(2-Isopropyl-1,1-methylcyclopropanesulfonyl)-benzene

Guido J. Reiß[a]* and Marek Masnyk[b]

[a]Institut für Anorganische Chemie und Strukturchemie, Heinrich-Heine-Universität Düsseldorf, Universitätsstraße, D-40225 Düsseldorf, Germany, and [b]Institute of Organic Chemitry, Polish Academy of Sciences, Kasprzaka 44, 01-224 Warsaw, Poland

Correspondence e-mail: reissg@uni-duesseldorf.de

The title compound, $C_{13}H_{18}O_2S$, crystallizes from a methanol solution in the centrosymmetric space group $P2_1/n$, with one molecule in the asymmetric unit. The stacking of the molecules shows layers of the phenylsulfonyl groups and of the aliphatic part of the molecule, respectively, each arranged parallel to the (101) plane.

Comment

In the course of our study on free-radical reactions of α-iodoalkyl phenyl sulfones, we found an easy route to compounds containing 1-iodo-3-phenylsulfonyl moieties as a mixture of diastereomers (Masnyk, 1991). These compounds undergo intramolecular nucleophilic substitution upon treatment with bases, providing cyclopropane phenylsulfonyl derivatives (Jankowski *et al.*, 1995). In particular, 3-iodo-2,5-dimethylhexane, (1), reacts with sodium bis(trimethylsilyl)-amide, yielding the title compound, (2), as a single stereo-isomer (see *Scheme*).
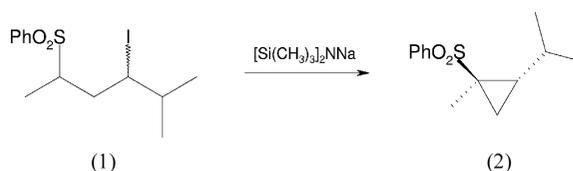
The crystal structure of (2) confirms the *trans* arrangement of the 1-phenylsulfonyl and the 2-isopropyl substituents on the cyclopropane ring, which seems to be a general rule for all related compounds coming from analogous reactions. All bond lengths, angles and torsion angles in the molecule (Fig. 1) are in the expected ranges. This includes the fact that the tetrahedral coordination at the S atom is seriously distorted (Table 1) and very similar to related iodo compounds (Reiß & Masnyk, 2001a,b). It is worth mentioning that the stacking of the molecules shows a separation of the phenylsulfonyl groups and of the aliphatic part of the molecule into layers aligned parallel to the (101) plane (Fig. 2).

Experimental

Sodium bis(trimethylsilyl)amide (90 mg, 0.50 mmol) was added to a stirred solution of (1) (120 mg, 0.33 mmol) in benzene (4.0 ml) at room temperature and the stirring was continued for an additional 3 h. The mixture was the diluted with ether (20 ml) and washed with water (2 × 20 ml). The organic layer was dried over $MgSO_4$ and the solvent was evaporated. The residue was filtered through silica gel (hexanes–ethyl acetate, 9:1), yielding the title compound, (2) (72 mg, 92%). Recrystallization from methanol gave isometric colourless single crystals (m.p. = 357–358 K; ^{1}H NMR (500 MHz, CDCl$_3$): δ 0.52
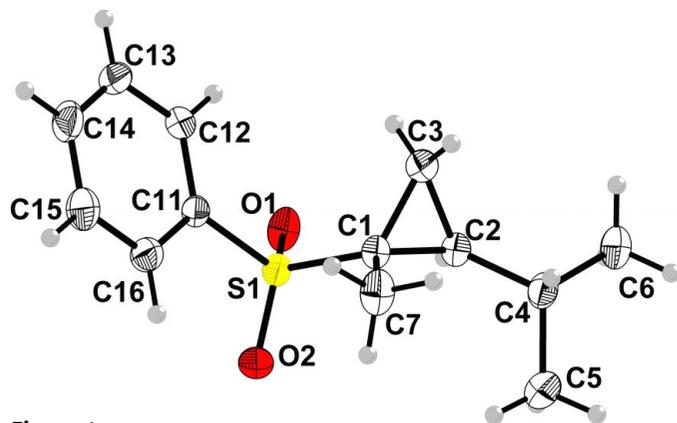
Figure 1
The molecular structure of the title compound. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn with an arbitrary radius.

(dd, J = 6.6, 5.0 Hz, 1H), 0.81 (d, J = 6.5 Hz, 3H), 1.05 (d, J = 6.5 Hz, 3H), 1.09–1.21 (m, 1H), 1.34 (s, 3H), 1.63 (td, J = 10.0, 6.6 Hz, 1H), 1.70 (dd, J = 10.0, 5.0 Hz, 1H), 7.53–7.57 (m, 2H), 7.61–7.65 (m, 1H), 7.85–7.88 (m, 2H). ^{13}C NMR (125 MHz, CDCl$_3$): δ 13.02, 17.67, 22.18, 22.31, 28.64, 31.06, 41.46, 128.80, 128.87, 133.21, 138.49. The IR spectra were recorded on a Bio-Rad FTS-3500 FT–IR spectrometer with a resolution of 8 cm^{-1}. A single-crystal of dimensions 0.4 × 0.3 × 0.2 mm was fixed on the ZnSe plate of the single reflection ATR (attenuated total reflectance)-accessory unit (MIRacle, PIKE-Technologies, Madison). 4000–650 cm^{-1}: 3069, 3023, 2970, 2934, 2870, 1585, 1447, 1297, 1280, 1139, 1082, 1043, 801, 762, 727, 690, 660, 573, 559.

