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2,4-Dinitrophenyl 4-toluenesulfonate: molecular aggregation through weak C—H···O interactions

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Key indicators

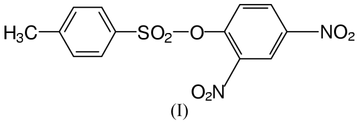
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.087
Data-to-parameter ratio = 12.8

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

The title molecule, $C_{13}H_{10}N_2O_7S$, is stabilized by weak intermolecular C—H···O interactions. The dihedral angle between the mean planes of the 4-tolyl and 2,4-dinitrophenyl rings is 19.26 (3)°. Both O atoms of the 4-nitro group form a 'fork-like' intermolecular hydrogen-bonding motif with the C—H groups of the aromatic rings. In addition, one of the O atoms of the 2-nitro group and one of the O atoms of the sulfonyl group are also involved in weak intermolecular hydrogen bonding.

Comment

p-Toluenesulfonates are used in monitoring the merging of lipids (Yachi *et al.*, 1989), studying membrane fusion during acrosome reaction (Spungin *et al.*, 1992), the development of immunoaffinity chromatography for the purification of the human coagulation factor (Tharakan *et al.*, 1992), chemical studies on viruses (Alford *et al.*, 1991), the development of technology for linking photosensitizers to model monoclonal antibodies (Jiang *et al.*, 1990) and the chemical modification of sigma subunits of the *E. coli* RNA polymerase (Narayanan & Krakow, 1983). The crystal and molecular structure determination of the title compound, (I), was undertaken because of the biological importance of its analogues.



(I)

A search of the Cambridge Structural Database (Version 5.23; Allen, 2002) revealed 16 structures [with the following refcodes: KAWDAN, FIXCAQ, NEDXUP, NEDYAW, NEDYIE, NUNCII, RASSOT, RELVUZ, SIMVUF, TCPTOS, TEBFOV, TMPDTS, TSMIPH, WOHCUR, ZZZBDA10 and MIWHIJ] that are closely related to the title compound. The S—C, S—O and S=O bond lengths in (I) (Table 1) are comparable to those found in these structures. The atoms of both of the —NO_2 groups are essentially in the plane of the phenyl ring to which they are bonded. The dihedral angle between the 2,4-dinitrophenyl and 4-tolyl rings is 19.26 (3)°.

The crystal structure of (I) is stabilized by several weak C—H···O interactions. The range for the H···O distances (2.4–2.6 Å) (see Table 2) found in (I) agree with those found for other weak C—H···O hydrogen bonds (Desiraju & Steiner, 1999). Both the O atoms of the 4-nitro group form 'fork-like' intermolecular weak hydrogen bonds (Baskar Raj *et al.*, 2003) with the C—H groups of the 2,4-dinitrophenyl and 4-tolyl

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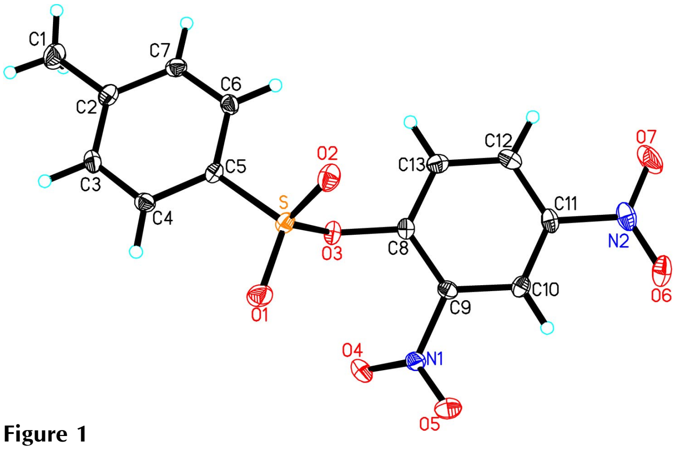

