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N-(2,3-Dimethylphenyl)methanesulfonamide

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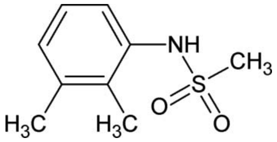
Key indicators: single-crystal X-ray study; T = 301 K; mean σ(C–C) = 0.005 Å; R factor = 0.054; wR factor = 0.130; data-to-parameter ratio = 16.6.

In the structure of the title compound (23DMPMSA), $C_9H_{13}NO_2S$, the conformation of the N—H bond lies between *syn* and *anti* to the methyl substituents at the *ortho* and *meta* positions, in contrast to the *syn* and *anti* conformations observed, respectively, for *N*-(2-methylphenyl)methanesulfonamide (2MPMSA) and *N*-(3-methylphenyl)methanesulfonamide (3MPMSA). The bond parameters in *N*-phenylmethanesulfonamide (PMSA), 2MPMSA, 3MPMSA and 23DMPMSA are similar except for some differences in the torsion angles. The H atom on N is on one side of the plane of the benzene ring, while the methanesulfonyl group is on the opposite side of the plane, as in PMSA, 2MPMSA and 3MPMSA. The amide H atom is thus available to a receptor molecule during its biological activity. The molecules in the title compound are packed into chains through N—H···O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2007*a*,*b*,*c*,*d*,*e*,*f*,*g*); Jayalakshmi & Gowda (2004); Klug (1968).



Experimental

Crystal data

$C_9H_{13}NO_2S$
M_r = 199.26
Orthorhombic, $P2_12_12_1$
a = 5.2259 (6) Å
b = 5.5567 (8) Å
c = 34.056 (1) Å
V = 988.94 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 301 (2) K
0.50 × 0.44 × 0.22 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: none
6529 measured reflections
2023 independent reflections
1831 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.130
S = 1.07
2023 reflections
122 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.24 e Å^{-3}
Absolute structure: Flack (1983), with 781 Friedel pairs
Flack parameter: 0.22 (16)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5N···O3$^{\rm i}$	0.854 (10)	2.130 (11)	2.981 (4)	175 (4)

Symmetry code: (i) $x + 1, y, z$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2017).

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supplementary materials

Acta Cryst. (2007). E**63**, o3014 [**doi:10.1107/S1600536807025202**]

N-(2,3-Dimethylphenyl)methanesulfonamide

B. T. Gowda, **S. Foro** and **H. Fuess**

Comment

The biological activity of alkyl sulfonanilides is thought to be due to the hydrogen of the phenyl N—H portion of the sulfonanilide molecules as it can align itself, in relation to a receptor site. Therefore the structural studies of sulfonanilides are of interest. In the present work, the structure of *N*-(2,3-dimethylphenyl)-methanesulfonamide (23DMPMSA) has been determined to explore the substituent effects on the solid state structures of sulfonanilides (Gowda *et al.*, 2007*a-g*). The structure of 23DMPMSA (Fig. 1) resembles those of *N*-(phenyl)-methanesulfonamide (PMSA) (Klug, 1968), *N*-(2-methylphenyl)-methanesulfonamide (2MPMSA) (Gowda *et al.*, 2007*d*), *N*-(3-methylphenyl)- methanesulfonamide (3MPMSA)(Gowda *et al.*, 2007*b*) and other alkyl sulfonanilides (Gowda *et al.*, 2007*a,c,e-g*). The conformation of the N—H bond lies between *syn* and *anti* conformations to the methyl substituents at *ortho* and *meta* positions, in contrast to the *syn* and *anti* conformations observed, respectively, for the 2MPMSA and 3MPMSA. Dimethyl substitutions at both *ortho* and *meta* positions in PMSA changes its space group from monoclinic $P2_1/c$ to orthorhombic $P2_12_12_1$, compared to the change over from monoclinic $P2_1/c$ to triclinic P-1 on *ortho* methyl substitution in PMSA and from monoclinic $P2_1/c$ to orthorhombic Pccn on *meta* substitution in PMSA. The geometric parameters in PMSA, 2MPMSA, 3MPMSA and 23DMPMSA are similar except for some difference in the torsional angles, C1S2N5C6, S2N5C6C7, S2N5C6C11, O3S2N5C6, O4S2N5C6: 62.2 (2)°, 75.5 (2)°, −106.6 (2)°, −54.4 (2)°, 177.7 (2)° (PMSA); −64.5 (2)°, 117.1 (2)°, −65.3 (3)°, 51.3 (2)°, 179.1 (2)° (2MPMSA); 57.9 (3)°, 68.1 (4)°, −114.3 (3)°, −57.7 (3)°, 174.7 (3)° (3MPMSA); 71.4 (3)°, 70.1 (4)°, −110.8 (3)°, −44.9 (3)°, −172.6 (3)° (23DMPMSA), respectively. The data included for PMSA are the values determined under the present conditions as the literature values were determined in Klug, 1968. The N—H hydrogen sits alone on one side of the plane of the phenyl group, while the whole methanesulfonyl group is on the opposite side of the plane, similar to that in PMSA, 2MPMSA and 3MPMSA. The amide hydrogen is thus available to a receptor molecule during its biological activity. The molecules in 23DMPMSA are packed into chains in the direction of *b* axis (Fig. 2) through N—H···O hydrogen bonds (Fig. 3 and Table 1).

