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3,4-Dimethylbenzenesulfonamide

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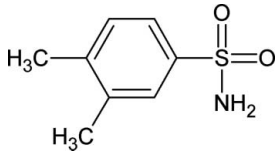
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.107; data-to-parameter ratio = 13.7.

The structure of the title compound (34DMBSA), $C_8H_{11}NO_2S$, resembles those of other arylsulfonamides. The molecules are packed into a layered supramolecular structure, in the *ac* plane, *via* N—H···O hydrogen bonds.

Related literature

For related literature, see: Gowda & Shetty (2004); Gowda *et al.* (2002, 2003, 2005, 2007); Jones & Weinkauf (1993); Kumar *et al.* (1992); O'Connor & Maslen (1965).



Experimental

Crystal data

$C_8H_{11}NO_2S$
M_r = 185.24
Monoclinic, $P2_1/c$
a = 9.7939 (4) Å
b = 9.5488 (5) Å
c = 10.3342 (8) Å
β = 109.936 (5)°
V = 908.54 (9) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.85 mm^{-1}
T = 299 (2) K
0.57 × 0.35 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.366, T_{max} = 0.586
3312 measured reflections
1618 independent reflections
1524 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
3 standard reflections
frequency: 120 min
intensity decay: 3.5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.107
S = 1.11
1618 reflections
118 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11N···O2^{i}	0.85 (1)	2.12 (1)	2.952 (2)	167 (2)
N1—H12N···O1^{ii}	0.85 (1)	2.16 (1)	2.999 (2)	176 (2)

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

Data collection: *CAD-4-PC* (Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*, *PLATON* (Spek, 2003) and *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2016).

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supplementary materials

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3,4-Dimethylbenzenesulfonamide

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Comment

Many arylsulfonamides and their N-halo compounds show distinct physical, chemical and biological properties due to their oxidizing action in aqueous, partial aqueous and non-aqueous media. This class of compounds therefore is of interest in synthetic, mechanistic, analytical and biological chemistry (Gowda *et al.*, 2002, 2003, 2005, 2007; Gowda & Shetty, 2004). In the present work, the structure of 3,4-dimethylbenzenesulfonamde (34DMBSA) has been determined to explore the substituent effects on the solid state structures of sulfonamides and N-halo arylsulfonamides (Gowda *et al.*, 2003, 2007). The structure of 34DMBSA (Fig. 1) resembles those of other aryl sulfonamides (Gowda *et al.*, 2003; Jones & Weinkauf, 1993; Kumar *et al.*, 1992; O'Connor & Maslen, 1965). 34DMBSA crystallizes in monoclinic P 2_1/c space group in contrast to the monoclinic *Pc* space group of the parent benzenesulfonamide, orthorhombic Pbca space group observed with 4-fluorobenzenesulfonamide (Jones & Weinkauf, 1993) and 4-aminobenzenesulfonamide (O'Connor & Maslen, 1965), monoclinic P21/n space group with 4-chlorobenzenesulfonamide and 4-bromobenzenesulfonamide (Gowda *et al.*, 2003), and 4-methylbenzenesulfonamide (Kumar *et al.*, 1992). Introduction of two methyl groups at the *meta* and *para* positions of the benzenesulfonamide slightly decreases the S—N bond length while increasing the S—O bond lengths. Nevertheless, the other bond parameters are not significantly altered. The molecules in the title compound are packed into a layered supramolecular structure as viewed down the *ac* plane through hydrogen bonding (Fig. 2).

Experimental

The title compound was prepared according to the literature method (Gowda *et al.*, 2002). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Gowda *et al.*, 2002). Single crystals of the title compound were obtained from a slow evaporation of its ethanolic solution and used for X-ray diffraction studies at room temperature.

Figures

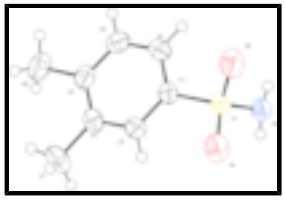

Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at 50% probability level.

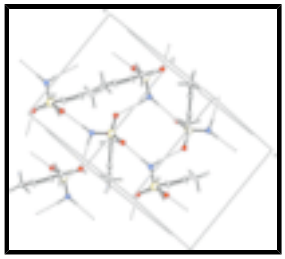

Fig. 2. Typical Hydrogen bond bridges observed in the title compound.

