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***N*-(3,5-Dimethylphenyl)-2,4-dimethylbenzenesulfonamide**B. Thimme Gowda,^{a*} Sabine Foro,^b P. G. Nirmala^a and Hartmut Fuess^b^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany
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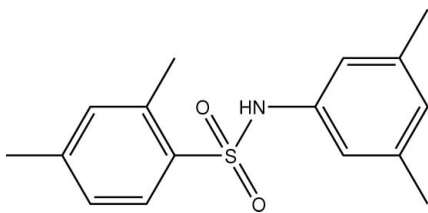
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.044; wR factor = 0.118; data-to-parameter ratio = 14.6.

In the title compound, $\text{C}_{16}\text{H}_{19}\text{NO}_2\text{S}$, the molecule is twisted about the $\text{S}-\text{N}$ bond, the $\text{C}-\text{S}(\text{O}_2)-\text{N}(\text{H})-\text{C}$ torsion angle being 53.9 (2)°. The dihedral angle between the two benzene rings is 82.1 (1)°. The crystal structure features inversion-related dimers linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the preparation of the title compound, see: Savitha & Gowda (2006). For our work on the effect of substituents on the structures of *N*-(aryl)arylsulfonamides, see: Gowda *et al.* (2009*a,b*); Nirmala *et al.* (2009). For related structures, see: Gelbrich *et al.* (2007); Perlovich *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{19}\text{NO}_2\text{S}$
 $M_r = 289.38$
Monoclinic, $C2/c$ $a = 23.490$ (2) Å
 $b = 8.1528$ (6) Å
 $c = 16.544$ (1) Å $\beta = 102.690$ (8)°
 $V = 3090.9$ (4) Å³
 $Z = 8$
Mo $K\alpha$ radiation $\mu = 0.21$ mm⁻¹
 $T = 299$ K
 $0.40 \times 0.20 \times 0.12$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford)Diffraction, 2009)
 $T_{\min} = 0.921$, $T_{\max} = 0.975$
6232 measured reflections
2751 independent reflections
2009 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.118$
 $S = 1.01$
2751 reflections
188 parameters
1 restraintH atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O1}^i$	0.84 (2)	2.10 (2)	2.945 (3)	176 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2587).

References

- Gelbrich, T., Hursthouse, M. B. & Threlfall, T. L. (2007). *Acta Cryst.* **B63**, 621–632.
- Gowda, B. T., Foro, S., Nirmala, P. G., Babitha, K. S. & Fuess, H. (2009*a*). *Acta Cryst.* **E65**, o576.
- Gowda, B. T., Foro, S., Nirmala, P. G. & Fuess, H. (2009*b*). *Acta Cryst.* **E65**, o1976.
- Nirmala, P. G., Gowda, B. T., Foro, S. & Fuess, H. (2009). *Acta Cryst.* **E65**, o3225.
- Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
- Perlovich, G. L., Tkachev, V. V., Schaper, K.-J. & Raevsky, O. A. (2006). *Acta Cryst.* **E62**, o780–o782.
- Savitha, M. B. & Gowda, B. T. (2006). *Z. Naturforsch. Teil A*, **60**, 600–606.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2009). E65, o3275 [doi:10.1107/S1600536809050740]

N-(3,5-Dimethylphenyl)-2,4-dimethylbenzenesulfonamide

B. T. Gowda, S. Foro, P. G. Nirmala and H. Fuess

Comment

As part of a study of substituent effects on the structures of *N*-(aryl)arylsulfonamides (Gowda *et al.*, 2009*a,b*; Nirmala *et al.*, 2009), in the present work, the structure of 2,4-dimethyl-*N*-(3,5-dimethylphenyl)benzenesulfonamide (I) has been determined (Fig. 1). The molecule is bent at the S–O bond atom with the C1—SO₂—NH—C7 torsion angle being 53.9 (2)°, compared to the values of 46.1 (3)° and 47.7 (3)° in the two independent molecules of 2,4-dimethyl-*N*-(3,5-dimethylphenyl)benzenesulfonamide (II) (Gowda *et al.*, 2009*a*), 67.9 (2)° in *N*-(3,5-dimethylphenyl)benzenesulfonamide (III) (Nirmala *et al.*, 2009) and -69.7 (2)° in 2,4-dimethyl-*N*-(3,4-dichlorophenyl)benzenesulfonamide (IV) (Gowda *et al.*, 2009*b*).

