the asymmetric unit. As a result of the bulky substituent, small deviations from ideal bond angles are found for the aliphatic part of the molecule.

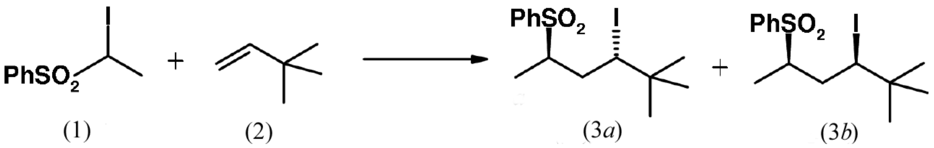
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.037
wR factor = 0.069
Data-to-parameter ratio = 13.4

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

Comment

In the course of our continuing study on atom-transfer free radical additions of α-iodoalkylphenyl sulfones to alkenes, we found secondary iodide (1) reacting with terminal alkene (2) under standard conditions (Jankowski *et al.*, 1995; Masnyk, 1991), producing a chromatographically separable mixture of diastereomers (3*a*) and (3*b*) in a 1:1.2 ratio. The correlation of the crystal structure with the corresponding NMR spectrum will enable the application of NMR techniques to stereochemical assignments of other related compounds resulting from analogous reactions. We now present the crystal structure of (3*a*) (Fig. 1).



(1) (2) (3*a*) (3*b*)

The compound crystallizes in the centrosymmetric space group *Pbca* with one molecule in the asymmetric unit. All bond lengths, bond angles and torsion angles are in the expected ranges. This includes a significantly enlarged O—S—O angle of 118.2° for the S═O double bonds. The geometric deviations at atoms C3 and C4, as expressed by bond angles of 116.3 (4) and 116.0 (4)°, respectively, can be related to the steric effect of bulky groups attached to C3 (*tert*-butyl and iodine) and C4 [–CH(CH_3) and –$SO_2(C_6H_5)$] favouring a more stretched arrangement of the aliphatic part of the molecule.

Experimental

A mixture of (1-iodoethanesulfonyl)benzene (1) (600 mg), 3,3-dimethylbut-1-ene (2) (1.0 ml), benzene (1.5 ml) and benzoyl peroxide (40 mg) was heated for 6 h at 373 K in a sealed tube. The reaction mixture was then chromatographed on silica gel (hexanes–ethyl acetate 93:7) yielding (3*a*) (280 mg, isomer *anti*, 2*S*4*R* and 2*R*4*S*) and (3*b*) (350 mg, isomer *syn*, 2*R*4*R* and 2*S*4*S*). Isometric crystals of (3*a*) were grown from hexane solution at room temperature over the course of a few days (m.p. = 346–348 K), ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.10 (*s*, 9H), 1.26 (*d*, J = 7.1 Hz, 1H), 1.88 (*ddd*, J = 16.4, 12.0, 1.9 Hz, 1H), 2.53 (*ddd*, J = 16.4, 8.5, 1.9 Hz, 1H), 3.45 (*m*, 1 H), 4.42 (*dd*, J = 12.0, 1.9 Hz, 1H), 7.57–7.61 (*m*, 2H), 7.68 (*tt*, J = 7.4, 1.3 Hz,

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1H), 7.89–7.92 (*m*, 2H). ^{13}C NMR (125 MHz, CDCl$_3$): δ = 16.72, 28.26, 35.98, 37.53, 56.14, 61.21, 128.88, 129.21, 133.79, 137.64.

Crystal data

$C_{14}H_{21}IO_2S$
$M_r = 380.27$
Orthorhombic, *Pbca*
$a = 7.6427$ (14) Å
$b = 11.035$ (2) Å
$c = 37.950$ (5) Å
$V = 3200.6$ (10) Å^3
$Z = 8$
$D_x = 1.578$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 30 reflections
$\theta = 7.7$–13.8°
$\mu = 2.13$ mm^{-1}
$T = 293$ (2) K
Isometric, colourless
0.90 × 0.80 × 0.57 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: numerical (Stoe & Cie, 1996)
$T_{\min} = 0.215$, $T_{\max} = 0.362$
2815 measured reflections
2815 independent reflections
1891 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 45$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.069$
$S = 1.01$
2815 reflections
210 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.012P)^2 + 9P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.55$ e Å^{-3}
$\Delta\rho_{\min} = -0.55$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00258 (8)

Table 1
Selected geometric parameters (Å, °).

I1—C3	2.198 (4)	S1—C5	1.803 (5)
S1—O1	1.437 (3)	C2—C3	1.535 (6)
S1—O2	1.443 (3)	C3—C4	1.526 (6)
S1—C11	1.776 (5)		
O1—S1—O2	118.2 (2)	C3—C2—C8	112.8 (4)
O1—S1—C11	107.4 (2)	C3—C2—C7	106.9 (4)
O2—S1—C11	108.2 (2)	C4—C3—C2	116.3 (4)
O1—S1—C5	107.8 (2)	C4—C3—I1	106.1 (3)
O2—S1—C5	107.9 (2)	C2—C3—I1	112.1 (3)
C11—S1—C5	106.9 (2)	C3—C4—C5	116.0 (4)
C1—C2—C3	110.0 (4)		
C1—C2—C3—C4	179.5 (4)	C7—C2—C3—I1	173.4 (4)
C8—C2—C3—C4	56.3 (6)	C2—C3—C4—C5	166.5 (4)
C7—C2—C3—C4	−64.4 (6)	I1—C3—C4—C5	−68.1 (4)
C1—C2—C3—I1	57.2 (5)	C3—C4—C5—C6	144.9 (4)
C8—C2—C3—I1	−66.0 (5)		

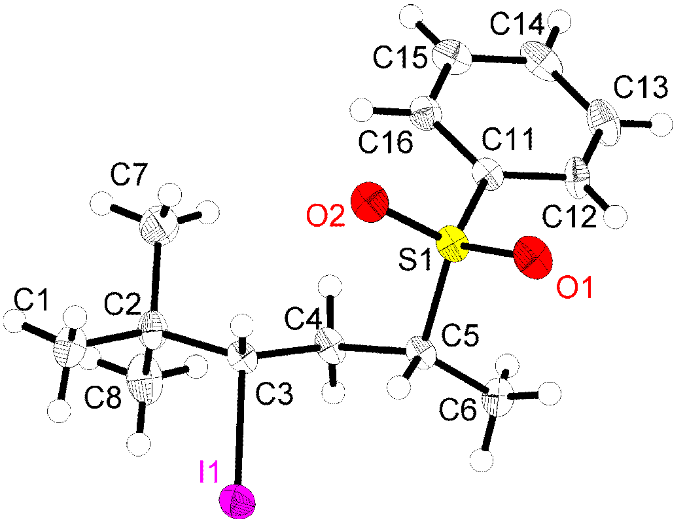

Figure 1
The molecular structure of (3*a*). Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn with an arbitrary radius.

H atoms belonging to CH$_2$ groups and aromatic H atoms were included in the final stages of refinement, using a riding model with one common $U_{\rm iso}$ value refined for each group. H atoms belonging to methyl groups were included in the refinement, riding on their attached C atom and allowed to rotate about the C—C bond with one common $U_{\rm iso}$ value refined for each methyl group. The H atoms at C3 and C5 (C—H groups) were refined freely, together with their individual $U_{\rm iso}$ values.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg, 1998); software used to prepare material for publication: *SHELXL*97.

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