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(*R*)-1-Iodo-1-phenyl-2-(*p*-tolylsulfonyl)ethene

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.028
wR factor = 0.076
Data-to-parameter ratio = 16.1

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

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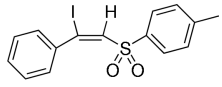
The title compound, $C_{15}H_{13}IO_2S$, displays an *anti* conformation with respect to the olefinic bond. Molecules are linked by weak C—H···O hydrogen bonds.

Comment

β-Iodovinyl sulfones have become generally accepted useful intermediates in organic synthesis, serving efficiently as both Michael acceptors and π partners in cycloaddition reactions (Fuchs & Braish, 1986). Also, as alkenyl halides, they are important building blocks because of their synthetic importance (Negishi, 2002).



(I)

In the structure of the title compound, (I), the phenyl and *p*-toluenesulfonyl groups are located in *cis* positions with respect to the olefinic bond, as shown in Fig. 1. Compared with a related structure reported recently (Gu *et al.*, 2004), the bond angles (Table 1) show unexpected features, probably as a result of steric effects. The angles C1—C2—C3 and S1—C1—C2 are larger than the expected value of 120°, whereas the I—C2—C3, I—C2—C1 and C9—S1—C1 angles are smaller.

The two benzene rings in the molecule are located in *cis* positions with respect to the olefinic bond, forming a dihedral angle of 44.70 (2)°. Atom C2 is coplanar with the C3–C8 benzene ring, and atoms S1 and C15 are coplanar with the other benzene ring (C9–C14). As well as van der Waals forces, the crystal packing (Fig. 2) appears to be influenced by weak C—H···O interactions. Atoms C4 and C6 act as hydrogen-bond donors to symmetry-related sulfone atoms O1 and O2

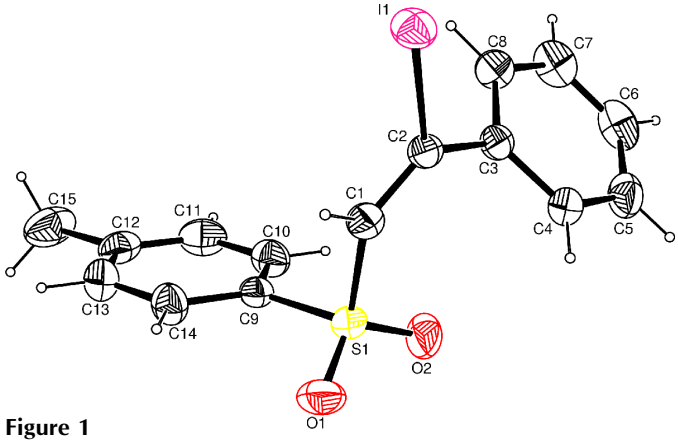


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

(Table 2), forming intermolecular hydrogen bonds. There may also be some weak π–π stacking effects involving the toluene rings. The distance between adjacent parallel toluene ring planes is 3.85 (5) Å, with a centroid-to-centroid distance of 4.52 Å.

Experimental

Sodium *p*-toluenesulfinate (3.75 g, 21 mmol) in 10 ml water was added to I_2 (2.54 g, 10 mmol) and phenylacetylene (1.02 g, 10 mmol) in 20 ml EtOAc. The mixture was stirred vigorously at room temperature for 24 h, then extracted with EtOAc, dried with anhydrous $MgSO_4$ and recrystallized from methanol to produce (I) (Iwata *et al.*, 1992).

Crystal data

$C_{15}H_{13}IO_2S$
M_r = 384.23
Monoclinic, $P2_1/n$
a = 8.0171 (3) Å
b = 12.3697 (4) Å
c = 15.0866 (5) Å
β = 95.685 (1)°
V = 1488.77 (9) Å^3
Z = 4
D_x = 1.714 Mg m^{-3}
Mo Kα radiation
Cell parameters from 10732 reflections
θ = 2.1–27.4°
μ = 2.29 mm^{-1}
T = 296 (1) K
Chunk, yellow
0.40 × 0.40 × 0.25 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.414, T_{max} = 0.565
14526 measured reflections
3396 independent reflections
2771 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.025
θ_{max} = 27.4°
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.076
S = 1.03
2771 reflections
172 parameters
H-atom parameters constrained
$w = 1/[0.0009F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.78 e Å^{-3}

Table 1
Selected bond angles (°).

I1—C2—C1	114.4 (2)	C9—S1—O2	108.4 (1)
I1—C2—C3	114.8 (2)	S1—C1—C2	128.9 (2)
C1—S1—O1	105.0 (1)	C9—S1—C1	102.5 (1)
C1—S1—O2	111.6 (1)	C1—C2—C3	130.8 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2^{i}	0.98	2.48	3.342 (4)	148
C8—H8···O1^{ii}	0.98	2.51	3.429 (4)	156

Symmetry codes: (i) $-x, -y, -z$; (ii) $x-1, y, z$.

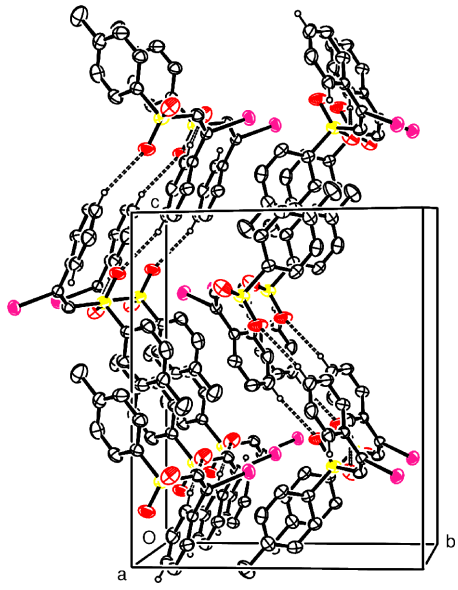


Figure 2
Crystal packing of the title compound along the *a* axis, showing the weak C—H···O interaction.

The H atoms were placed in calculated positions, with C—H = 0.98 Å, and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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