Experimental

The title compound was prepared according to the literature method (Jayalakshmi & Gowda, 2004). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Jayalakshmi & Gowda, 2004). Single crystals of the title compound were obtained from a slow evaporation of its ethanolic solution and used for X-ray diffraction studied at room temperature.

Refinement

The H atom of the NH group was located in a diffrerence map and its position refined. The carbon-bound H atoms were positioned with idealized geometry and refined using a riding model with C—H = 0.93 Å (CH aromatic) or 0.96 Å (CH_3). Isotropic displacement parameters for all H atoms were set equal to 1.2 U_{eq} (parent atom).

Figures

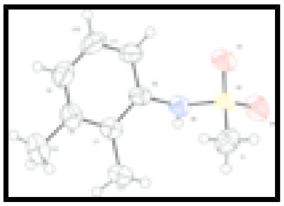

Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

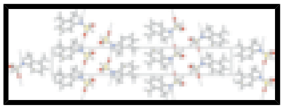

Fig. 2. The crystal packing of the title compound, viewed down the *b* axis.

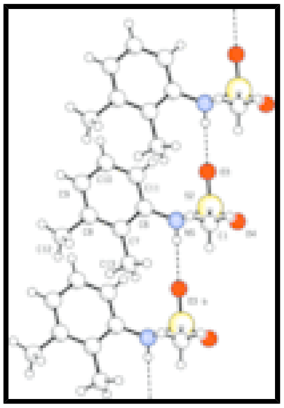

Fig. 3. Hydrogen bonding in the title compound. Hydrogen bonds are shown as dashed lines.