The two benzene rings in (I) are tilted relative to each other by 82.1 (1)°, compared to the values of 67.5 (1)° (molecule 1) and 72.9 (1)° (molecule 2) in the two independent molecules of (II), 54.6 (1)° in (III) and 82.4 (1)° in (IV). The other bond parameters in (I) are similar to those observed in (II), (III), (IV) and other aryl sulfonamides (Perlovich *et al.*, 2006; Gelbrich *et al.*, 2007). The crystal packing of molecules in (I) is through pairs of N—H···O(S) hydrogen bonds (Table 1).

Experimental

The solution of *m*-xylene (10 cc) in chloroform (40 cc) was treated drop-wise with chlorosulfonic acid (25 ml) at 273 K. After the initial evolution of hydrogen chloride subsided, the reaction mixture was brought to room temperature and poured into crushed ice in a beaker. The chloroform layer was separated, washed with cold water and allowed to evaporate slowly. The residual 2,4-dimethylbenzenesulfonylchloride was treated with a stoichiometric ratio of 3,4-dimethylaniline and boiled for ten minutes. The reaction mixture was then cooled to room temperature and added to ice-cold water (100 ml). The resultant solid, 2,4-dimethyl-*N*-(3,5-dimethylphenyl)benzenesulfonamide, was filtered under suction and washed thoroughly with cold water. It was then recrystallized to constant melting point from dilute ethanol. The purity of the compound was checked and characterized by recording its infrared and NMR spectra (Savitha & Gowda, 2006). The single crystals used in X-ray diffraction studies were grown in ethanolic solution by slow evaporation at room temperature.

Refinement

The H atom of the NH group was located in a difference map and refined with the distance restraint N—H = 0.86 (2) Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The C atoms were included in the riding model approximation with C—H = 0.93–0.96 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

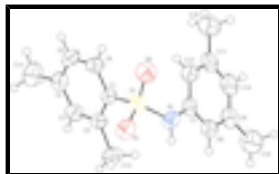


Fig. 1. Molecular structure of (I), showing the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level. The H atoms are represented as small spheres of arbitrary radii.

N-(3,5-Dimethylphenyl)-2,4-dimethylbenzenesulfonamide

Crystal data

$C_{16}H_{19}NO_2S$

$M_r = 289.38$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 23.490\ (2)\ \text{\AA}$

$b = 8.1528\ (6)\ \text{\AA}$

$c = 16.544\ (1)\ \text{\AA}$

$\beta = 102.690\ (8)^\circ$

$V = 3090.9\ (4)\ \text{\AA}^3$

$Z = 8$

$F(000) = 1232$

$D_x = 1.244\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2194 reflections

$\theta = 2.5\text{--}27.9^\circ$

$\mu = 0.21\ \text{mm}^{-1}$

$T = 299\ \text{K}$

Prism, colourless

$0.40 \times 0.20 \times 0.12\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector

Radiation source: fine-focus sealed tube graphite

Rotation method data acquisition using ω and ϕ scans.

Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.921$, $T_{\max} = 0.975$

6232 measured reflections

2751 independent reflections

2009 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 25.3^\circ$, $\theta_{\min} = 2.7^\circ$

$h = -28 \rightarrow 17$

$k = -9 \rightarrow 9$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.118$

$S = 1.01$

2751 reflections

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 3.0162P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$

188 parameters

$$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$$

1 restraint

$$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.05610 (3)	0.54362 (8)	0.39886 (4)	0.0472 (2)
O1	-0.00491 (7)	0.5510 (2)	0.40012 (10)	0.0581 (5)
O2	0.08128 (8)	0.6784 (2)	0.36408 (12)	0.0655 (5)
N1	0.08841 (9)	0.5250 (3)	0.49593 (13)	0.0519 (5)
H1N	0.0648 (10)	0.498 (3)	0.5253 (15)	0.062*
C1	0.07002 (9)	0.3654 (3)	0.34572 (13)	0.0382 (5)
C2	0.05851 (9)	0.2067 (3)	0.37077 (14)	0.0421 (5)
C3	0.06794 (10)	0.0785 (3)	0.32105 (16)	0.0492 (6)
H3	0.0611	-0.0277	0.3372	0.059*
C4	0.08716 (10)	0.0999 (3)	0.24799 (16)	0.0498 (6)
C5	0.09849 (10)	0.2566 (3)	0.22560 (15)	0.0525 (6)
H5	0.1119	0.2739	0.1774	0.063*
C6	0.09019 (10)	0.3886 (3)	0.27382 (15)	0.0480 (6)
H6	0.0982	0.4940	0.2580	0.058*
C7	0.14835 (10)	0.4895 (3)	0.52826 (15)	0.0458 (6)
C8	0.16296 (10)	0.4248 (3)	0.60749 (15)	0.0508 (6)
H8	0.1337	0.4019	0.6357	0.061*
C9	0.22056 (11)	0.3939 (3)	0.64514 (17)	0.0606 (7)
C10	0.26309 (12)	0.4250 (4)	0.60070 (19)	0.0675 (8)
H10	0.3019	0.4029	0.6252	0.081*
C11	0.24971 (11)	0.4873 (4)	0.52134 (18)	0.0612 (8)
C12	0.19150 (11)	0.5218 (3)	0.48498 (17)	0.0564 (7)
H12	0.1817	0.5663	0.4320	0.068*
C13	0.03632 (13)	0.1703 (4)	0.44804 (16)	0.0636 (7)
H13A	0.0022	0.2352	0.4479	0.076*
H13B	0.0660	0.1966	0.4961	0.076*
H13C	0.0266	0.0561	0.4491	0.076*
C14	0.09508 (14)	-0.0463 (4)	0.1958 (2)	0.0796 (9)
H14A	0.0580	-0.0979	0.1752	0.096*
H14B	0.1212	-0.1231	0.2289	0.096*

