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N-(3-Chlorophenyl)benzenesulfonamide

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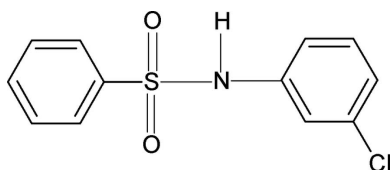
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.036; wR factor = 0.094; data-to-parameter ratio = 14.1.

In the crystal structure of the title compound, $\text{C}_{12}\text{H}_{10}\text{ClNO}_2\text{S}$, the N–H bond is *trans* to one of the S=O bonds. The two aromatic rings form a dihedral angle of $65.4(1)^\circ$, compared with a value of $49.1(1)^\circ$ in *N*-(2-chlorophenyl)-benzenesulfonamide. The molecules are connected by intermolecular N–H \cdots O hydrogen bonds into chains running along the *b* axis.

Related literature

For related literature, see: Gelbrich *et al.* (2007); Gowda *et al.* (2005, 2008a,b); Perlovich *et al.* (2006).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{10}\text{ClNO}_2\text{S}$
 $M_r = 267.72$
Tetragonal, $P4_32_12$
 $a = 8.8357(7)$ Å
 $c = 32.081(5)$ Å
 $V = 2504.6(5)$ Å³

$Z = 8$
Cu $K\alpha$ radiation
 $\mu = 4.18$ mm⁻¹
 $T = 299(2)$ K
 $0.38 \times 0.35 \times 0.33$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.222$, $T_{\max} = 0.251$
5004 measured reflections

2232 independent reflections
2054 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.071$
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.094$
 $S = 1.10$
2232 reflections
158 parameters
19 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³
Absolute structure: Flack (1983), 840 Friedel pairs
Flack parameter: $-0.01(2)$

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O2}^i$	0.88 (1)	2.029 (13)	2.875 (2)	162 (2)

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + \frac{1}{4}$.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); 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: CI2659).

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supporting information

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***N*-(3-Chlorophenyl)benzenesulfonamide**

B. Thimme Gowda, Sabine Foro, K. S. Babitha and Hartmut Fuess

S1. Comment

As part of a study of the substituent effects on the crystal structures of *N*-(aryl)-benzenesulfonamides, in the present work, the structure of *N*-(3-chlorophenyl)-benzenesulfonamide (N3CPBSA) has been determined (Gowda *et al.*, 2008*a,b*). The N—H bond is trans to one of the S=O bonds (Fig. 1). Further, the conformation of the N—H bond is *anti* to the *meta*-chloro group in the aniline benzene ring, in contrast to the *syn* conformation observed with respect to the *ortho*-chloro group in *N*-(2-chlorophenyl)-benzenesulfonamide (N2CPBSA) (Perlovich *et al.*, 2006). The two benzene rings form a dihedral angle of 65.4 (1)° compared with the value of 49.1 (1)° in N2CPBSA. The other bond parameters in N3CPBSA are similar to those observed in N2CPBSA (Perlovich *et al.*, 2006) and other *N*-(aryl)-benzenesulfonamides (Gelbrich *et al.*, 2007; Gowda *et al.*, 2008*a,b*).

The packing diagram of N3CPBSA showing the N—H···O hydrogen bonds (Table 1) is shown in Fig. 2.

S2. Experimental

The solution of benzene (10 cc) in chloroform (40 cc) was treated dropwise with chlorosulfonic acid (25 cc) 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 benzenesulfonylchloride was treated with *m*-chloroaniline in the stoichiometric ratio and boiled for 10 min. The reaction mixture was then cooled to room temperature and added to ice cold water (100 cc). The resultant solid *N*-(3-chlorophenyl)-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 (Gowda *et al.*, 2005). Single crystals used in X-ray diffraction studies were grown in an ethanolic solution by evaporating it at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and was refined with a N-H distance restraint of 0.90 (1) Å. The other H atoms were positioned with idealized geometry (C-H = 0.93 Å) and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The U^{ij} components of C4, C5 and C6 were restrained to approximate isotropic behaviour.

