C10	0.2294 (2)	0.7950 (2)	−0.0056 (2)	0.0546 (7)
C11	0.5384 (2)	0.9175 (2)	−0.1636 (2)	0.0478 (6)
C12	0.4801 (2)	0.8905 (2)	−0.2785 (2)	0.0539 (7)
C13	0.4322 (2)	0.9605 (2)	−0.3644 (2)	0.0576 (7)
C14	0.4479 (3)	1.0590 (2)	−0.3339 (3)	0.0652 (8)
C15	0.5078 (3)	1.0852 (2)	−0.2211 (3)	0.0640 (8)
C16	0.5509 (2)	1.0166 (2)	−0.1350 (2)	0.0566 (7)
C17	0.3652 (3)	0.9282 (3)	−0.4874 (2)	0.0837 (11)
N1	0.5736 (2)	0.83871 (15)	−0.0851 (2)	0.0528 (6)
N2	0.7791 (2)	0.8142 (2)	0.2604 (2)	0.0512 (6)
N3	0.4117 (2)	0.67763 (14)	0.0037 (2)	0.0440 (5)
N4	0.3896 (2)	0.6036 (2)	0.1676 (2)	0.0591 (6)
N5	0.2573 (2)	0.7234 (2)	0.0720 (2)	0.0553 (6)
N6	0.1978 (2)	0.8602 (2)	−0.0699 (2)	0.0663 (7)
O1	0.55310 (14)	0.63347 (11)	−0.08854 (13)	0.0460 (5)
O2	0.59174 (15)	0.56223 (12)	0.10331 (13)	0.0489 (4)
S1	0.54599 (5)	0.64297 (4)	0.02414 (5)	0.0403 (2)

Table 2. *Selected geometric parameters* (Å, °)

C1—N1	1.359 (3)	C10—N6	1.161 (4)
C1—C2	1.400 (3)	C10—N5	1.326 (4)
C1—C5	1.423 (3)	C11—C16	1.386 (3)
C2—C3	1.359 (3)	C11—C12	1.395 (3)
C3—N2	1.337 (3)	C11—N1	1.407 (3)
C4—N2	1.346 (3)	C12—C13	1.391 (4)
C4—C5	1.360 (3)	C13—C14	1.384 (4)
C5—S1	1.783 (2)	C13—C17	1.513 (4)
C6—N4	1.322 (3)	C14—C15	1.373 (4)
C6—N5	1.334 (3)	C15—C16	1.372 (4)
C6—N3	1.375 (3)	N3—S1	1.576 (2)
C7—N4	1.476 (3)	O1—S1	1.440 (2)
C7—C9	1.513 (4)	O2—S1	1.442 (2)
C7—C8	1.509 (4)		
N1—C1—C2	124.5 (2)	C13—C12—C11	121.6 (3)
N1—C1—C5	118.8 (2)	C14—C13—C12	118.1 (3)
C2—C1—C5	116.7 (2)	C14—C13—C17	121.8 (3)
C3—C2—C1	120.3 (2)	C12—C13—C17	120.0 (3)
N2—C3—C2	121.4 (2)	C15—C14—C13	120.0 (3)
N2—C4—C5	121.1 (2)	C16—C15—C14	122.2 (3)
C4—C5—C1	119.8 (2)	C15—C16—C11	118.9 (3)
C4—C5—S1	117.6 (2)	C1—N1—C11	133.1 (2)
C1—C5—S1	122.6 (2)	C4—N2—C3	120.5 (2)
N4—C6—N5	117.3 (2)	C6—N3—S1	124.8 (2)
N4—C6—N3	122.8 (2)	C6—N4—C7	125.0 (2)
N5—C6—N3	119.8 (2)	C10—N5—C6	117.5 (2)
N4—C7—C9	107.5 (2)	O1—S1—O2	117.37 (10)
N4—C7—C8	111.2 (3)	O1—S1—N3	105.85 (10)
C9—C7—C8	112.1 (3)	O2—S1—N3	116.30 (10)
N6—C10—N5	174.4 (3)	O1—S1—C5	105.51 (10)
C16—C11—C12	119.1 (2)	O2—S1—C5	105.00 (10)
C16—C11—N1	125.6 (2)	N3—S1—C5	105.71 (10)
C12—C11—N1	115.2 (2)		
N1—C1—C2—C3	177.5 (2)	C8—C7—N4—C6	−74.0 (4)
N1—C1—C5—S1	−1.6 (3)	N6—C10—N5—C6	−171.9 (29)
N1—C11—C12—C13	175.1 (2)	N4—C6—N5—C10	173.3 (2)
C2—C1—N1—C11	−15.2 (4)	N3—C6—N5—C10	−9.1 (4)
C12—C11—N1—C1	170.1 (3)	C6—N3—S1—O1	159.9 (2)
N4—C6—N3—S1	−21.8 (3)	C6—N3—S1—O2	27.6 (2)
N5—C6—N3—S1	160.8 (2)	C6—N3—S1—C5	−88.4 (2)
N5—C6—N4—C7	−1.7 (4)	C1—C5—S1—O1	41.3 (2)
N3—C6—N4—C7	−179.2 (2)	C1—C5—S1—O2	165.9 (2)
C9—C7—N4—C6	162.9 (3)	C1—C5—S1—N3	−70.6 (2)

