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N,*N*-Bis(2-cyanoethyl)-*p*-toluenesulfonamide

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.080
Data-to-parameter ratio = 15.6

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

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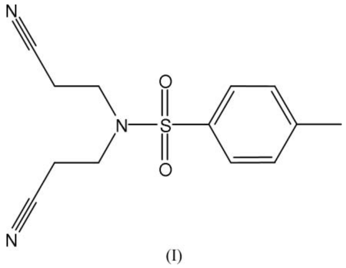
In the title compound, $C_{13}H_{15}N_3O_2S$, the geometric parameters are in the normal ranges. The amide N atom is coordinated in an almost trigonal-planar fashion.

Comment

The title compound, (I), is a derivative of *p*-toluenesulfonamide, which is itself used in pharmaceuticals and dyestuffs. Derivatives of *p*-toluenesulfonamide, *viz*. chloramine-T and dichloramine-T, are used as strong oxidizing agents (Yathirajan *et al.*, 1980). In view of the importance of (I), its crystal structure is reported here.



(I)

A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.7; *MOGUL* Version 1.0.1; Allen, 2002).

The coordination of the amide N atom is almost trigonal planar. The sum of the bond angles at N is 354.68°. Furthermore, it is interesting to note that both ethylene links adopt a *gauche* conformation (Table 1).

Experimental

A mixture of *p*-toluenesulfonamide (1.72 g, 10 mmol), acrylonitrile (1.35 g, 25 mmol) and tetrabutylammonium bromide (0.32 g, 1 mmol) in tetrahydrofuran (5 ml) was treated with KOH powder (1.12 g, 20 mmol) at room temperature for 10 h with constant stirring. The tetrahydrofuran solvent was evaporated and the residue was dissolved in diethyl ether (5 ml) and washed thoroughly with water to remove the unreacted sulfonamide. Diethyl ether was then removed by slow evaporation (yield 70%, m.p. 374 K). The product, (I), was recrystallized from methanol.

Crystal data

$C_{13}H_{15}N_3O_2S$
M_r = 277.34
Triclinic, $P\bar{1}$
a = 5.7994 (7) Å
b = 9.1494 (12) Å
c = 12.8573 (15) Å
α = 92.152 (10)°
β = 94.282 (10)°
γ = 90.551 (10)°
V = 679.78 (14) Å^3
Z = 2
D_x = 1.355 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7309 reflections
θ = 3.9–26.3°
μ = 0.24 mm^{-1}
T = 173 (2) K
Block, colourless
0.40 × 0.24 × 0.14 mm

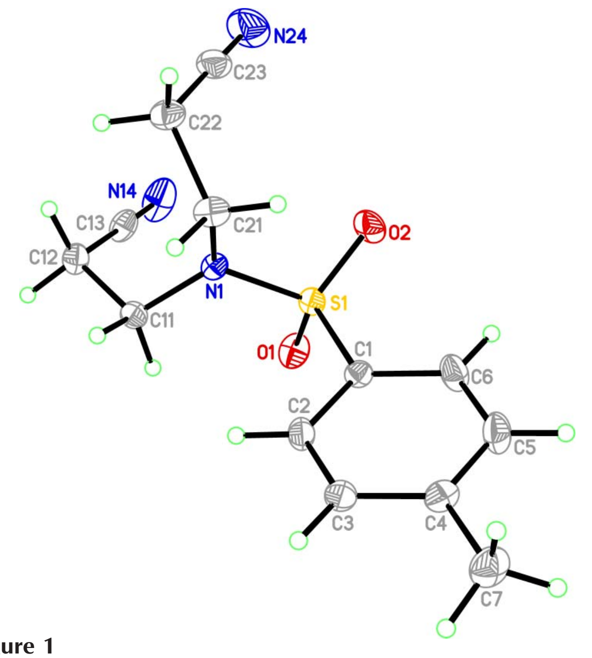


Figure 1
A perspective view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{\min} = 0.910$, $T_{\max} = 0.969$
7309 measured reflections
2696 independent reflections
2389 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.029$
$\theta_{\max} = 26.3^\circ$
$h = -6 \rightarrow 7$
$k = -11 \rightarrow 11$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.080$
$S = 1.04$
2696 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.3573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.37$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.4337 (11)	N1—C11	1.4756 (18)
S1—O1	1.4574 (11)	N1—C21	1.4909 (18)
S1—N1	1.6341 (12)	C13—N14	1.147 (2)
S1—C1	1.7885 (14)	C23—N24	1.141 (2)
O2—S1—O1	118.84 (7)	N1—S1—C1	106.15 (6)
O2—S1—N1	107.96 (7)	C11—N1—C21	118.92 (11)
O1—S1—N1	106.35 (6)	C11—N1—S1	119.46 (9)
O2—S1—C1	106.26 (7)	C21—N1—S1	116.30 (9)
O1—S1—C1	110.61 (7)		
N1—C11—C12—C13	55.19 (17)	N1—C21—C22—C23	−56.53 (17)

All H atoms were located in a difference map but were subsequently positioned geometrically, with C—H = 0.95, 0.98 and 0.99 Å for aromatic H, methyl H and methylene H atoms, respectively, and refined using a riding model, with $U_{\rm iso}$(H) set to 1.2 times $U_{\rm eq}$ of the parent atom, or 1.5 times $U_{\rm eq}$ for methyl groups. In addition, the torsion angle about the methyl group was refined.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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