3,4-Dimethylbenzenesulfonamide

Crystal data

$C_8H_{11}NO_2S$	F_{000} = 392
M_r = 185.24	D_x = 1.354 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation λ = 1.54180 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 9.7939 (4) Å	θ = 9.3–30.1º
b = 9.5488 (5) Å	μ = 2.85 mm^{-1}
c = 10.3342 (8) Å	T = 299 (2) K
β = 109.936 (5)º	Prism, colourless
V = 908.54 (9) Å^3	0.58 × 0.35 × 0.2 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.040
Radiation source: fine-focus sealed tube	θ_{max} = 66.9º
Monochromator: graphite	θ_{min} = 4.8º
T = 299(2) K	h = −10→11
ω/2θ scans	k = −11→11
Absorption correction: ψ scan (North *et al.*, 1968)	l = −12→0
T_{min} = 0.366, T_{max} = 0.586	3 standard reflections
3312 measured reflections	every 120 min
1618 independent reflections	intensity decay: 3.5%
1524 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.107	$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.283P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.11	$(\Delta/\sigma)_{max} < 0.001$
1618 reflections	$\Delta\rho_{max} = 0.46$ e Å^{-3}
118 parameters	$\Delta\rho_{min} = -0.44$ e Å^{-3}
2 restraints	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0240 (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}
C1	0.74966 (18)	0.27510 (19)	0.30043 (17)	0.0353 (4)
C2	0.86716 (19)	0.2192 (2)	0.40414 (19)	0.0395 (4)
H2	0.8540	0.1410	0.4522	0.047*
C3	1.00472 (19)	0.2784 (2)	0.43747 (19)	0.0408 (4)
C4	1.02302 (19)	0.3965 (2)	0.36621 (19)	0.0430 (5)
C5	0.9025 (2)	0.4516 (2)	0.2627 (2)	0.0499 (5)
H5	0.9145	0.5309	0.2154	0.060*
C6	0.7666 (2)	0.3922 (2)	0.22840 (19)	0.0458 (5)
H6	0.6880	0.4299	0.1585	0.055*
C7	1.1309 (2)	0.2136 (3)	0.5497 (3)	0.0624 (6)
H7A	1.1701	0.2804	0.6224	0.075*
H7B	1.2046	0.1869	0.5127	0.075*
H7C	1.0981	0.1323	0.5854	0.075*
C8	1.1693 (2)	0.4650 (3)	0.3989 (2)	0.0601 (6)
H8A	1.1579	0.5548	0.3547	0.072*
H8B	1.2308	0.4070	0.3663	0.072*
H8C	1.2127	0.4770	0.4967	0.072*
N1	0.47754 (17)	0.29125 (18)	0.31866 (16)	0.0436 (4)
H11N	0.502 (2)	0.294 (2)	0.4059 (10)	0.052*
H12N	0.457 (2)	0.3705 (15)	0.281 (2)	0.052*
O1	0.59702 (15)	0.06501 (15)	0.33109 (17)	0.0574 (4)
O2	0.51100 (16)	0.19748 (19)	0.11336 (15)	0.0629 (5)
S1	0.57753 (4)	0.19636 (5)	0.25981 (4)	0.0384 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0331 (8)	0.0384 (9)	0.0333 (8)	0.0005 (7)	0.0098 (7)	−0.0049 (7)
C2	0.0383 (9)	0.0374 (10)	0.0393 (9)	0.0011 (7)	0.0087 (7)	0.0002 (7)
C3	0.0348 (9)	0.0437 (10)	0.0401 (10)	0.0022 (8)	0.0078 (7)	−0.0046 (8)
C4	0.0380 (9)	0.0517 (11)	0.0417 (9)	−0.0043 (8)	0.0168 (7)	−0.0074 (8)
C5	0.0527 (11)	0.0530 (12)	0.0464 (10)	−0.0059 (9)	0.0200 (9)	0.0098 (9)
C6	0.0419 (10)	0.0515 (12)	0.0393 (9)	0.0037 (9)	0.0078 (7)	0.0075 (8)
C7	0.0427 (11)	0.0646 (15)	0.0625 (14)	0.0030 (10)	−0.0046 (10)	0.0050 (11)
C8	0.0464 (11)	0.0774 (16)	0.0598 (13)	−0.0166 (11)	0.0225 (10)	−0.0082 (11)
N1	0.0402 (8)	0.0527 (11)	0.0380 (9)	0.0082 (7)	0.0135 (7)	0.0017 (7)
O1	0.0498 (8)	0.0372 (8)	0.0800 (11)	−0.0059 (6)	0.0154 (7)	−0.0012 (7)
O2	0.0478 (8)	0.1006 (14)	0.0346 (8)	−0.0125 (8)	0.0066 (6)	−0.0214 (7)
S1	0.0327 (3)	0.0430 (3)	0.0363 (3)	−0.00293 (16)	0.0076 (2)	−0.00863 (16)

Geometric parameters (Å, °)