N-(2,3-dimethylphenyl)methanesulfonamide

Crystal data

$C_9H_{13}NO_2S$

$M_r = 199.26$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.2259\ (6)$ Å

$b = 5.5567\ (8)$ Å

$c = 34.056\ (1)$ Å

$V = 988.94\ (18)$ Å^3

$Z = 4$

$F_{000} = 424$

$D_x = 1.338$ Mg m^{-3}

Mo *K*α radiation
λ = 0.71073 Å

Cell parameters from 2323 reflections

θ = 3.6–23.2°

$\mu = 0.30$ mm^{-1}

$T = 301\ (2)$ K

Prism, gray

0.50 × 0.44 × 0.22 mm

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 301(2)$ K

Rotation method data acquisition using ω scans

Absorption correction: none

1831 reflections with $I > 2\sigma(I)$

$R_{int} = 0.068$

$\theta_{max} = 26.4$°

$\theta_{min} = 4.1$°

$h = -6 \rightarrow 5$

$k = -6 \rightarrow 6$

6529 measured reflections	$l = -40 \rightarrow 42$
2023 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2 + 0.7116P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.130$	$(\Delta/\sigma)_{max} = 0.035$
$S = 1.07$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
2023 reflections	$\Delta\rho_{min} = -0.24$ e Å^{-3}
122 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.016 (4)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 781 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.22 (16)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S2	0.70935 (16)	0.32902 (18)	0.19688 (2)	0.0436 (3)
O3	0.4634 (4)	0.4002 (5)	0.18247 (8)	0.0547 (7)
O4	0.8125 (6)	0.4515 (5)	0.22983 (8)	0.0623 (8)
N5	0.9112 (5)	0.3672 (6)	0.16118 (8)	0.0415 (7)
H5N	1.067 (3)	0.383 (8)	0.1683 (10)	0.050*
C1	0.7014 (10)	0.0208 (8)	0.20732 (12)	0.0621 (11)
H1A	0.8676	−0.0311	0.2158	0.075*
H1B	0.6534	−0.0665	0.1841	0.075*
H1C	0.5787	−0.0093	0.2277	0.075*
C6	0.8579 (6)	0.2927 (6)	0.12190 (9)	0.0404 (8)
C7	0.9912 (6)	0.0995 (6)	0.10470 (10)	0.0422 (8)
C8	0.9385 (8)	0.0450 (7)	0.06527 (11)	0.0518 (9)

C9	0.7513 (7)	0.1698 (10)	0.04547 (11)	0.0622 (11)
H9	0.7136	0.1287	0.0196	0.075*
C10	0.6192 (8)	0.3537 (10)	0.06313 (11)	0.0643 (12)
H10	0.4922	0.4348	0.0493	0.077*
C11	0.6736 (7)	0.4186 (7)	0.10106 (11)	0.0526 (9)
H11	0.5873	0.5463	0.1127	0.063*
C12	1.0809 (9)	−0.1528 (10)	0.04468 (13)	0.0711 (12)
H12A	1.0426	−0.3040	0.0570	0.085*
H12B	1.2615	−0.1226	0.0462	0.085*
H12C	1.0295	−0.1583	0.0176	0.085*
C13	1.1869 (8)	−0.0438 (7)	0.12723 (11)	0.0530 (9)
H13A	1.1731	−0.0073	0.1547	0.064*
H13B	1.3553	−0.0031	0.1181	0.064*
H13C	1.1571	−0.2125	0.1232	0.064*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S2	0.0330 (4)	0.0514 (5)	0.0465 (4)	0.0027 (4)	−0.0021 (3)	−0.0061 (4)
O3	0.0293 (12)	0.0688 (18)	0.0659 (16)	0.0086 (12)	−0.0012 (11)	−0.0061 (13)
O4	0.0571 (16)	0.0693 (18)	0.0606 (15)	0.0058 (16)	−0.0078 (14)	−0.0272 (14)
N5	0.0264 (12)	0.0488 (17)	0.0492 (16)	−0.0008 (14)	−0.0008 (11)	−0.0026 (13)
C1	0.068 (3)	0.063 (2)	0.056 (2)	−0.003 (2)	0.001 (2)	0.0135 (18)
C6	0.0276 (15)	0.0455 (19)	0.0480 (17)	−0.0013 (14)	0.0009 (13)	0.0069 (15)
C7	0.0344 (17)	0.0362 (17)	0.056 (2)	−0.0033 (14)	0.0031 (15)	0.0020 (14)
C8	0.048 (2)	0.051 (2)	0.056 (2)	−0.0109 (18)	0.0101 (18)	−0.0011 (18)
C9	0.055 (2)	0.085 (3)	0.0461 (18)	−0.008 (3)	−0.0027 (17)	0.003 (2)
C10	0.054 (2)	0.087 (3)	0.052 (2)	0.014 (2)	−0.0065 (17)	0.021 (2)
C11	0.047 (2)	0.052 (2)	0.059 (2)	0.0118 (18)	0.0027 (18)	0.0139 (17)
C12	0.068 (3)	0.075 (3)	0.070 (3)	−0.008 (3)	0.016 (2)	−0.021 (2)
C13	0.045 (2)	0.047 (2)	0.067 (2)	0.0093 (19)	−0.0044 (19)	−0.0040 (17)