supplementary materials

H14C	0.1111	-0.0109	0.1501	0.096*
C15	0.23625 (14)	0.3267 (5)	0.7320 (2)	0.0931 (11)
H15A	0.2522	0.2184	0.7310	0.112*
H15B	0.2019	0.3220	0.7544	0.112*
H15C	0.2647	0.3968	0.7659	0.112*
C16	0.29662 (13)	0.5197 (5)	0.4739 (2)	0.0881 (11)
H16A	0.3020	0.4238	0.4428	0.106*
H16B	0.3325	0.5457	0.5121	0.106*
H16C	0.2852	0.6102	0.4368	0.106*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0477 (4)	0.0440 (4)	0.0490 (4)	0.0102 (3)	0.0089 (3)	0.0005 (3)
O1	0.0450 (9)	0.0733 (12)	0.0548 (10)	0.0213 (9)	0.0082 (8)	-0.0019 (10)
O2	0.0843 (13)	0.0390 (10)	0.0738 (13)	0.0028 (9)	0.0189 (10)	0.0074 (10)
N1	0.0434 (12)	0.0667 (14)	0.0455 (12)	0.0089 (10)	0.0098 (9)	-0.0063 (11)
C1	0.0364 (11)	0.0397 (13)	0.0386 (12)	0.0039 (9)	0.0083 (9)	0.0025 (10)
C2	0.0403 (13)	0.0429 (13)	0.0435 (12)	0.0022 (10)	0.0103 (10)	0.0042 (11)
C3	0.0502 (14)	0.0397 (13)	0.0576 (15)	0.0004 (11)	0.0114 (12)	0.0040 (12)
C4	0.0441 (13)	0.0535 (16)	0.0507 (14)	0.0080 (11)	0.0078 (11)	-0.0066 (13)
C5	0.0549 (15)	0.0656 (18)	0.0405 (13)	0.0069 (13)	0.0177 (11)	0.0018 (13)
C6	0.0504 (14)	0.0478 (14)	0.0471 (14)	0.0012 (11)	0.0134 (11)	0.0089 (12)
C7	0.0405 (13)	0.0456 (14)	0.0498 (14)	0.0034 (10)	0.0065 (10)	-0.0145 (12)
C8	0.0473 (14)	0.0528 (15)	0.0512 (15)	0.0002 (11)	0.0086 (11)	-0.0078 (13)
C9	0.0523 (16)	0.0623 (17)	0.0615 (17)	-0.0007 (13)	0.0002 (13)	-0.0054 (15)
C10	0.0425 (15)	0.080 (2)	0.074 (2)	0.0033 (14)	-0.0010 (13)	-0.0087 (17)
C11	0.0429 (14)	0.0707 (19)	0.0708 (19)	-0.0047 (13)	0.0140 (13)	-0.0191 (16)
C12	0.0518 (15)	0.0667 (18)	0.0498 (14)	-0.0008 (13)	0.0095 (12)	-0.0092 (14)
C13	0.0804 (19)	0.0584 (17)	0.0598 (17)	-0.0053 (14)	0.0324 (14)	0.0097 (15)
C14	0.089 (2)	0.075 (2)	0.076 (2)	0.0131 (18)	0.0195 (17)	-0.0212 (18)
C15	0.069 (2)	0.116 (3)	0.083 (2)	-0.0011 (19)	-0.0087 (16)	0.024 (2)
C16	0.0550 (18)	0.120 (3)	0.094 (2)	-0.0072 (18)	0.0268 (16)	-0.016 (2)

Geometric parameters (\AA , $^\circ$)

S1—O2	1.4276 (18)	C8—H8	0.9300
S1—O1	1.4392 (17)	C9—C10	1.388 (4)
S1—N1	1.626 (2)	C9—C15	1.506 (4)
S1—C1	1.766 (2)	C10—C11	1.378 (4)
N1—C7	1.422 (3)	C10—H10	0.9300
N1—H1N	0.844 (16)	C11—C12	1.396 (3)
C1—C6	1.386 (3)	C11—C16	1.510 (4)
C1—C2	1.403 (3)	C12—H12	0.9300
C2—C3	1.378 (3)	C13—H13A	0.9600
C2—C13	1.512 (3)	C13—H13B	0.9600
C3—C4	1.391 (3)	C13—H13C	0.9600
C3—H3	0.9300	C14—H14A	0.9600
C4—C5	1.373 (4)	C14—H14B	0.9600

