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Key indicators

Single-crystal X-ray study
 $T = 93\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$
 R factor = 0.046
 wR factor = 0.081
Data-to-parameter ratio = 16.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

(2S)-2-[(1R)-1-Iodoethyl]-1-(4-methylphenyl-sulfonyl)pyrrolidine

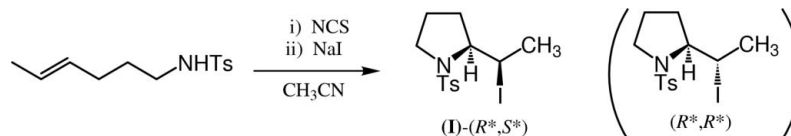
The spectroscopically confused title compound, $\text{C}_{13}\text{H}_{18}\text{INO}_2\text{S}$, wrongly assigned previously, has been crystallographically identified. The relative configuration is the (R^*,S^*) form and not the (R^*,R^*) type as previously reported.

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Comment

The title compound, (I), was obtained by (i) *N*-chlorination of *N*-[(*E*)-4-hexenyl](4-methylbenzene)sulfonamide with *N*-chlorosuccinimide (NCS), and then (ii) iodocycloamination with NaI, as shown in the scheme. The spectroscopic data (^1H and ^{13}C NMR) were unexpectedly identical to those of a compound that had been synthesized from (*Z*)-1-iodo-4-hexene with chloramine-T and which has been reported as having an (R^*,R^*) configuration (Minakata *et al.*, 2002). Having a heavy element (iodine) in the molecule allowed for the assignment of the absolute configuration. Consequently, the relative configuration was confirmed, as shown in Fig. 1 to be (R^*,S^*).The sum of the bond angles at the sulfonamide N atom is 346° , with an average angle of 115° . Pyramidal N atoms have average angles in the range $108\text{--}114^\circ$. The five-membered ring has the following puckering parameters (Cremer & Pople, 1975): $q_2 = 0.394(5)\text{ \AA}$ and $\varphi_2 = 280.4(7)^\circ$. The latter parameter is closer to the value (288°) for an envelope conformation than that of a twist conformation (270°). C4 is the envelope flap.

Experimental

The title compound was prepared by a partially modified method of another route reported by Minakata *et al.* (2002). A solution of *N*-[(*E*)-4-hexenyl]-4-methylbenzenesulfonamide (0.056 mmol) and *N*-chlorosuccinimide (0.061 mmol) in CH_3CN (0.7 ml) was stirred overnight under N_2 . Subsequently, NaI (0.067 mmol) was added to the reaction mixture, and stirring was continued overnight. The reaction mixture was diluted with diethyl ether and washed with 5% aqueous $\text{Na}_2\text{S}_2\text{O}_3$. The aqueous layer was extracted by diethyl ether. The combined organic layers were washed with brine and dried over MgSO_4 . The solvent was removed to yield the crude title compound. Recrystallization from an ethyl acetate solution gave small colorless needles (m.p. $399\text{--}402\text{ K}$). Spectroscopic analysis: IR (KBr): ν 1445, 1376, 1341, 1154, 994, 815 cm^{-1} ; ^1H NMR (500 MHz; CDCl_3 ; assignments aided by a COSY spectrum; coupling constants in Hz): δ

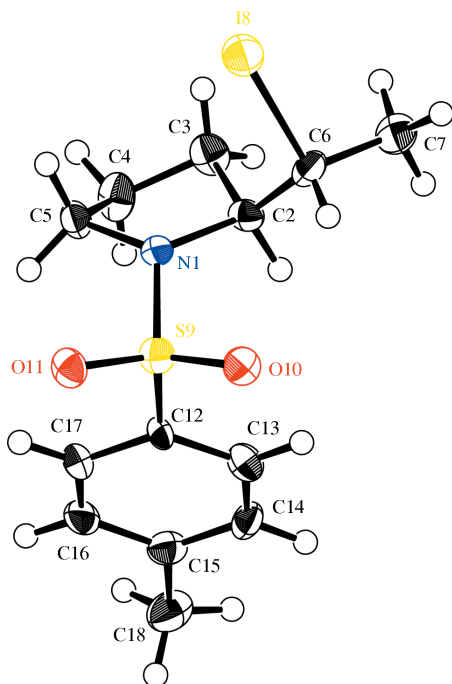


Figure 1
An ORTEP (Johnson, 1976) drawing of the molecule of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