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTO* (Motherwell, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including bond distances and angles involving H atoms, and torsion angles have been deposited with the IUCr (Reference: PA1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Nimesulide

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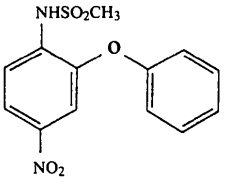
Abstract

4-Nitro-2-phenoxymethanesulfonanilide, $C_{13}H_{12}N_2O_5S$, is an anti-inflammatory drug. The molecular conformation is stabilized by an intramolecular N—H···O hydrogen bond. The angle between the two phenyl rings

is 74.7°. The cohesion of the crystal is the result of NH···O intermolecular hydrogen bonds and van der Waals interactions.

Comment

Nimesulide, (I), has analgesic, anti-inflammatory and antipyretic properties. It is an inhibitor of prostaglandin synthetase and of platelet aggregation (Moore & Harrington, 1974; Swingle & Moore, 1984; Ward & Brodgen, 1988). In the crystal structure the angle between the O5–phenyl least-squares planes is 74.69 (8)°.



The equations of the planes and the deviations of atomic positions from them have been deposited. N1—H1 is involved in two hydrogen bonds: an intramolecular hydrogen bond, N1—H1···O5 [N1···O5 2.583 (3), H1···O5 2.172 (2) Å, N1—H1···O5 104.1 (2)°], and an intermolecular hydrogen bond, N1—H1···O3^{i} [symmetry code: (i) x, 2 − y, 0.5 + z; N1···O3^{i} 3.093 (3), H1···O3^{i} 2.210 (2) Å, N1—H1···O3^{i} 151.2 (2)°].

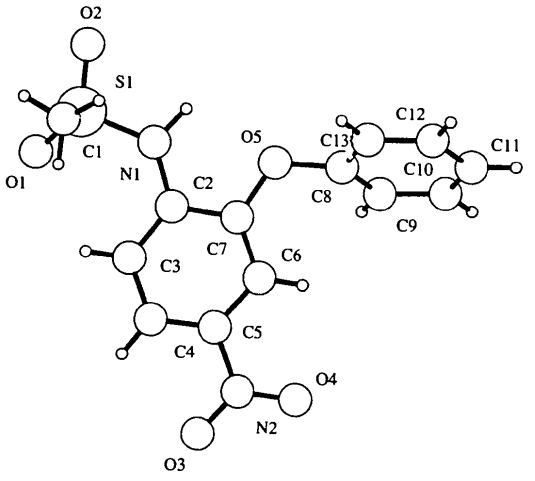


Fig. 1. Molecular structure with atom-labelling scheme.

Experimental

Crystal data

$C_{13}H_{12}N_2O_5S$
M_r = 308.31
Monoclinic
$C2/c$
a = 33.657 (3) Å
b = 5.1305 (3) Å
c = 16.0816 (10) Å
β = 92.368 (8)°
V = 2774.5 (3) Å^3
Z = 8
D_x = 1.476 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 36 reflections
θ = 28.31–32.35°
μ = 2.310 mm^{-1}
T = 293 (2) K
Prism
0.30 × 0.30 × 0.27 mm
Colourless
Crystal source: Therabel Research, Bruxelles

Data collection

Stoe Siemens AED four-circle diffractometer
ω-scans
Absorption correction: semi-empirical
T_{min} = 0.640, T_{max} = 0.746
1999 measured reflections
1910 independent reflections
1355 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0201
θ_{max} = 57.52°
h = −36 → 36
k = 0 → 5
l = −17 → 0
2 standard reflections monitored every 100 reflections
intensity decay: 3.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0401
$wR(F^2)$ = 0.1146
S = 1.248
1908 reflections
194 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 0.0512P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.008
$\Delta\rho_{max}$ = 0.221 e Å^{-3}
$\Delta\rho_{min}$ = −0.211 e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001x \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient: x = 0.0016 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	0.48964 (10)	0.6862 (7)	0.1404 (2)	0.0619 (9)
C2	0.40549 (8)	0.7137 (6)	0.0321 (2)	0.0464 (7)
C3	0.42703 (9)	0.7117 (6)	−0.0388 (2)	0.0563 (8)
C4	0.41775 (9)	0.8823 (7)	−0.1037 (2)	0.0582 (8)
C5	0.38691 (9)	1.0515 (6)	−0.0955 (2)	0.0519 (8)
C6	0.36387 (9)	1.0571 (7)	−0.0264 (2)	0.0576 (8)
C7	0.37342 (8)	0.8849 (6)	0.0370 (2)	0.0531 (8)
C8	0.32013 (9)	1.0199 (7)	0.1220 (2)	0.0580 (9)
C9	0.28473 (11)	0.9546 (8)	0.0824 (2)	0.0711 (10)
C10	0.25139 (10)	1.0975 (8)	0.0990 (2)	0.0769 (11)
C11	0.25387 (11)	1.2985 (8)	0.1542 (2)	0.0752 (10)
C12	0.28944 (12)	1.3575 (8)	0.1935 (2)	0.0802 (11)
C13	0.32284 (10)	1.2184 (7)	0.1784 (2)	0.0686 (10)
N1	0.41351 (7)	0.5527 (5)	0.10176 (14)	0.0549 (7)
N2	0.37749 (9)	1.2385 (6)	−0.1622 (2)	0.0642 (7)
O1	0.46955 (6)	0.2742 (4)	0.05712 (13)	0.0620 (6)
O2	0.45181 (7)	0.2970 (5)	0.20453 (13)	0.0689 (7)
O3	0.40020 (8)	1.2561 (6)	−0.21917 (14)	0.0851 (8)
O4	0.34810 (8)	1.3751 (7)	−0.1574 (2)	0.1036 (11)
O5	0.35423 (7)	0.8698 (5)	0.11083 (13)	0.0793 (8)
S1	0.45701 (2)	0.42371 (14)	0.12648 (4)	0.0511 (3)

