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2-Methyl-2-nitropropyl methanesulfonate

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Key indicators

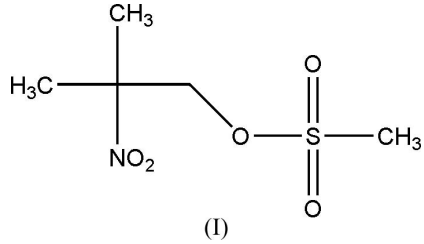
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.027
wR factor = 0.078
Data-to-parameter ratio = 8.4

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

The title compound, $C_5H_{11}NO_5S$, has been obtained by reacting 2-methyl-2-nitropropanol with methanesulfonyl chloride. The average S═O and S—O bond lengths are 1.414 (3) and 1.570 (3) Å, respectively. The molecules are linked *via* C—H···O interactions, forming a three-dimensional network.

Comment

The title compound, (I), is an intermediate in the synthesis of 2-methyl-2-nitropropylamine (Durand *et al.*, 2003). Compound (I) was prepared by reacting 2-methyl-2-nitropropanol with methanesulfonyl chloride. The present paper reports the structure analysis of (I). The molecular structure is illustrated in Fig. 1, and selected bond lengths and angles are listed in Table 1. As illustrated in Fig. 2, there are non-classical intermolecular C—H···O hydrogen bonds (Table 2), forming a three-dimensional network.



(I)

Experimental

A solution of 2-methyl-2-nitropropanol (2.4 g, 20 mmol) in toluene (30 ml) was added to a 100 ml flask and cooled with an ice bath.

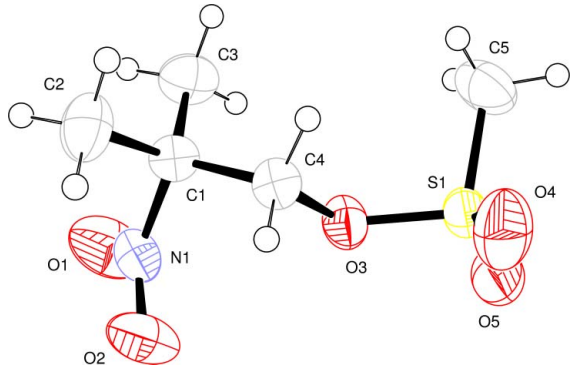


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

When the solution was cooled to 273 K, a solution of methanesulfonyl chloride (2.5 g, 22 mmol) in toluene (10 ml) was added slowly dropwise over the course of 30 min with vigorous stirring. After the addition was complete, the reaction continued at room temperature until thin-layer chromatography showed that the reaction was complete. The solution was then concentrated and cooled to give the crystalline product (yield 3.7 g, 93.4%; m.p. 323–325 K). Crystals of (I) suitable for X-ray analysis were grown from a toluene solution by slow evaporation.

Crystal data

$C_5H_{11}NO_5S$
M_r = 197.21
Orthorhombic, *F*2*dd*
a = 8.652 (2) Å
b = 19.808 (9) Å
c = 20.934 (4) Å
V = 3588 (2) Å^3
Z = 16
D_x = 1.460 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.7–13.2°
μ = 0.35 mm^{-1}
T = 295 (2) K
Prism, colourless
0.50 × 0.40 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.846, T_{max} = 0.903
909 measured reflections
861 independent reflections
796 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.1°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 23$
$l = -1 \rightarrow 24$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.078
S = 1.14
861 reflections
103 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2 + 2.7911P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00104 (14)
Absolute structure: Flack (1983), no Friedel pairs
Flack parameter: −0.02 (13)

Table 1
Selected geometric parameters (Å, °).

S1—O5	1.409 (3)	S1—C5	1.742 (4)
S1—O4	1.419 (3)	O1—N1	1.207 (4)
S1—O3	1.570 (3)	O2—N1	1.207 (4)
O5—S1—O4	119.8 (2)	O5—S1—C5	109.5 (2)
O5—S1—O3	104.37 (16)	O4—S1—C5	109.7 (2)
O4—S1—O3	108.98 (17)		
S1—O3—C4—C1	154.8 (2)	C2—C1—C4—O3	172.8 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O5^{i}	0.97	2.44	3.339 (4)	155
C4—H4B···O1^{ii}	0.97	2.57	3.499 (4)	160

Symmetry codes: (i) $x+\frac{1}{4}, -y+\frac{1}{4}, z+\frac{1}{4}$; (ii) $x-\frac{1}{4}, y+\frac{1}{4}, -z+\frac{7}{4}$.

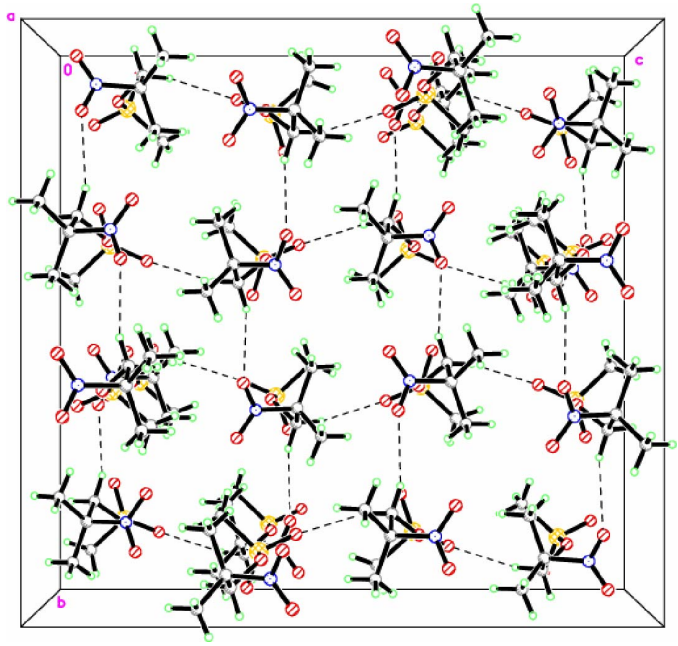


Figure 2
A packing diagram of (I), showing the intermolecular C—H···O hydrogen bonds (dashed lines), viewed along the *a* axis.

Space group *F*2*dd* is the *cab* setting of *Fdd*2. H atoms were added at calculated positions (C—H = 0.96–0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}). The unconventional space group setting was chosen by the structure solution program.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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