organic papers

N,N′-Ethylenebis(p-toluenesulfonamide)

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.036
wR factor = 0.110
Data-to-parameter ratio = 15.3

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

The title compound, $C_{16}H_{20}N_2O_4S_2$, crystallizes in a 'partially extended' conformation, with crystallographically imposed centrosymmetry and intermolecular N—H···O hydrogen bonding.

Comment

N,N′-Ethylenebis(p-toluenesulfonamide), (I), was prepared for use as a starting material in the synthesis of N-substituted alkylaminopolycarboxylate compounds, which were sought for development as potential biomedical diagnostics. The molecule crystallizes in a 'partially extended' conformation in which the tosyl group folds back towards the diaminoethane unit. The molecule possesses crystallographically imposed centrosymmetry and is associated in the crystal via intermolecular N—H···O hydrogen bonds.

(I)
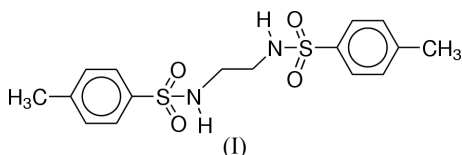
Experimental

The title compound was prepared according to the procedure of Vogel (1989) and recrystallized from methanol. Analysis calculated for $C_{16}H_{20}N_2O_4S_2$: C 51.9, H 5.4, N, 7.6%; found: C 51.9, H 5.8; N, 7.5%.

Crystal data

$C_{16}H_{20}N_2O_4S_2$	$D_m = 1.4$ Mg m^{-3}
$M_r = 368.46$	D_m measured by flotation
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 5.8070$ (12) Å	Cell parameters from 25
$b = 8.0970$ (16) Å	reflections
$c = 18.513$ (4) Å	$\theta = 14.2$–$18.7°$
$\beta = 98.35$ (3)°	$\mu = 0.33$ mm^{-1}
$V = 861.2$ (3) Å^3	$T = 293$ (2) K
$Z = 2$	Column, colourless
$D_x = 1.421$ Mg m^{-3}	$0.40 \times 0.36 \times 0.23$ mm

Data collection

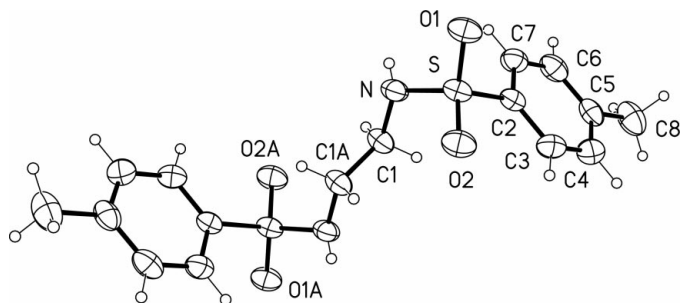|	
Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.025$
	$\theta_{max} = 26.0°$
$\theta/2\theta$ scans	$h = -7 \rightarrow 7$
Absorption correction: empirical via ψ scans (North et al., 1968)	$k = 0 \rightarrow 9$
	$l = -22 \rightarrow 22$
$T_{min} = 0.817$, $T_{max} = 0.829$	2 standard reflections
3356 measured reflections	frequency: 120 min
1682 independent reflections	intensity decay: 4.3%
1283 reflections with $I > 2\sigma(I)$	

Figure 1
Perspective view of the title molecule. The pairs of atoms C1/C1*A*, O1/O1*A* etc are related by the crystallographic inversion centre.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.110$
$S = 1.03$
1682 reflections
110 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0683P)^2 + 0.1576P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.008$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N—H1N$\cdots$O1^i	0.86	2.34	2.971 (2)	131

Symmetry code: (i) $1 - x, 2 - y, -z$.

H atoms were refined with fixed individual displacement parameters [U(H) = 1.2 U_{eq}(C), U(H) = 1.3 U_{eq}(C$_{methyl}$) or U(H) = 1.2 U_{eq}(N)] using a riding model with N—H = 0.86, C—H(aromatic) = 0.93, C—H(methylene) = 0.97 or C—H(methyl) = 0.96 Å. The methyl group was allowed to rotate about its local threefold axis.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms & Wocadlo, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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