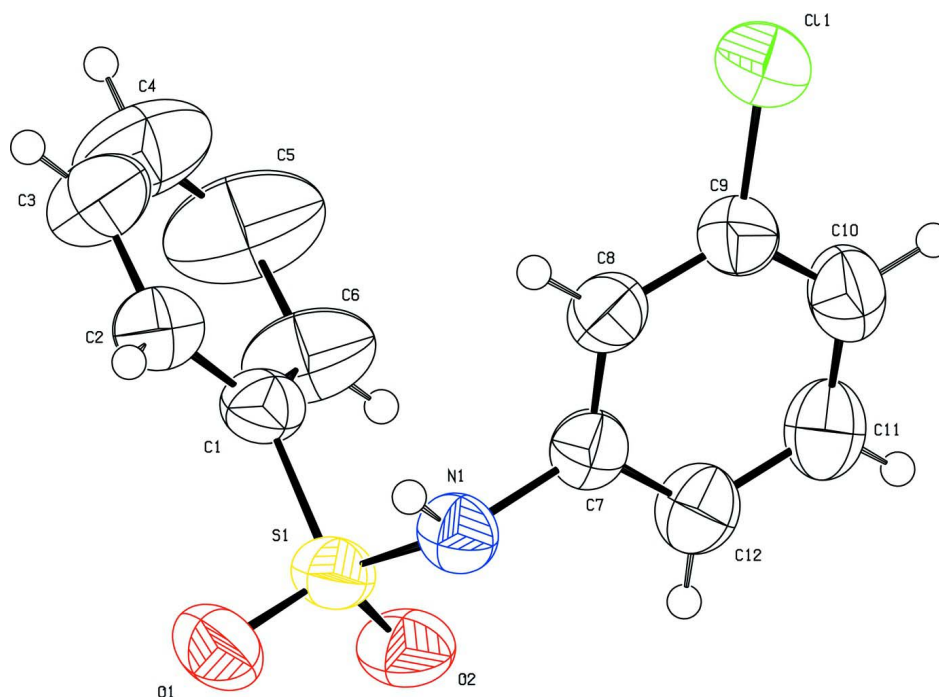


Figure 1

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.

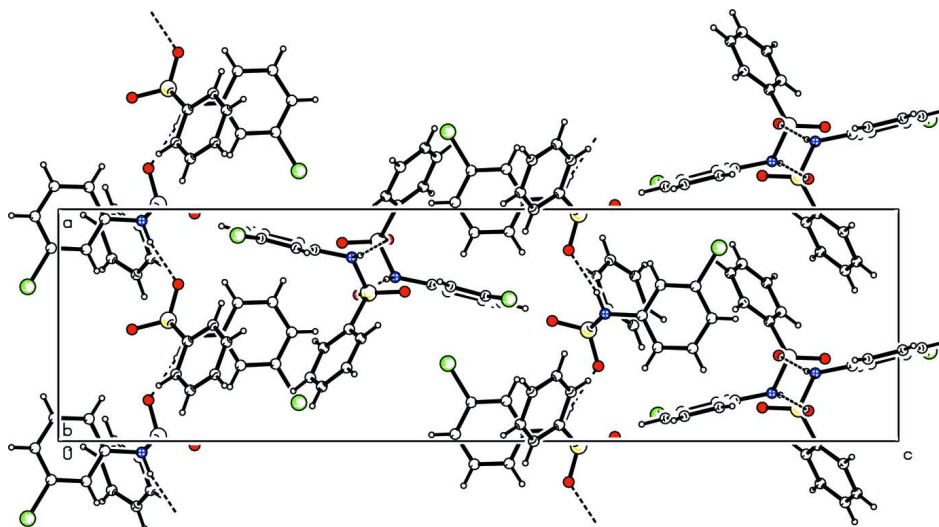


Figure 2

Molecular packing of the title compound, showing hydrogen-bonded (dashed lines) chains.

***N*-(3-Chlorophenyl)benzenesulfonamide**

Crystal data

$C_{12}H_{10}ClNO_2S$

$M_r = 267.72$

Tetragonal, $P4_32_12$

Hall symbol: P 4nw 2abw

$a = 8.8357 (7) \text{ \AA}$

$c = 32.081 (5) \text{ \AA}$

$V = 2504.6 (5) \text{ \AA}^3$
 $Z = 8$
 $F(000) = 1104$
 $D_x = 1.420 \text{ Mg m}^{-3}$
 Cu $K\alpha$ radiation, $\lambda = 1.54180 \text{ \AA}$
 Cell parameters from 25 reflections

$\theta = 6.5\text{--}18.9^\circ$
 $\mu = 4.18 \text{ mm}^{-1}$
 $T = 299 \text{ K}$
 Prism, colourless
 $0.38 \times 0.35 \times 0.33 \text{ mm}$

Data collection

Enraf–Nonius CAD-4
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.222$, $T_{\max} = 0.251$
 5004 measured reflections

2232 independent reflections
 2054 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$
 $\theta_{\max} = 66.8^\circ$, $\theta_{\min} = 5.2^\circ$
 $h = -10 \rightarrow 0$
 $k = -10 \rightarrow 0$
 $l = -38 \rightarrow 37$
 3 standard reflections every 120 min
 intensity decay: 1.0%