C4—C14	1.507 (4)	C14—H14C	0.9600
C5—C6	1.379 (3)	C15—H15A	0.9600
C5—H5	0.9300	C15—H15B	0.9600
C6—H6	0.9300	C15—H15C	0.9600
C7—C8	1.384 (3)	C16—H16A	0.9600
C7—C12	1.389 (3)	C16—H16B	0.9600
C8—C9	1.383 (3)	C16—H16C	0.9600
O2—S1—O1	118.40 (11)	C10—C9—C15	121.2 (3)
O2—S1—N1	108.98 (12)	C11—C10—C9	122.1 (2)
O1—S1—N1	103.88 (11)	C11—C10—H10	118.9
O2—S1—C1	107.12 (11)	C9—C10—H10	118.9
O1—S1—C1	109.61 (10)	C10—C11—C12	118.8 (2)
N1—S1—C1	108.52 (11)	C10—C11—C16	121.3 (3)
C7—N1—S1	126.83 (17)	C12—C11—C16	119.9 (3)
C7—N1—H1N	117.0 (19)	C7—C12—C11	119.8 (3)
S1—N1—H1N	111.7 (19)	C7—C12—H12	120.1
C6—C1—C2	120.2 (2)	C11—C12—H12	120.1
C6—C1—S1	116.78 (18)	C2—C13—H13A	109.5
C2—C1—S1	122.89 (17)	C2—C13—H13B	109.5
C3—C2—C1	117.2 (2)	H13A—C13—H13B	109.5
C3—C2—C13	119.1 (2)	C2—C13—H13C	109.5
C1—C2—C13	123.7 (2)	H13A—C13—H13C	109.5
C2—C3—C4	123.3 (2)	H13B—C13—H13C	109.5
C2—C3—H3	118.3	C4—C14—H14A	109.5
C4—C3—H3	118.3	C4—C14—H14B	109.5
C5—C4—C3	118.1 (2)	H14A—C14—H14B	109.5
C5—C4—C14	121.7 (2)	C4—C14—H14C	109.5
C3—C4—C14	120.2 (3)	H14A—C14—H14C	109.5
C4—C5—C6	120.7 (2)	H14B—C14—H14C	109.5
C4—C5—H5	119.7	C9—C15—H15A	109.5
C6—C5—H5	119.7	C9—C15—H15B	109.5
C5—C6—C1	120.6 (2)	H15A—C15—H15B	109.5
C5—C6—H6	119.7	C9—C15—H15C	109.5
C1—C6—H6	119.7	H15A—C15—H15C	109.5
C8—C7—C12	120.2 (2)	H15B—C15—H15C	109.5
C8—C7—N1	116.9 (2)	C11—C16—H16A	109.5
C12—C7—N1	122.8 (2)	C11—C16—H16B	109.5
C9—C8—C7	120.7 (2)	H16A—C16—H16B	109.5
C9—C8—H8	119.7	C11—C16—H16C	109.5
C7—C8—H8	119.7	H16A—C16—H16C	109.5
C8—C9—C10	118.4 (3)	H16B—C16—H16C	109.5
C8—C9—C15	120.4 (3)		
O2—S1—N1—C7	-62.4 (2)	C14—C4—C5—C6	179.1 (2)
O1—S1—N1—C7	170.5 (2)	C4—C5—C6—C1	-0.2 (4)
C1—S1—N1—C7	53.9 (2)	C2—C1—C6—C5	0.8 (3)
O2—S1—C1—C6	-12.4 (2)	S1—C1—C6—C5	-175.71 (18)
O1—S1—C1—C6	117.20 (18)	S1—N1—C7—C8	-158.6 (2)
N1—S1—C1—C6	-129.97 (18)	S1—N1—C7—C12	23.5 (4)

supplementary materials

O2—S1—C1—C2	171.16 (18)	C12—C7—C8—C9	1.0 (4)
O1—S1—C1—C2	-59.2 (2)	N1—C7—C8—C9	-177.0 (2)
N1—S1—C1—C2	53.6 (2)	C7—C8—C9—C10	-1.8 (4)
C6—C1—C2—C3	-0.2 (3)	C7—C8—C9—C15	178.8 (3)
S1—C1—C2—C3	176.07 (17)	C8—C9—C10—C11	1.0 (4)
C6—C1—C2—C13	-179.8 (2)	C15—C9—C10—C11	-179.6 (3)
S1—C1—C2—C13	-3.6 (3)	C9—C10—C11—C12	0.6 (4)
C1—C2—C3—C4	-1.0 (3)	C9—C10—C11—C16	-179.8 (3)
C13—C2—C3—C4	178.7 (2)	C8—C7—C12—C11	0.7 (4)
C2—C3—C4—C5	1.5 (4)	N1—C7—C12—C11	178.5 (2)
C2—C3—C4—C14	-178.5 (2)	C10—C11—C12—C7	-1.5 (4)
C3—C4—C5—C6	-0.9 (4)	C16—C11—C12—C7	179.0 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1N \cdots O1 ⁱ	0.84 (2)	2.10 (2)	2.945 (3)	176 (3)

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

Fig. 1