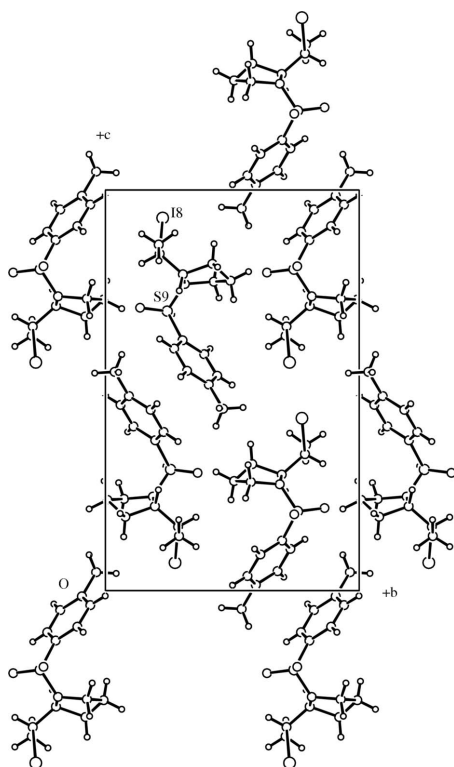


Figure 2
The packing of (I), viewed down the *a* axis.

1.34–1.40 (1H, *m*, NCH₂CH_a), 1.77–1.92 (3H, *m*, NCH₂CH_b and NCHCH_aH_b), 1.87 (3H, *d*, *J* = 6.8 Hz, CH₃), 2.44 (3H, *s*, ArCH₃), 3.12 (1H, *ddd*, *J* = 4.7, 5.6 and 7.7 Hz, NCH), 3.36–3.38 (2H, *m*, NCH_aH_b), 4.77 (1H, *dq*, *J* = 4.7, 6.8, 6.8 and 6.8 Hz, CHI), 7.33 (2H, *d*, *J* = 8.1 Hz,

H_{arom} of Ts), 7.75 (2H, *d*, *J* = 8.1 Hz, H_{arom} of Ts); ¹³C NMR (125 MHz; CDCl₃; assignments aided by an HMQC spectrum): δ 21.6 (ArCH₃), 24.5 (NCH₂–CH₂–), 25.1 (CH₃), 30.4 (NCH–CH₂–), 36.8 (CHI), 49.4 (NCH₂), 65.5 (NCH), 127.6 (C_{arom}H of Ts), 129.8 (C_{arom}H of Ts), 135.1 (C_{arom} of Ts), 143.7 (C_{arom} of Ts).

Crystal data

C₁₃H₁₈INO₂S
M_r = 379.26
 Orthorhombic, *P*2₁2₁2₁
a = 6.2297 (4) Å
b = 12.2538 (8) Å
c = 19.3613 (13) Å
V = 1477.99 (17) Å³
Z = 4
D_x = 1.704 Mg m⁻³

Cu Kα radiation
 Cell parameters from 16475 reflections
 θ = 3.6–68.2°
 μ = 18.30 mm⁻¹
T = 93.1 K
 Needle, colorless
 0.15 × 0.03 × 0.02 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (Higashi, 1995)
T_{min} = 0.510, *T_{max}* = 0.694
 17233 measured reflections

2702 independent reflections
 2426 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.099
 θ_{\max} = 68.2°
h = −7 → 7
k = −14 → 14
l = −22 → 23

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.046
 $wR(F^2)$ = 0.081
S = 1.04
 2702 reflections
 166 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2)]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max}$ = 0.002
 $\Delta\rho_{\max}$ = 1.47 e Å⁻³
 $\Delta\rho_{\min}$ = −0.75 e Å⁻³
 Absolute structure: Flack (1983),
 1110 Friedel Pairs
 Flack parameter: −0.014 (9)

H atoms were refined using a riding model (C–H = 0.95–1.00 Å), with *U_{iso}*(H) set equal to 1.2 (1.5 for methyl groups) times *U_{eq}* of the bonded C atom. The puckering parameters were calculated by PLATON (Spek, 2003). The highest electron-density peak was found about 1 Å from atom I8.

Data collection: *PROCESS-AUTO* (Rigaku Corporation, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku Corporation, 2005); program(s) used to solve structure: *SIR97* (Altomare, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP II* (Johnson, 1976); software used to prepare material for publication: *CrystalStructure*.

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