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

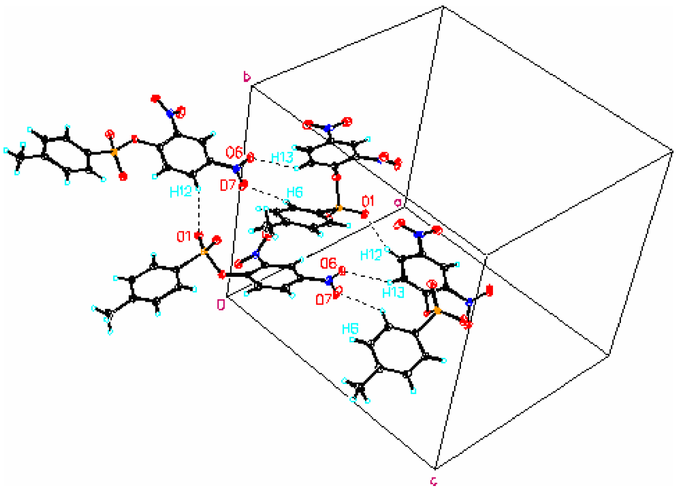

Figure 2
The packing of the molecule in the unit cell, showing the C—H···O interactions (dashed lines).

rings (Fig. 2). One of the sulfonyl O atoms (O1) acts as an acceptor to form an intermolecular weak hydrogen bond with the C—H group of a neighbouring 2,4-dinitrophenyl ring (Fig. 2). The atom O4 of the 2-nitro group acts as an acceptor to form an almost linear intermolecular weak hydrogen bond with the C—H group of a neighbouring 4-tolyl ring. The above intermolecular C—H···O interactions contribute to the molecular aggregation in the title molecule.

Experimental

Solutions of 2,4-dinitrophenol (1.8 g, 9.78 mmol) and triethylamine (1.0 g, 9.88 mmol) in acetone (25 ml) were prepared and mixed. To this mixture, a solution of 4-toluenesulfonyl chloride (1.6 g, 8.39 mmol) in acetone was added. The solution was kept overnight at 303 K and then evaporated. The residue was treated with chloroform and extracted several times with water. The chloroform layer was dried with anhydrous sodium sulfate and evaporated to yield crude 2,4-dinitrophenyl 4-toluenesulfonate (2.0 g, 70% yield). Diffraction-quality crystals were obtained by crystallizing the crude product from aqueous ethanol.

Crystal data

$C_{13}H_{10}N_2O_7S$
$M_r = 338.29$
Orthorhombic, *Pbca*
$a = 13.3935\ (7)$ Å
$b = 13.4828\ (8)$ Å
$c = 15.2291\ (8)$ Å
$V = 2750.1\ (3)$ Å^3
$Z = 8$
$D_x = 1.634$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8997 reflections
$\theta = 2.5$–$27.5°$
$\mu = 0.28$ mm^{-1}
$T = 100\ (2)$ K
Plate, colourless
0.40 × 0.30 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.897$, $T_{\max} = 0.981$
31334 measured reflections
3170 independent reflections
2867 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 27.6°$
$h = -17 \rightarrow 17$
$k = -17 \rightarrow 17$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.087$
$S = 1.05$
3170 reflections
248 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 1.1269P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.55$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S—O1	1.4204 (10)	O5—N1	1.2251 (15)
S—O2	1.4246 (10)	O6—N2	1.2220 (16)
S—O3	1.6195 (9)	O7—N2	1.2214 (16)
S—C5	1.7460 (12)	N1—C9	1.4656 (15)
O3—C8	1.3855 (14)	N2—C11	1.4693 (16)
O4—N1	1.2201 (14)		
O1—S—O2	119.79 (6)	O4—N1—O5	125.01 (11)
O1—S—O3	107.05 (5)	O4—N1—C9	118.04 (10)
O2—S—O3	107.54 (5)	O5—N1—C9	116.92 (10)
O1—S—C5	110.57 (6)	O7—N2—O6	124.33 (12)
O2—S—C5	111.34 (6)	O7—N2—C11	117.89 (12)
O3—S—C5	98.17 (5)	O6—N2—C11	117.78 (11)
C8—O3—S	120.25 (8)		
C5—S—O3—C8	141.02 (9)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O6$^{\rm i}$	0.950 (16)	2.560 (16)	3.2777 (16)	132.5 (12)
C12—H12···O1$^{\rm ii}$	0.932 (18)	2.470 (17)	3.2549 (16)	141.9 (13)
C6—H6···O7$^{\rm i}$	0.929 (16)	2.551 (16)	3.2706 (16)	134.6 (12)
C3—H3···O4$^{\rm iii}$	0.971 (18)	2.398 (18)	3.3325 (15)	161.2 (14)

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

All the H atoms were located in a difference Fourier map and their coordinates and isotropic displacement parameters were refined. The C—H bond lengths are in the range 0.88 (3)–0.99 (2) Å, the H—C—H angles for the methyl group are in the range 101 (2)–114 (2)°, and the C—C—H angles for the aromatic rings are in the range 120 (1)–124 (1)°.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

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