

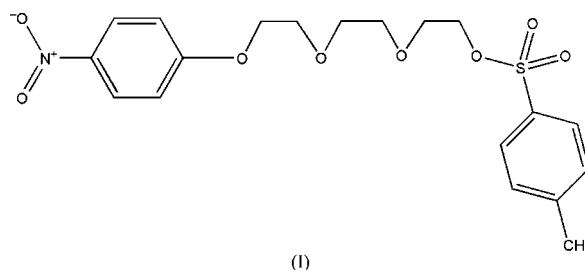
2-{2-[2-(4-Nitrophenoxy)ethoxy]ethoxy}ethyl
toluene-4-sulfonateDian-Shun Guo,* Xin-Yan Zhang,
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Key indicators

Single-crystal X-ray study
 $T = 123$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.034
 wR factor = 0.088
Data-to-parameter ratio = 10.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{19}\text{H}_{23}\text{NO}_8\text{S}$, an important asymmetric alkylating agent, contains a polyether linkage and two substituted benzene rings, which make a dihedral angle of $79.95(7)^\circ$. In the crystal structure, there are $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.Received 19 July 2006
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Comment

Acyclic polyethers bearing functionalized terminal groups have received considerable attention because of their binding abilities with alkali metal ions (Hayashita & Takagi, 1996). In particular, the tosylates of oligoethylene glycols are significant alkylating agents for constructing receptors such as crown ethers and related structures in supramolecular chemistry (Saadioui *et al.*, 1997). We report here the structure of an asymmetric derivative, (I), of triethylene glycol, in which nitrophenyl and tosyl groups are attached to the ends of the polyether chain.The title compound, (I), contains a triethylene glycol bridge and two substituted benzene rings (Fig. 1). The nitro-substituted benzene ring and tosyl group are linked to the polyether chain; the benzene rings form a dihedral angle of $79.95(7)^\circ$. The whole molecule looks like a hook or a spoon. In the crystal structure of (I), there are intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1 and Fig. 2). Other dimensions are as expected.

Experimental

To a mixture of 2-[2-[2-(4-nitrophenoxy)ethoxy]ethoxy]ethanol (2.17 g, 8 mmol) and sodium hydroxide (0.48 g, 12 mmol) in tetrahydrofuran (THF, 10 ml) together with water (2 ml) cooled in an ice-water bath, was added dropwise a solution of *p*-toluenesulfonyl chloride (1.72 g, 9 mmol) in THF (10 ml). The resulting mixture was stirred at 273 K for 6 h under an N_2 atmosphere, and diluted with ethyl acetate (5 ml) and water (5 ml). The organic layer was separated and washed with 5% HCl, saturated NaHCO_3 and brine, and dried over anhydrous MgSO_4 . Removal of the solvent under reduced pressure gave the product (I) as a transparent oil in 95% yield. Single crystals of (I) suitable for X-ray diffraction were obtained by evaporation of a CH_2Cl_2 solution at 273 K.

Crystal data

C₁₉H₂₃NO₈S
M_r = 425.44
 Monoclinic, *Cc*
a = 11.515 (2) Å
b = 11.765 (2) Å
c = 14.883 (3) Å
 β = 97.117 (3)°
V = 2000.7 (6) Å³

Z = 4
D_x = 1.412 Mg m⁻³
 Mo *K*α radiation
 μ = 0.21 mm⁻¹
T = 123 (2) K
 Block, colourless
 0.55 × 0.43 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1999)
T_{min} = 0.883, *T_{max}* = 0.940

5051 measured reflections
 2808 independent reflections
 2739 reflections with *I* > 2σ(*I*)
R_{int} = 0.035
 θ_{max} = 25.5°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.034
wR(*F*²) = 0.088
S = 1.06
 2808 reflections
 263 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.3667P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.23 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 952 Friedel Pairs
 Flack parameter: 0.09 (7)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C5—H5...O3 ⁱ	0.95	2.45	3.271 (3)	145
C7—H7A...O8 ⁱⁱⁱ	0.99	2.35	3.261 (3)	152
C10—H10A...O6 ⁱ	0.99	2.42	3.400 (3)	173
C17—H17...O5 ⁱⁱⁱ	0.95	2.46	3.401 (3)	169
C19—H19A...O2 ^{iv}	0.98	2.59	3.567 (4)	176

Symmetry codes: (i) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (ii) $x - \frac{1}{2}, y + \frac{1}{2}, z$; (iii) $x - \frac{1}{2}, y - \frac{1}{2}, z$; (iv) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$

Methyl H atoms were placed in calculated positions, with C—H = 0.98 Å and *U_{iso}*(H) = 1.5*U_{eq}*(C). Other H atoms were placed in calculated positions with C—H = 0.95 (aromatic) and 0.99 Å (methylene), and refined in riding mode, with *U_{iso}*(H) = 1.2*U_{eq}*(C).

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

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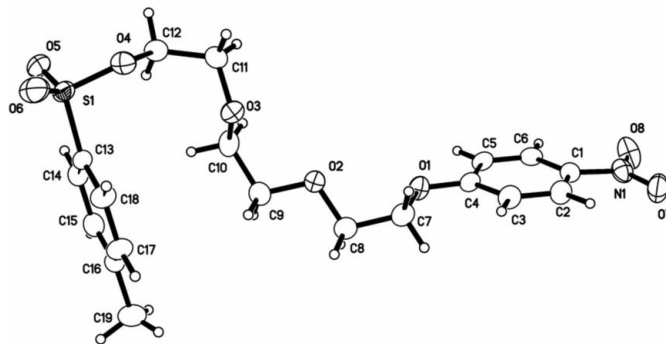


Figure 1

The molecular structure of (I). Displacement ellipsoids for non-H atoms are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

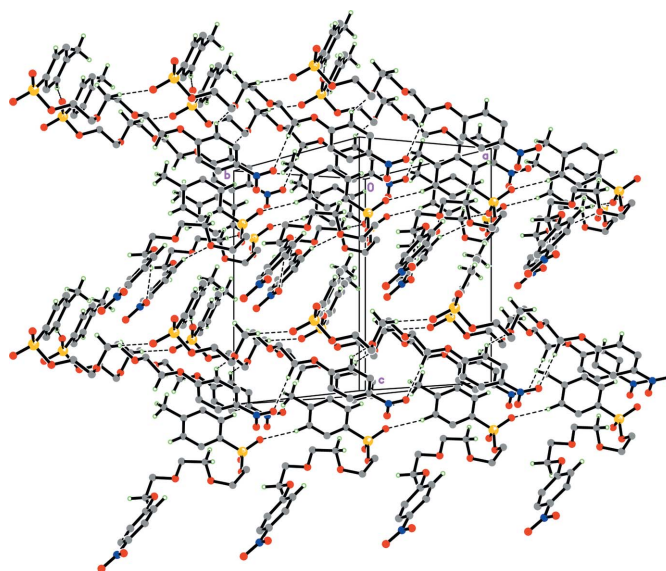


Figure 2

A view of the packing of (I), showing the C—H...O contacts (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

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