C1—C6	1.384 (3)	C7—H7A	0.9600
C1—C2	1.384 (3)	C7—H7B	0.9600
C1—S1	1.7610 (18)	C7—H7C	0.9600
C2—C3	1.392 (3)	C8—H8A	0.9600
C2—H2	0.9300	C8—H8B	0.9600
C3—C4	1.392 (3)	C8—H8C	0.9600
C3—C7	1.509 (3)	N1—S1	1.5987 (16)
C4—C5	1.397 (3)	N1—H11N	0.851 (10)
C4—C8	1.505 (3)	N1—H12N	0.845 (10)
C5—C6	1.378 (3)	O1—S1	1.4340 (16)
C5—H5	0.9300	O2—S1	1.4292 (15)
C6—H6	0.9300		
C6—C1—C2	120.49 (17)	H7A—C7—H7B	109.5
C6—C1—S1	119.81 (14)	C3—C7—H7C	109.5
C2—C1—S1	119.71 (14)	H7A—C7—H7C	109.5
C1—C2—C3	120.95 (18)	H7B—C7—H7C	109.5
C1—C2—H2	119.5	C4—C8—H8A	109.5
C3—C2—H2	119.5	C4—C8—H8B	109.5
C2—C3—C4	119.12 (17)	H8A—C8—H8B	109.5
C2—C3—C7	119.58 (19)	C4—C8—H8C	109.5
C4—C3—C7	121.30 (19)	H8A—C8—H8C	109.5
C3—C4—C5	118.86 (17)	H8B—C8—H8C	109.5
C3—C4—C8	121.20 (18)	S1—N1—H11N	115.8 (16)
C5—C4—C8	119.94 (19)	S1—N1—H12N	114.3 (15)
C6—C5—C4	122.11 (19)	H11N—N1—H12N	114 (2)
C6—C5—H5	118.9	O2—S1—O1	118.90 (10)
C4—C5—H5	118.9	O2—S1—N1	106.34 (9)
C5—C6—C1	118.47 (17)	O1—S1—N1	106.97 (9)
C5—C6—H6	120.8	O2—S1—C1	107.64 (9)

C1—C6—H6	120.8	O1—S1—C1	107.58 (9)
C3—C7—H7A	109.5	N1—S1—C1	109.15 (9)
C3—C7—H7B	109.5		
C6—C1—C2—C3	−0.6 (3)	C4—C5—C6—C1	0.7 (3)
S1—C1—C2—C3	179.33 (14)	C2—C1—C6—C5	−0.2 (3)
C1—C2—C3—C4	0.8 (3)	S1—C1—C6—C5	179.89 (15)
C1—C2—C3—C7	−179.12 (19)	C6—C1—S1—O2	40.23 (17)
C2—C3—C4—C5	−0.4 (3)	C2—C1—S1—O2	−139.66 (16)
C7—C3—C4—C5	179.6 (2)	C6—C1—S1—O1	169.48 (15)
C2—C3—C4—C8	179.62 (18)	C2—C1—S1—O1	−10.41 (17)
C7—C3—C4—C8	−0.4 (3)	C6—C1—S1—N1	−74.79 (16)
C3—C4—C5—C6	−0.4 (3)	C2—C1—S1—N1	105.31 (16)
C8—C4—C5—C6	179.60 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H11N···O2^{i}	0.85 (1)	2.12 (1)	2.952 (2)	167 (2)
N1—H12N···O1^{ii}	0.85 (1)	2.16 (1)	2.999 (2)	176 (2)

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1

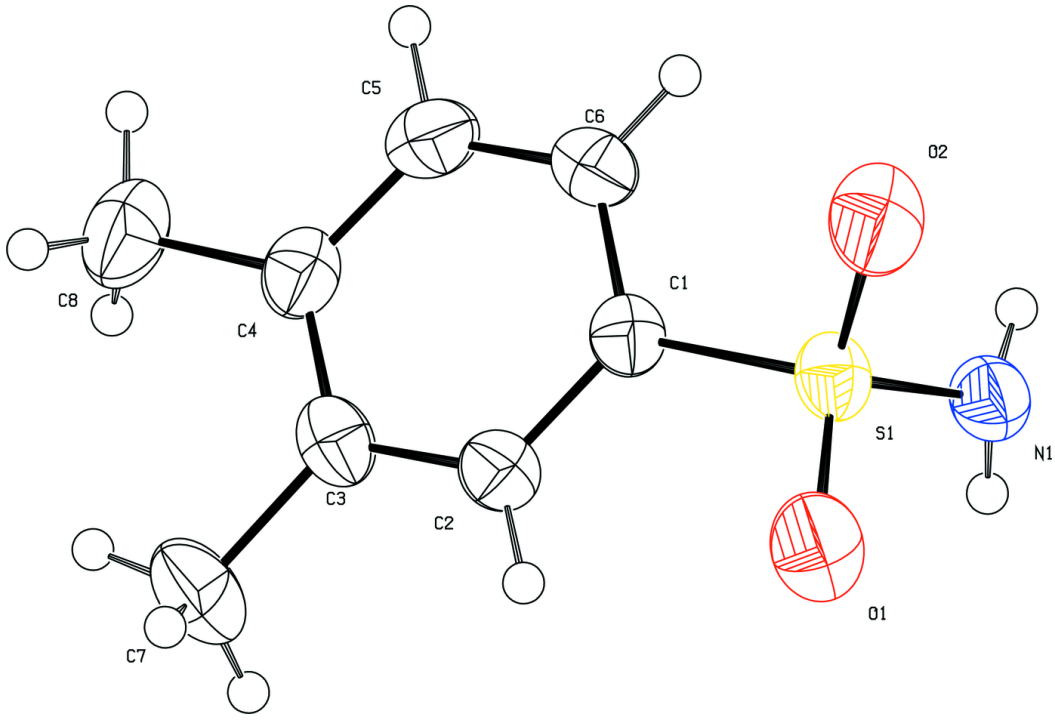

Fig. 2