Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,3-Dimethyl-2-(*p*-tolylsulfonyl)butyric acid

Peter G. Jones,[a] Violetta Łysiak,[b] Krystyna Jarzembek[b] and Piotr Kuś[b]*

[a]Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and [b]Department of Chemistry, Silesian University, 9 Szkolna Street, 40-006 Katowice, Poland

Correspondence e-mail: p.jones@tu-bs.de

Key indicators

Single-crystal X-ray study
T = 133 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.105
Data-to-parameter ratio = 23.8

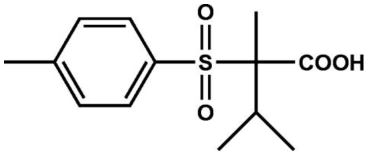
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 May 2006
Accepted 15 May 2006

The title compound, $C_{13}H_{18}O_4S$, displays a $C_{Ph}-S-C-C_{acid}$ torsion angle of 33.23 (8)°; it has been suggested that this angle should be approximately 60° for the molecule to act as an artificial sweetener. Molecules are connected by classical carboxylic acid hydrogen-bond dimers.

Comment

Arylsulfonylalkanoic acids have been synthesized as potential sweeteners (Polański *et al.*, 1997; Doleżych *et al.*, 1999; Ratajczak & Polański, 1991). Crystal structures have been reported for the compounds 2,3-dimethyl-2-phenylsulfonylbutyric acid [which differs from the title compound, (I), an effective artificial sweetener, only in the missing *p*-methyl group (Polański *et al.*, 1997)] and ethyl *p*-nitrophenylsulfonylacetate (Anulewicz *et al.*, 1990).



(I)

The structure of (I) is shown in Fig. 1. The molecule has a chiral centre at C1 (crystallographic numbering), which in Fig. 1 has the *R* configuration, but the overall structure is a racemate. Molecular dimensions may be regarded as normal. Atom O2 is approximately coplanar with the ring. The C11—S—C1—C3 torsion angle is 33.23 (8)°; Polański *et al.* (1997) suggested that this $C_{Ph}-S-C-C_{acid}$ torsion angle should be approximately 60° for the artificial sweetener to be effective.

The packing involves the classical carboxylic acid hydrogen-bond dimer over an inversion centre (Fig. 2). There are no unusually short C—H···O or C—H···π contacts.

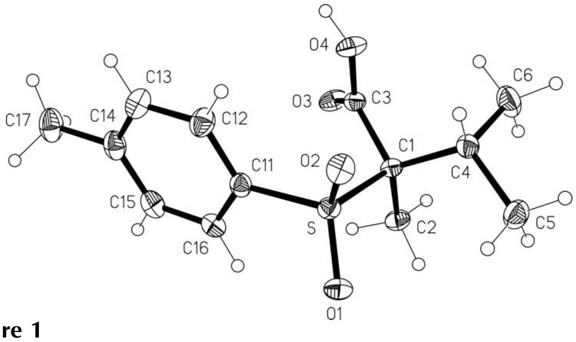


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

doi:10.1107/S1600536806017879

Experimental

Compound (I) was prepared by methylation of 2-methylpropyl-*p*-toluenesulfone (Yi *et al.*, 2004) according to the literature method of Ratajczak & Polański (1991) and subsequent carboxylation according to Cram & Ratajczak, (1971). Single crystals were obtained by slow evaporation in air of a solution in methanol (m.p. 417–419 K). Consistent analytical data were obtained.

Crystal data

$C_{13}H_{18}O_4S$
M_r = 270.33
Orthorhombic, *Pbca*
a = 10.6198 (11) Å
b = 12.4497 (12) Å
c = 20.141 (2) Å
V = 2662.9 (5) Å^3
Z = 8
D_x = 1.349 Mg m^{-3}
Mo *K*α radiation
μ = 0.25 mm^{-1}
T = 133 (2) K
Block, colourless
0.27 × 0.24 × 0.22 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
36385 measured reflections
4064 independent reflections
3280 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.103
$\theta_{\rm max}$ = 30.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.105$
S = 1.02
4064 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 0.5795P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max}$ = 0.51 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S—O1	1.4427 (9)	S—C11	1.7661 (12)
S—O2	1.4443 (9)	S—C1	1.8557 (11)
O1—S—O2	118.86 (5)	O1—S—C1	107.31 (5)
O1—S—C11	108.59 (6)	O2—S—C1	108.77 (5)
O2—S—C11	108.19 (5)	C11—S—C1	104.16 (5)
C11—S—C1—C3	33.23 (8)	O2—S—C11—C12	18.67 (11)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H04···O3$^{\rm i}$	0.90 (2)	1.70 (2)	2.6051 (12)	178 (2)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

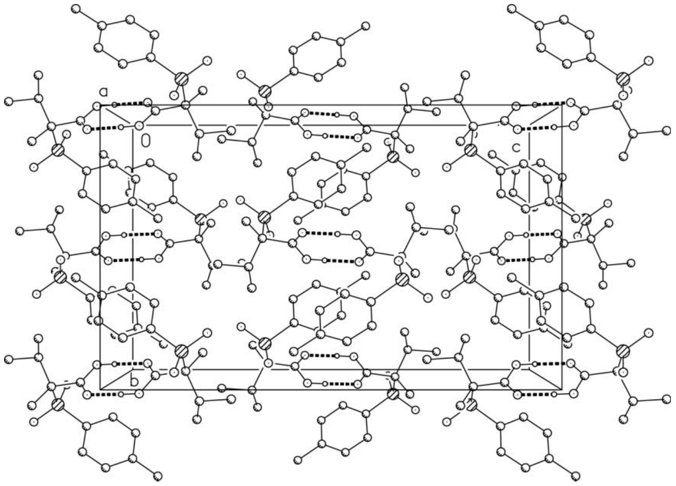


Figure 2
Packing of the title compound, viewed parallel to the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms have been omitted, except for the acidic H atom.

The acidic H atom was refined freely. Methyl H atoms were identified in difference syntheses, idealized and then refined using a rigid methyl group (C—H = 0.98 Å and H—C—H = 109.5°) allowed to rotate but not tip. Other H atoms were included using a riding model, with C—H = 0.95 (aromatic) and 1.00 Å (sp^3-CH). $U_{\rm iso}$(H) values were fixed at $1.2U_{\rm eq}$(C) of the parent C atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

References

Anulewicz, A., Luboradzki, R., Suwińska, K. & Zakrzewski, A. (1990). *Acta Cryst.* C**46**, 907–909.
Bruker (1998). *SMART* (Version 5.0), *SAINT* (Version 4.0) and *SADABS* (Version 2.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Cram, D. J. & Ratajczak, A. (1971). US Patent No. 3 598 868.
Doleżych, B., Łaszczyca, P., Polański, J., Ratajczak, A., Mekail, A. & Orchel, A. (1999). *Acta Pol. Pharm.* 56 (Suppl.), 45–47.
Polański, J., Ratajczak, A., Gastaiger, J., Gałecki, Z. & Gałecka, E. (1997). *J. Mol. Struct.* **407**, 71–80.
Ratajczak, A. & Polański, J. (1991). *Pol. J. Chem.* **65**, 1963–1971.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. Univ. of Göttingen, Germany.
Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Yi, H., Zen-Chu, C., Zhang-Gao, L. & Qin-Guo, Z. (2004). *Synth. Commun.* **34**, 4031–4036.