

2-Chloro-*N*-(4-methylbenzoyl)benzene-sulfonamide

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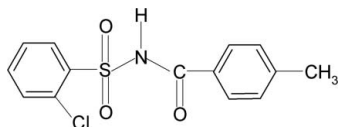
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.058; wR factor = 0.153; data-to-parameter ratio = 16.4.

In the title compound, $\text{C}_{14}\text{H}_{12}\text{ClNO}_3\text{S}$, the conformation of the $\text{N}-\text{H}$ bond in the $\text{C}-\text{SO}_2-\text{NH}-\text{C}(\text{O})$ segment is *anti* to the $\text{C}=\text{O}$ bond. The dihedral angle between the sulfonyl benzene ring and the $-\text{SO}_2-\text{NH}-\text{C}-\text{O}$ segment is 89.4 (1)° and that between the sulfonyl and benzoyl benzene rings is 89.1 (2)°. The crystal structure features inversion-related dimers linked by pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For background to our study of the effect of ring and side-chain substituents on the crystal structures of *N*-aromatic sulfonamides and for similar structures, see: Gowda *et al.* (2010*a,b*); Suchetan *et al.* (2010*a,b*).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{12}\text{ClNO}_3\text{S}$
 $M_r = 309.76$
Monoclinic, $P2_1/n$
 $a = 8.0554$ (8) Å
 $b = 23.209$ (2) Å

$c = 8.1199$ (9) Å
 $\beta = 103.52$ (1)°
 $V = 1476.0$ (3) Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.41$ mm⁻¹
 $T = 299$ K

$0.40 \times 0.30 \times 0.25$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford)

Diffraction, 2009)
 $T_{\min} = 0.855$, $T_{\max} = 0.906$
6106 measured reflections
3012 independent reflections
2396 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.012$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.153$
 $S = 1.07$
3012 reflections
184 parameters
19 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.43$ e Å⁻³
 $\Delta\rho_{\min} = -0.54$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{O2}^i$	0.84 (2)	2.14 (2)	2.970 (4)	169 (4)

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2212).

References

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supplementary materials

Acta Cryst. (2010). E66, o1466 [doi:10.1107/S1600536810018908]

2-Chloro-*N*-(4-methylbenzoyl)benzenesulfonamide

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Comment

Diaryl acylsulfonamides are known as potent antitumor agents against a broad spectrum of human tumor xenografts in nude mice. As a part of studying the effect of ring and the side chain substituents on the crystal structures of *N*-aromatic sulfonamides (Gowda *et al.*, 2010*a,b*; Suchetan *et al.*, 2010*a,b*), the structure of 2-chloro-*N*-(4-methylbenzoyl)benzenesulfonamide (I) has been determined (Fig. 1). The conformations of the N—C bonds in the C—SO₂—NH—C(O) segments have *gauche* torsions with respect to the SO bonds. Further, the conformation of the N—H bond in the C—SO₂—NH—C(O) segment is *anti* to the C=O bond, similar to that observed in 2-methyl-*N*-(4-methylbenzoyl)-benzenesulfonamide (II) (Gowda *et al.*, 2010*a*), 2-chloro-*N*-(3-methylbenzoyl)-benzenesulfonamide (III) (Suchetan *et al.*, 2010*b*), 2-chloro-*N*-(2-methylbenzoyl)-benzenesulfonamide (IV) (Suchetan *et al.*, 2010*a*) and 2-chloro-*N*-(benzoyl)-benzenesulfonamide (V) (Gowda *et al.*, 2010*b*).

The molecules are twisted at the *S* atom with the torsional angle of 60.4 (3)°, compared to those of -53.1 (2)° and 61.2 (2)°, in the two molecules of (II), -66.5 (2)° in (III), -64.0 (2)° in (IV) and 66.7 (2)° in (V). The dihedral angle between the sulfonyl benzene ring and the —SO₂—NH—C—O segment is 89.4 (1)°, compared to the values of 86.0 (1)° and 87.9 (1)° in the 2 molecules of (II), 88.4 (1)° in (III), 84.8 (1)° in (IV) and 87.3 (1)° in (V).

Furthermore, the dihedral angle between the sulfonyl and the benzoyl benzene ring is 89.1 (2)°, compared to the values of 88.1 (1)° (molecule 1) and 83.5 (1)° (molecule 2) of (II), 74.7 (1)° in (III), 78.7 (1)° in (IV) and 73.3 (1)° in (V).

The packing of molecules linked by of N—H···O(S) hydrogen bonds (Table 1) is shown in Fig. 2.

Experimental

The title compound was prepared by refluxing a mixture of 4-methylbenzoic acid, 2-chlorobenzenesulfonamide and phosphorous oxy chloride for 5 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid, 2-chloro-*N*-(4-methylbenzoyl)benzenesulfonamide obtained was filtered, washed thoroughly with water and then dissolved in sodium bicarbonate solution. The compound was later reprecipitated by acidifying the filtered solution with dilute HCl. The filtered and dried compound was recrystallized to the constant melting point.