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.094$
 $S = 1.10$
 2232 reflections
 158 parameters
 19 restraints
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 0.1574P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.004$
 $\Delta\rho_{\max} = 0.19 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.21 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*,
 $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0031 (3)
 Absolute structure: Flack (1983), 840 Friedel
 pairs
 Absolute structure parameter: $-0.01 (2)$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	−0.0696 (3)	−0.0170 (3)	0.09615 (8)	0.0566 (6)
C2	−0.2118 (3)	−0.0569 (4)	0.10996 (9)	0.0763 (8)
H2	−0.2603	0.0001	0.1304	0.092*
C3	−0.2814 (4)	−0.1829 (4)	0.09306 (12)	0.1005 (11)
H3	−0.3771	−0.2112	0.1023	0.121*
C4	−0.2119 (5)	−0.2644 (4)	0.06353 (16)	0.1213 (15)

H4	-0.2601	-0.3486	0.0523	0.146*
C5	-0.0724 (5)	-0.2253 (5)	0.04988 (17)	0.1362 (17)
H5	-0.0247	-0.2840	0.0297	0.163*
C6	0.0006 (4)	-0.0978 (4)	0.06576 (12)	0.0987 (12)
H6	0.0951	-0.0688	0.0558	0.118*
C7	-0.0413 (3)	0.3249 (2)	0.05535 (7)	0.0518 (5)
C8	-0.1736 (3)	0.3333 (3)	0.03316 (7)	0.0571 (5)
H8	-0.2659	0.3123	0.0458	0.069*
C9	-0.1672 (3)	0.3736 (3)	-0.00854 (8)	0.0633 (6)
C10	-0.0304 (3)	0.4020 (3)	-0.02766 (8)	0.0685 (7)
H10	-0.0269	0.4276	-0.0558	0.082*
C11	0.1000 (3)	0.3923 (3)	-0.00488 (8)	0.0687 (7)
H11	0.1925	0.4114	-0.0176	0.082*
C12	0.0959 (3)	0.3547 (3)	0.03655 (8)	0.0633 (6)
H12	0.1850	0.3494	0.0519	0.076*
C11	-0.33446 (9)	0.38734 (11)	-0.03637 (3)	0.1003 (3)
N1	-0.0492 (2)	0.2925 (2)	0.09923 (6)	0.0542 (5)
H1N	-0.1414 (15)	0.300 (3)	0.1090 (7)	0.065*
O1	-0.0183 (2)	0.1447 (2)	0.16226 (5)	0.0712 (5)
O2	0.17669 (17)	0.1309 (2)	0.10738 (6)	0.0678 (5)
S1	0.02122 (6)	0.13880 (7)	0.119048 (18)	0.05501 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0552 (12)	0.0546 (11)	0.0599 (13)	0.0022 (9)	-0.0124 (11)	0.0011 (11)
C2	0.0731 (16)	0.0876 (19)	0.0681 (16)	-0.0161 (14)	-0.0063 (14)	0.0007 (15)
C3	0.092 (2)	0.093 (2)	0.117 (3)	-0.0326 (19)	-0.020 (2)	0.009 (2)
C4	0.109 (3)	0.075 (2)	0.181 (4)	-0.0087 (19)	-0.027 (3)	-0.033 (2)
C5	0.118 (3)	0.107 (3)	0.184 (4)	0.008 (2)	0.001 (3)	-0.087 (3)
C6	0.0725 (19)	0.098 (2)	0.126 (3)	0.0044 (15)	0.0010 (19)	-0.050 (2)
C7	0.0577 (12)	0.0460 (11)	0.0518 (12)	-0.0002 (9)	0.0055 (11)	-0.0063 (10)
C8	0.0565 (12)	0.0552 (12)	0.0595 (12)	-0.0069 (10)	0.0015 (11)	-0.0013 (11)
C9	0.0750 (15)	0.0592 (13)	0.0556 (13)	-0.0098 (12)	-0.0075 (12)	0.0017 (12)
C10	0.0926 (18)	0.0598 (13)	0.0532 (13)	-0.0039 (13)	0.0102 (14)	0.0022 (12)
C11	0.0671 (14)	0.0712 (15)	0.0679 (15)	0.0016 (12)	0.0192 (13)	0.0048 (13)
C12	0.0578 (12)	0.0658 (14)	0.0663 (14)	0.0007 (11)	0.0094 (12)	-0.0008 (13)
C11	0.0939 (5)	0.1250 (7)	0.0821 (5)	-0.0306 (5)	-0.0317 (4)	0.0267 (5)
N1	0.0536 (10)	0.0611 (10)	0.0481 (10)	0.0027 (8)	0.0041 (9)	-0.0041 (9)
O1	0.0762 (11)	0.0904 (12)	0.0470 (8)	0.0010 (10)	-0.0033 (8)	-0.0001 (9)
O2	0.0467 (8)	0.0850 (11)	0.0717 (11)	0.0016 (8)	-0.0091 (8)	-0.0047 (10)
S1	0.0492 (3)	0.0659 (3)	0.0499 (3)	-0.0007 (2)	-0.0058 (2)	-0.0026 (3)