Table 2. *Selected geometric parameters* (Å, °)

C1—S1	1.747 (3)	C8—C9	1.369 (5)
C2—C3	1.377 (4)	C8—O5	1.400 (4)
C2—C7	1.396 (4)	C9—C10	1.376 (5)
C2—N1	1.409 (4)	C10—C11	1.360 (5)
C3—C4	1.387 (4)	C11—C12	1.364 (5)
C4—C5	1.364 (4)	C12—C13	1.362 (5)
C5—C6	1.382 (4)	N1—S1	1.640 (2)
C5—N2	1.464 (4)	N2—O4	1.217 (4)
C6—C7	1.377 (4)	N2—O3	1.221 (3)
C7—O5	1.377 (3)	O1—S1	1.431 (2)
C8—C13	1.364 (5)	O2—S1	1.431 (2)
C3—C2—C7	119.1 (3)	C11—C10—C9	120.1 (3)
C3—C2—N1	124.3 (3)	C10—C11—C12	120.0 (3)
C7—C2—N1	116.6 (2)	C13—C12—C11	121.1 (4)
C2—C3—C4	120.6 (3)	C12—C13—C8	118.5 (3)
C5—C4—C3	118.6 (3)	C2—N1—S1	124.7 (2)
C4—C5—C6	123.1 (3)	O4—N2—O3	122.7 (3)
C4—C5—N2	119.3 (3)	O4—N2—C5	118.9 (3)
C6—C5—N2	117.6 (3)	O3—N2—C5	118.4 (3)
C7—C6—C5	117.4 (3)	C7—O5—C8	119.7 (2)
C6—C7—O5	124.8 (3)	O2—S1—O1	119.40 (14)
C6—C7—C2	121.3 (3)	O2—S1—N1	104.82 (12)
O5—C7—C2	113.8 (3)	O1—S1—N1	108.43 (13)
C13—C8—C9	121.6 (3)	O2—S1—C1	109.7 (2)
C13—C8—O5	117.6 (3)	O1—S1—C1	108.01 (14)
C9—C8—O5	120.5 (3)	N1—S1—C1	105.7 (2)
C8—C9—C10	118.7 (3)		
C7—C2—N1—S1	−155.6 (2)	C6—C7—O5—C8	6.2 (5)
C6—C5—N2—O4	−6.0 (4)	C13—C8—O5—C7	−111.4 (4)
C6—C5—N2—O3	171.9 (3)	C2—N1—S1—O2	175.1 (3)

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: PA1137). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Methyl 3-[2-(*tert*-Butylthio)phenyl]-propenoate

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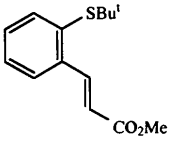
Abstract

The effects of steric strain in methyl 3-[2-(*tert*-butylthio)phenyl]propenoate ($C_{14}H_{18}O_2S$) are seen in the distribution of C—C bond lengths within the phenyl ring and in the placement of the *tert*-butyl substituent.

Comment

The title compound, (1), represents a simple example of a sterically congested *ortho*-substituted cinnamate ester. *Ortho*-substitution does not appear to affect the bond lengths in the acrylate chain, which are very similar to those seen in remotely substituted cinnamates (Talberg, 1978; Nakanishi & Sasada, 1978). Similarly, the C—S bonds are only slightly different from their normal values (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). In contrast, the geometry of the benzene ring is substantially distorted, with the bond between the two substituted C atoms [C1—C2 1.409 (2) Å] somewhat longer than the other *ortho* bonds [C2—C3 1.398 (2), C1—C6 1.401 (2) Å]. These three bonds are significantly longer than the other three ring C—C distances [1.375 (2), 1.378 (3), 1.385 (3) Å].



(1)

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