Geometric parameters (Å, °)

S2—O4	1.419 (3)	C8—C9	1.376 (6)
S2—O3	1.432 (2)	C8—C12	1.501 (6)
S2—N5	1.623 (3)	C9—C10	1.373 (6)
S2—C1	1.750 (4)	C9—H9	0.9300
N5—C6	1.428 (4)	C10—C11	1.371 (6)
N5—H5N	0.854 (10)	C10—H10	0.9300
C1—H1A	0.9600	C11—H11	0.9300
C1—H1B	0.9600	C12—H12A	0.9600
C1—H1C	0.9600	C12—H12B	0.9600
C6—C11	1.386 (5)	C12—H12C	0.9600
C6—C7	1.408 (5)	C13—H13A	0.9600
C7—C8	1.404 (5)	C13—H13B	0.9600
C7—C13	1.506 (5)	C13—H13C	0.9600
O4—S2—O3	118.66 (18)	C7—C8—C12	120.5 (4)

O4—S2—N5	106.41 (17)	C10—C9—C8	121.2 (4)
O3—S2—N5	106.90 (15)	C10—C9—H9	119.4
O4—S2—C1	108.5 (2)	C8—C9—H9	119.4
O3—S2—C1	108.6 (2)	C11—C10—C9	120.3 (4)
N5—S2—C1	107.20 (19)	C11—C10—H10	119.9
C6—N5—S2	122.5 (2)	C9—C10—H10	119.9
C6—N5—H5N	119 (3)	C10—C11—C6	119.6 (4)
S2—N5—H5N	115 (2)	C10—C11—H11	120.2
S2—C1—H1A	109.5	C6—C11—H11	120.2
S2—C1—H1B	109.5	C8—C12—H12A	109.5
H1A—C1—H1B	109.5	C8—C12—H12B	109.5
S2—C1—H1C	109.5	H12A—C12—H12B	109.5
H1A—C1—H1C	109.5	C8—C12—H12C	109.5
H1B—C1—H1C	109.5	H12A—C12—H12C	109.5
C11—C6—C7	121.1 (3)	H12B—C12—H12C	109.5
C11—C6—N5	118.0 (3)	C7—C13—H13A	109.5
C7—C6—N5	121.0 (3)	C7—C13—H13B	109.5
C8—C7—C6	117.7 (3)	H13A—C13—H13B	109.5
C8—C7—C13	120.4 (3)	C7—C13—H13C	109.5
C6—C7—C13	121.8 (3)	H13A—C13—H13C	109.5
C9—C8—C7	120.0 (4)	H13B—C13—H13C	109.5
C9—C8—C12	119.5 (4)		

O4—S2—N5—C6	−172.6 (3)	C13—C7—C8—C9	177.1 (4)
O3—S2—N5—C6	−44.9 (3)	C6—C7—C8—C12	177.7 (3)
C1—S2—N5—C6	71.4 (3)	C13—C7—C8—C12	−1.5 (5)
S2—N5—C6—C11	70.1 (4)	C7—C8—C9—C10	2.2 (6)
S2—N5—C6—C7	−110.8 (3)	C12—C8—C9—C10	−179.2 (4)
C11—C6—C7—C8	2.5 (5)	C8—C9—C10—C11	0.6 (7)
N5—C6—C7—C8	−176.5 (3)	C9—C10—C11—C6	−1.9 (6)
C11—C6—C7—C13	−178.3 (3)	C7—C6—C11—C10	0.2 (5)
N5—C6—C7—C13	2.7 (5)	N5—C6—C11—C10	179.3 (3)
C6—C7—C8—C9	−3.7 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5N···O3$^{\rm i}$	0.854 (10)	2.130 (11)	2.981 (4)	175 (4)

Symmetry codes: (i) $x+1, y, z$.