Geometric parameters (Å, °)

C1—C6	1.358 (4)	C7—N1	1.438 (3)
C1—C2	1.378 (4)	C8—C9	1.386 (3)
C1—S1	1.755 (2)	C8—H8	0.93

C2—C3	1.382 (4)	C9—C10	1.378 (4)
C2—H2	0.93	C9—C11	1.731 (3)
C3—C4	1.339 (5)	C10—C11	1.367 (4)
C3—H3	0.93	C10—H10	0.93
C4—C5	1.353 (6)	C11—C12	1.371 (4)
C4—H4	0.93	C11—H11	0.93
C5—C6	1.394 (5)	C12—H12	0.93
C5—H5	0.93	N1—S1	1.623 (2)
C6—H6	0.93	N1—H1N	0.876 (10)
C7—C8	1.371 (3)	O1—S1	1.4305 (17)
C7—C12	1.379 (3)	O2—S1	1.4256 (17)
C6—C1—C2	120.8 (3)	C9—C8—H8	120.7
C6—C1—S1	120.3 (2)	C10—C9—C8	120.9 (2)
C2—C1—S1	118.9 (2)	C10—C9—C11	120.41 (19)
C1—C2—C3	119.0 (3)	C8—C9—C11	118.73 (19)
C1—C2—H2	120.5	C11—C10—C9	119.3 (2)
C3—C2—H2	120.5	C11—C10—H10	120.3
C4—C3—C2	120.5 (3)	C9—C10—H10	120.3
C4—C3—H3	119.8	C10—C11—C12	120.8 (2)
C2—C3—H3	119.8	C10—C11—H11	119.6
C3—C4—C5	120.6 (4)	C12—C11—H11	119.6
C3—C4—H4	119.7	C11—C12—C7	119.6 (2)
C5—C4—H4	119.7	C11—C12—H12	120.2
C4—C5—C6	120.6 (4)	C7—C12—H12	120.2
C4—C5—H5	119.7	C7—N1—S1	122.09 (15)
C6—C5—H5	119.7	C7—N1—H1N	112.2 (16)
C1—C6—C5	118.4 (3)	S1—N1—H1N	106.4 (17)
C1—C6—H6	120.8	O2—S1—O1	119.45 (11)
C5—C6—H6	120.8	O2—S1—N1	107.89 (11)
C8—C7—C12	120.81 (19)	O1—S1—N1	104.81 (11)
C8—C7—N1	118.6 (2)	O2—S1—C1	107.00 (12)
C12—C7—N1	120.5 (2)	O1—S1—C1	108.81 (12)
C7—C8—C9	118.7 (2)	N1—S1—C1	108.50 (10)
C7—C8—H8	120.7		
C6—C1—C2—C3	1.4 (4)	C10—C11—C12—C7	0.7 (4)
S1—C1—C2—C3	-177.7 (2)	C8—C7—C12—C11	-0.4 (4)
C1—C2—C3—C4	-0.4 (5)	N1—C7—C12—C11	-176.9 (2)
C2—C3—C4—C5	0.3 (7)	C8—C7—N1—S1	115.6 (2)
C3—C4—C5—C6	-1.2 (8)	C12—C7—N1—S1	-67.8 (3)
C2—C1—C6—C5	-2.2 (5)	C7—N1—S1—O2	55.5 (2)
S1—C1—C6—C5	176.8 (3)	C7—N1—S1—O1	-176.22 (17)
C4—C5—C6—C1	2.1 (8)	C7—N1—S1—C1	-60.1 (2)
C12—C7—C8—C9	-0.5 (3)	C6—C1—S1—O2	-13.9 (3)
N1—C7—C8—C9	176.1 (2)	C2—C1—S1—O2	165.1 (2)
C7—C8—C9—C10	1.2 (3)	C6—C1—S1—O1	-144.3 (3)
C7—C8—C9—C11	-178.94 (17)	C2—C1—S1—O1	34.8 (2)

C8—C9—C10—C11	-1.0 (4)	C6—C1—S1—N1	102.2 (3)
C11—C9—C10—C11	179.2 (2)	C2—C1—S1—N1	-78.7 (2)
C9—C10—C11—C12	0.0 (4)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1N...O2 ⁱ	0.88 (1)	2.03 (1)	2.875 (2)	162 (2)

Symmetry code: (i) $x-1/2, -y+1/2, -z+1/4$.