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N-[4-(Ethoxycarbonyl)phenyl]-*p*-tolylsulfonamide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.129
Data-to-parameter ratio = 18.2

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

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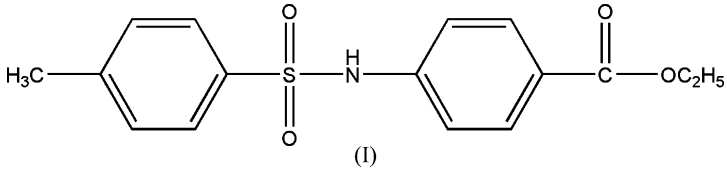
In the molecule of the title compound {systematic name: ethyl 4-[(4-methylphenyl)sulfonylamino]benzoate}, $C_{16}H_{17}NO_4S$, the dihedral angle between the two benzene rings is 93.4 (1)°. The C—N—S—C torsion angle in the central part of the molecule is 71.09 (16)°. The crystal structure is stabilized by intermolecular N—H···O interactions.

Comment

The title compound, (I), is the result of an important synthesis for the preparation of a variety of sulfonamide drugs which, for example, possess anti-neoplastic activities (Kelley & Bigham, 1989; Bigham *et al.*, 1992; Piper *et al.*, 1982).



(I)

The molecular structure of the title compound (I) is illustrated in Fig.1 and selected bond distances and anges are given in Table 1. The dihedral angle between the two benzene rings of the carbethoxyphenyl moiety and the tolylsulfonamide moiety is 93.4 (1)°. The torsion angle C5—S1—N1—C8 in the central part of the molecule is equal to 71.09 (16)°.

In the crystal structure, the molecules stack along the *b* axis and adjacent molecules are linked by intermolecular N—H···O hydrogen bonds (Fig. 2 and Table 2).

Experimental

Compound (I) was prepared, according to the method of Bekar *et al.* (1964). To a stirred solution of 16.5 g (0.1 mol) of ethyl *p*-amino-benzoate in 50 ml. of pyridine, 21.0 g (0.11 mol) of *p*-tolylsulfonyl chloride was added. The mixture was heated on a steam-bath for 30 minutes protected from moisture. Dilution of the hot reaction mixture with 150 mol of 50% ethanol and chilling gave 30.0 g (94%) of the product. Recrystallization from ethanol gave white crystals, m.p. 478–480 K.

Crystal data

$C_{16}H_{17}NO_4S$
M_r = 319.37
Monoclinic, $P2_1/n$
a = 7.8159 (16) Å
b = 8.0701 (16) Å
c = 25.165 (5) Å
β = 95.36 (3)°
V = 1580.3 (5) Å^3
Z = 4
D_x = 1.342 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15144 reflections
θ = 3.0–27.5°
μ = 0.22 mm^{-1}
T = 293 (2) K
Rod, white
0.70 × 0.63 × 0.59 mm

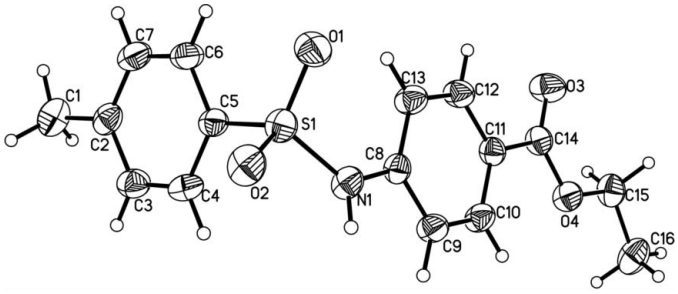


Figure 1
A view of the molecular structure of compound (I), showing the numbering scheme and displacement ellipsoids drawn at the 30% probability level.

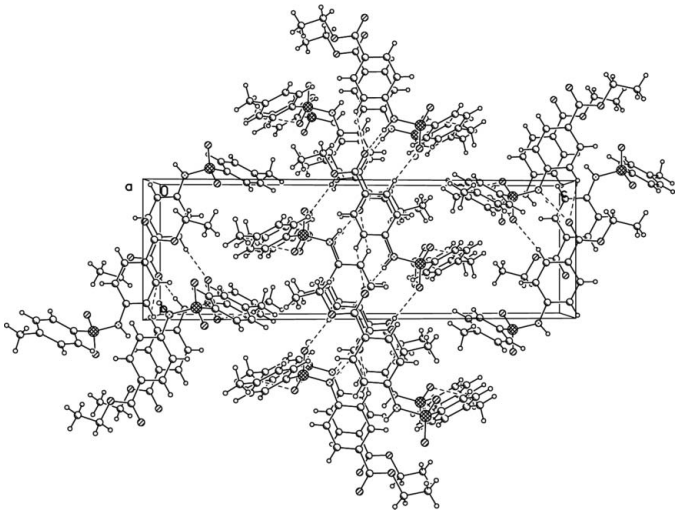


Figure 2
The crystal packing of compound (I), viewed along the *b* axis. Intermolecular N—H···O hydrgen bonds are shown as dashed lines.

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
oscillation scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.860, T_{max} = 0.880
15144 measured reflections
3624 independent reflections
2706 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.129
S = 1.08
3624 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0774P)^2 + 0.0238P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4302 (13)	N1—C8	1.4112 (18)
S1—N1	1.6219 (14)	O3—C14	1.2035 (18)
S1—C5	1.7573 (18)	C1—C2	1.499 (3)
O2—S1—O1	119.32 (8)	C3—C2—C1	120.95 (17)
O1—S1—N1	109.42 (8)	C4—C5—S1	119.77 (12)
C8—N1—S1	127.74 (11)	O3—C14—C11	124.28 (15)
C5—S1—N1—C8	71.09 (16)	S1—N1—C8—C13	16.4 (2)
C1—C2—C3—C4	−178.41 (16)	C10—C11—C14—O4	−2.4 (2)
O2—S1—C5—C6	115.66 (13)	C14—O4—C15—C16	−178.08 (15)
N1—S1—C5—C4	51.88 (13)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.86	2.09	2.844 (2)	147

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

All H atoms were positioned geometrically and refined using a riding model, with C—H 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(C) [U_{iso}(H) = 1.5U_{eq}(C) for Me H atoms].

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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