Fig. 1

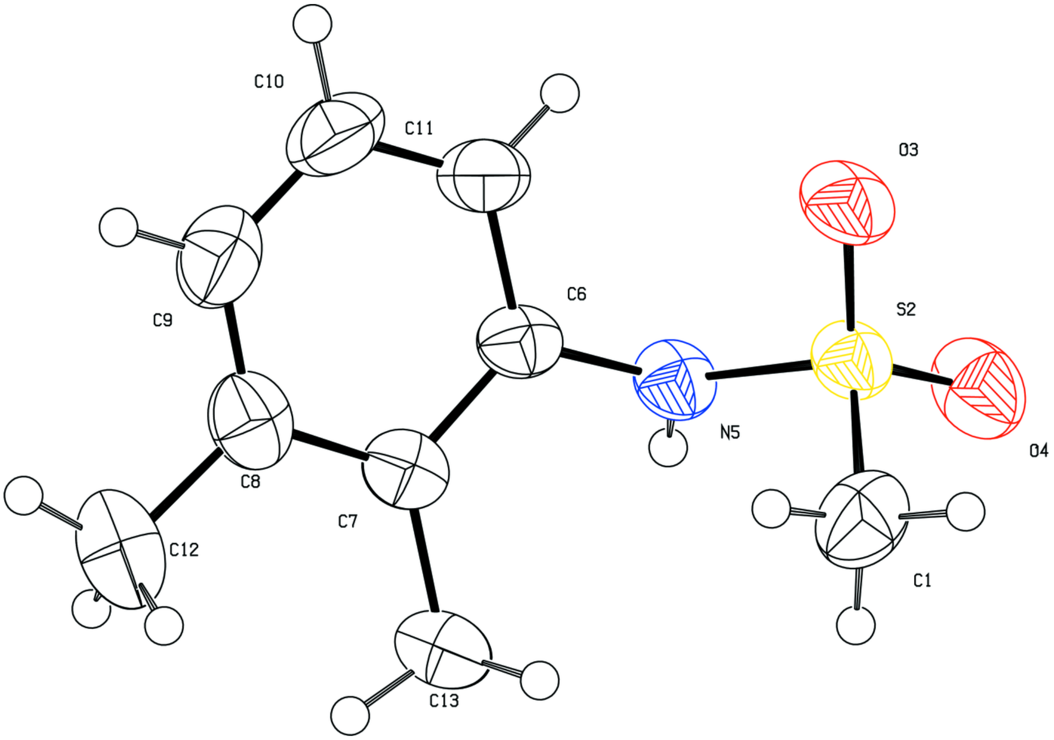

Fig. 2

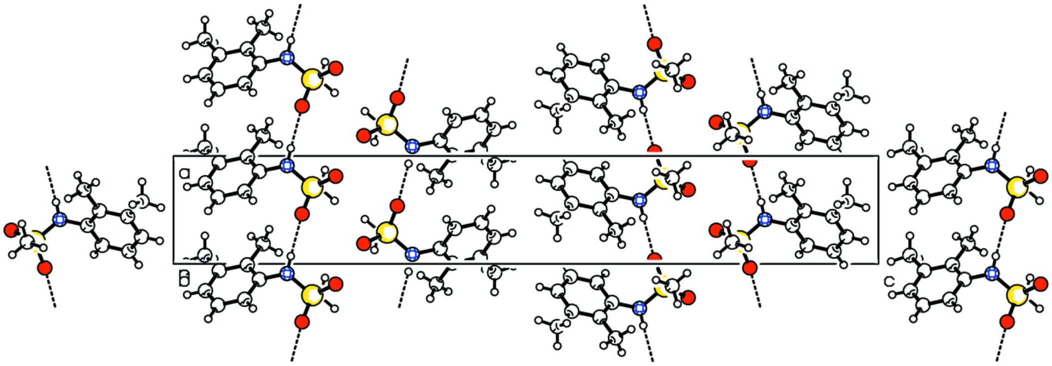

Fig. 3