Prism like colorless single crystals of the title compound used in X-ray diffraction studies were grown from a slow evaporation of its toluene solution at room temperature.

Refinement

The H atoms of the NH groups were located in a difference map and later restrained to N—H = 0.86 (2) %A. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.96 Å. All H atoms were refined

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with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom). The U^{ij} components of C4, C9 and C10 atoms were restrained to approximate isotropic behavior.

Figures

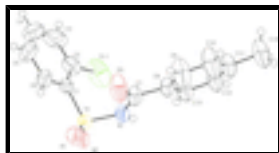


Fig. 1. Molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

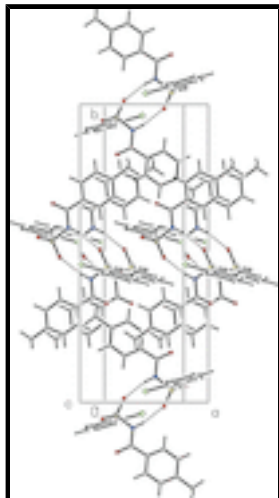


Fig. 2. Molecular packing in the title compound. Hydrogen bonds are shown as dashed lines.

2-Chloro-*N*-(4-methylbenzoyl)benzenesulfonamide

Crystal data

$C_{14}H_{12}ClNO_3S$

$M_r = 309.76$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 8.0554\ (8)\ \text{\AA}$

$b = 23.209\ (2)\ \text{\AA}$

$c = 8.1199\ (9)\ \text{\AA}$

$\beta = 103.52\ (1)^\circ$

$V = 1476.0\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 640$

$D_x = 1.394\ \text{Mg m}^{-3}$

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

Cell parameters from 2713 reflections

$\theta = 2.6\text{--}27.7^\circ$

$\mu = 0.41\ \text{mm}^{-1}$

$T = 299\ \text{K}$

Prism, colourless

$0.40 \times 0.30 \times 0.25\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur
diffractometer with a Sapphire CCD detector

Radiation source: fine-focus sealed tube
graphite

3012 independent reflections

2396 reflections with $I > 2\sigma(I)$

$R_{int} = 0.012$

Rotation method data acquisition using ω and phi scans $\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.7^\circ$
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009) $h = -10 \rightarrow 7$
 $T_{\min} = 0.855$, $T_{\max} = 0.906$ $k = -29 \rightarrow 20$
 6106 measured reflections $l = -7 \rightarrow 10$

Refinement

Refinement on F^2 Primary atom site location: structure-invariant direct methods
 Least-squares matrix: full Secondary atom site location: difference Fourier map
 $R[F^2 > 2\sigma(F^2)] = 0.058$ Hydrogen site location: inferred from neighbouring sites
 $wR(F^2) = 0.153$ H atoms treated by a mixture of independent and constrained refinement
 $S = 1.07$ $w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 1.5332P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 3012 reflections $(\Delta/\sigma)_{\max} = 0.009$
 184 parameters $\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$
 19 restraints $\Delta\rho_{\min} = -0.54 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. *CrysAlis RED* (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	0.8834 (4)	0.06157 (12)	0.7360 (4)	0.0399 (6)
C2	0.8115 (5)	0.05804 (15)	0.8744 (5)	0.0609 (9)
C3	0.9088 (9)	0.0714 (2)	1.0333 (6)	0.1017 (18)
H3	0.8610	0.0693	1.1268	0.122*
C4	1.0737 (10)	0.0875 (3)	1.0534 (7)	0.112 (2)
H4	1.1389	0.0957	1.1614	0.135*
C5	1.1463 (6)	0.0921 (2)	0.9173 (7)	0.0901 (15)
H5	1.2589	0.1042	0.9325	0.108*
C6	1.0509 (4)	0.07867 (14)	0.7569 (5)	0.0564 (8)

