

Tritosylate of diethanolamine

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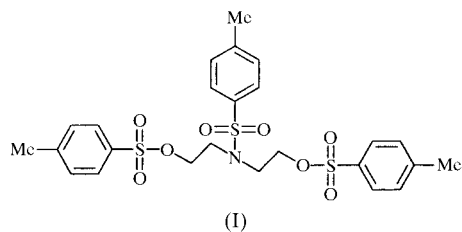
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The crystal structure of the tritosylated diethanolamine *N,N*-bis(tosyloxyethyl)-*p*-toluenesulfonamide, $C_{25}H_{29}NO_8S_3$, has been determined. The conformation of the molecule is such that the tosyl groups are as far apart as possible. The molecules are interconnected by $C-H\cdots O$ hydrogen bonds and form columns in the crystal structure.

Comment

The development of mixed-donor cyclic ligands has received considerable attention due to their ability to discriminate between metal ions (Adam *et al.*, 1995). Among tridentate cyclic ligands, [9]aneNS₂ is interesting since it has both hard N-donor and soft S-donor atoms (Blake *et al.*, 1998). One strategy for the synthesis of macrocycles based on an NS₂-donor set involves the title compound, (I), as an important precursor, followed by macrocyclization with the corresponding dithiol and detosylation (Blake *et al.*, 1998). During our search for new macrocycles containing NS₂X_{*n*} (*X* = O or S, *n* = 0–4) mixed-donor atoms, we have determined the crystal structure of this compound.



The backbone of the molecule (Fig. 1) is fully stretched and the torsion angles in both $-NCH_2CH_2O-$ units correspond to the *anti* conformation [179.3 (2) and 167.7 (2) $^\circ$]. The three tosyl groups are separated from one another as much as possible, presumably to minimize steric repulsion. The interplanar angle between the two *O*-tosyl aromatic rings is 76.7 (1) $^\circ$, and those between the *O*-tosyl and *N*-tosyl aromatic rings are 53.3 (1) and 27.9 (1) $^\circ$.

Although H atoms have been included in their idealized positions, some aromatic and methylene H atoms are situated close to the sulfonyl O atoms of the *O*-tosyl groups, providing evidence for $C-H\cdots O$ hydrogen bonds (Taylor & Kennard,

1982). As illustrated in Fig. 2, the molecules are interconnected by hydrogen bonds in such a way that they form columns parallel to the crystallographic *c* axis. Aromatic and aliphatic $C-H\cdots O$ hydrogen bonds alternate in the column; their parameters are given in Table 1.