Crystal data

C$_{13}$H$_{18}$O$_2$S	D_x = 1.207 Mg m^{-3}
M_r = 238.33	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1240
a = 8.514 (3) Å	reflections
b = 15.212 (5) Å	θ = 4.9–10.2°
c = 10.260 (3) Å	μ = 0.23 mm^{-1}
β = 99.31 (3)°	T = 293 (2) K
V = 1311.4 (7) Å^3	Block, colourless
Z = 4	0.40 × 0.35 × 0.30 mm

Data collection

Stoe CCD diffractometer	R_{int} = 0.050
ω scans	θ_{max} = 25.0°
Absorption correction: none	h = −10 → 10
22065 measured reflections	k = −18 → 18
2313 independent reflections	l = −12 → 12
2164 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.012P)^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$+ 1.1P]$
$wR(F^2)$ = 0.090	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.002
2313 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
173 parameters	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: $SHELXL$97
	Extinction coefficient: 0.0034 (7)

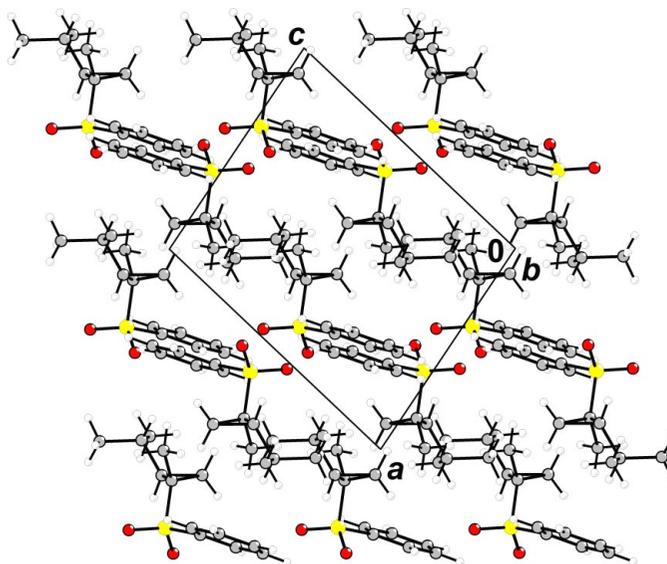**Figure 2**
The packing of the title compound, showing layers parallel to the (101) plane.

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4388 (16)	S1—C1	1.766 (2)
S1—O2	1.4405 (17)	S1—C11	1.767 (2)
O1—S1—O2	118.47 (11)	C2—C1—C7	122.41 (19)
O1—S1—C1	108.67 (10)	C3—C1—S1	115.67 (17)
O2—S1—C1	108.16 (10)	C2—C1—S1	113.54 (15)
O1—S1—C11	107.33 (10)	C7—C1—S1	114.07 (16)
O2—S1—C11	108.18 (10)	C3—C2—C4	123.0 (2)
C1—S1—C11	105.25 (10)	C3—C2—C1	60.34 (15)
C3—C1—C2	59.31 (15)	C2—C3—C1	60.35 (15)
C3—C1—C7	121.2 (2)		
C11—S1—C1—C3	−81.73 (18)	O2—S1—C11—C12	−156.87 (17)
S1—C1—C2—C4	−140.77 (19)	C1—S1—C11—C12	87.69 (19)
O1—S1—C11—C12	−28.0 (2)		

For the data collection, 23 ω scans (1.0°) with different χ, φ and 2θ settings and a detector-to-crystal distance of 60 mm and an exposure time of 18 s were performed. Integration procedures using variable integration masks and an integration width of 1.0° yielded a data completeness of 99.9%. Atomic coordinates of the H atoms belonging to CH$_2$ and CH groups were refined freely, as were the related individual U_{iso} values. Aromatic H atoms and the H atoms of the methyl groups were included in the final stages of refinement, using a riding model, with individual U_{iso} values for aromatic H atoms and one common U_{iso} value refined for the H atoms of each methyl group.

Data collection: *CrysAlis* (Kuma, 2000); cell refinement: *CrysAlis RED* (Kuma, 2000); data reduction: *CrysAlis RED*; 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.

References

Brandenburg, K. (1998). *DIAMOND*. Version 2.0. Gerhard-Domagk-Straße 1, Bonn, Germany.

Jankowski, P., Masnyk, M. & Wicha, J. (1995). *Synlett*, pp. 866–868.

Kuma (2000). *CrysAlis* (Version 1.166) and *CrysAlis RED* (Version 1.166). Kuma Diffraction Instruments, Wrocław, Poland.

Masnyk, M. (1991). *Tetrahedron Lett.* **32**, 3259–3262.

Reiß, G. J. & Masnyk, M. (2001*a*). *Acta Cryst.* E**57**, o959–o960.

Reiß, G. J. & Masnyk, M. (2001*b*). *Acta Cryst.* E**57**, o961–o963.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.