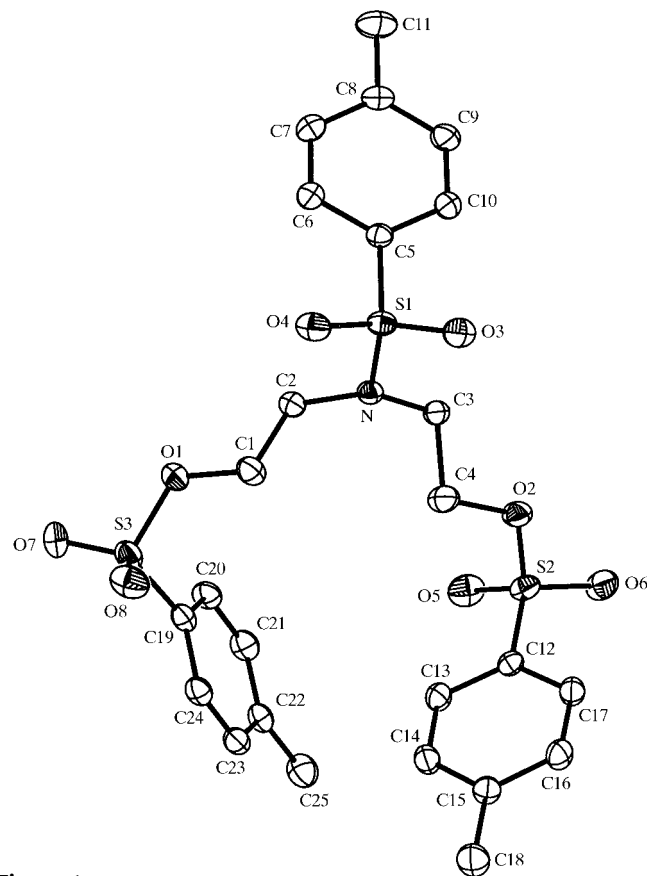


Figure 1

Perspective view of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at 30% probability level and H atoms have been omitted for clarity.

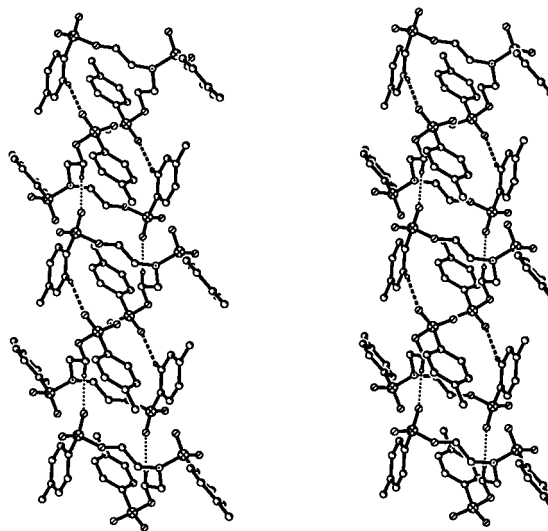


Figure 2

Stereoview of one molecular column stabilized by alternating aromatic (dashed lines) and aliphatic (dotted lines) $C-H\cdots O$ hydrogen bonds. Non-hydrogen-bonded H atoms have been omitted for clarity.

Experimental

Compound (I) was prepared by the reaction of diethanolamine and tosyl chloride according to the published procedure of Searle & Geue (1984). Single crystals suitable for X-ray crystallographic analysis were prepared by vapor diffusion of diethyl ether into an acetonitrile solution of compound (I) at room temperature.

Crystal data

$C_{25}H_{29}NO_8S_3$	$D_x = 1.394 \text{ Mg m}^{-3}$
$M_r = 567.67$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 17590 reflections
$a = 11.3887 (10) \text{ \AA}$	$\theta = 1.85\text{--}28.39^\circ$
$b = 22.0576 (19) \text{ \AA}$	$\mu = 0.322 \text{ mm}^{-1}$
$c = 11.7301 (10) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 113.3300 (10)^\circ$	Plate, colourless
$V = 2705.8 (4) \text{ \AA}^3$	$0.40 \times 0.30 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

CCD area-detector diffractometer	$R_{\text{int}} = 0.047$
φ and ω scans	$\theta_{\text{max}} = 28.39^\circ$
17590 measured reflections	$h = -15 \rightarrow 13$
6572 independent reflections	$k = -29 \rightarrow 23$
3614 reflections with $I > 2\sigma(I)$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0680P)^2]$
$wR(F^2) = 0.136$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.003$	$(\Delta/\sigma)_{\text{max}} < 0.001$
6572 reflections	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
334 parameters	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$

H atoms were added at calculated positions (C—H = 0.96 Å) and were refined using a riding model. They were assigned isotropic displacement parameters equal to 1.2 times the equivalent isotropic displacement parameter of their parent atoms.

Table 1

Hydrogen-bonding geometry (Å, °).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
C4—H4A \cdots O8 ⁱ	0.96	2.50	3.451 (4)	174
C20—H20 \cdots O6 ⁱⁱ	0.96	2.53	3.376 (3)	148

Symmetry codes: (i) $-x, 1 - y, 1 - z$; (ii) $-x, 1 - y, 2 - z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SHELXTL* (Siemens, 1996); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1127). Services for accessing these data are described at the back of the journal.

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