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H6	1.0995	0.0812	0.6639	0.068*
C7	0.6201 (4)	0.14562 (15)	0.4825 (5)	0.0553 (8)
C8	0.4601 (4)	0.17935 (14)	0.4640 (5)	0.0525 (8)
C9	0.4691 (6)	0.23742 (19)	0.4380 (8)	0.1039 (18)
H9	0.5712	0.2538	0.4266	0.125*
C10	0.3274 (6)	0.27170 (19)	0.4289 (9)	0.113 (2)
H10	0.3359	0.3110	0.4108	0.136*
C11	0.1739 (5)	0.24948 (16)	0.4456 (6)	0.0738 (12)
C12	0.1647 (4)	0.19155 (15)	0.4642 (6)	0.0667 (10)
H12	0.0615	0.1750	0.4714	0.080*
C13	0.3054 (4)	0.15666 (14)	0.4729 (5)	0.0581 (9)
H13	0.2950	0.1171	0.4849	0.070*
C14	0.0207 (6)	0.2878 (2)	0.4412 (8)	0.1062 (18)
H14A	-0.0105	0.3071	0.3338	0.127*
H14B	0.0489	0.3159	0.5302	0.127*
H14C	-0.0733	0.2648	0.4570	0.127*
N1	0.6050 (3)	0.08618 (12)	0.4850 (4)	0.0524 (7)
H1N	0.511 (3)	0.0699 (15)	0.482 (5)	0.063*
O1	0.8805 (3)	0.05469 (13)	0.4188 (3)	0.0701 (7)
O2	0.7012 (3)	-0.01317 (10)	0.5312 (4)	0.0727 (8)
O3	0.7585 (3)	0.16727 (12)	0.4989 (5)	0.0902 (10)
Cl1	0.60112 (17)	0.03731 (6)	0.85557 (19)	0.1056 (5)
S1	0.77166 (9)	0.04311 (3)	0.52941 (10)	0.0470 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0441 (15)	0.0354 (14)	0.0400 (15)	0.0042 (12)	0.0098 (12)	0.0020 (12)
C2	0.083 (2)	0.0542 (19)	0.053 (2)	0.0150 (18)	0.0314 (18)	0.0112 (16)
C3	0.165 (6)	0.097 (4)	0.047 (2)	0.046 (4)	0.034 (3)	0.008 (2)
C4	0.140 (5)	0.105 (4)	0.067 (3)	0.035 (4)	-0.027 (3)	-0.023 (3)
C5	0.072 (3)	0.077 (3)	0.098 (4)	-0.001 (2)	-0.027 (3)	-0.013 (3)
C6	0.0454 (17)	0.0522 (19)	0.067 (2)	-0.0015 (14)	0.0028 (15)	0.0024 (16)
C7	0.0408 (17)	0.0527 (19)	0.072 (2)	0.0017 (14)	0.0127 (15)	0.0123 (16)
C8	0.0416 (16)	0.0453 (17)	0.070 (2)	0.0008 (13)	0.0113 (15)	0.0130 (15)
C9	0.062 (2)	0.058 (2)	0.194 (5)	0.0004 (19)	0.035 (3)	0.035 (3)
C10	0.079 (3)	0.049 (2)	0.214 (6)	0.010 (2)	0.040 (3)	0.038 (3)
C11	0.056 (2)	0.048 (2)	0.117 (4)	0.0122 (16)	0.019 (2)	0.020 (2)
C12	0.0449 (17)	0.050 (2)	0.107 (3)	0.0012 (15)	0.0212 (19)	0.007 (2)
C13	0.0466 (17)	0.0397 (17)	0.089 (3)	-0.0005 (14)	0.0184 (17)	0.0054 (16)
C14	0.079 (3)	0.070 (3)	0.174 (6)	0.030 (2)	0.037 (3)	0.027 (3)
N1	0.0356 (13)	0.0464 (15)	0.0708 (18)	0.0023 (11)	0.0038 (12)	-0.0026 (13)
O1	0.0647 (15)	0.106 (2)	0.0433 (13)	0.0244 (14)	0.0207 (11)	0.0014 (13)
O2	0.0460 (13)	0.0452 (14)	0.118 (2)	0.0032 (10)	0.0014 (13)	-0.0245 (14)
O3	0.0441 (14)	0.0663 (17)	0.163 (3)	-0.0014 (12)	0.0291 (16)	0.0272 (18)
Cl1	0.1019 (9)	0.1066 (9)	0.1379 (12)	0.0130 (7)	0.0877 (9)	0.0378 (8)
S1	0.0388 (4)	0.0499 (4)	0.0500 (4)	0.0057 (3)	0.0059 (3)	-0.0087 (3)

Geometric parameters (Å, °)

C1—C6	1.378 (4)	C9—C10	1.379 (6)
C1—C2	1.381 (4)	C9—H9	0.9300
C1—S1	1.761 (3)	C10—C11	1.376 (6)
C2—C3	1.379 (7)	C10—H10	0.9300
C2—C11	1.734 (4)	C11—C12	1.357 (5)
C3—C4	1.353 (8)	C11—C14	1.515 (5)
C3—H3	0.9300	C12—C13	1.382 (5)
C4—C5	1.370 (8)	C12—H12	0.9300
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.384 (6)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C6—H6	0.9300	C14—H14C	0.9600
C7—O3	1.201 (4)	N1—S1	1.645 (3)
C7—N1	1.385 (4)	N1—H1N	0.840 (19)
C7—C8	1.485 (4)	O1—S1	1.420 (3)
C8—C9	1.369 (5)	O2—S1	1.426 (3)
C8—C13	1.370 (4)		
C6—C1—C2	120.1 (3)	C11—C10—C9	122.0 (4)
C6—C1—S1	117.1 (2)	C11—C10—H10	119.0
C2—C1—S1	122.8 (3)	C9—C10—H10	119.0
C3—C2—C1	119.5 (4)	C12—C11—C10	117.1 (4)
C3—C2—C11	118.3 (4)	C12—C11—C14	121.3 (4)
C1—C2—C11	122.1 (3)	C10—C11—C14	121.6 (4)
C4—C3—C2	120.2 (5)	C11—C12—C13	121.4 (3)
C4—C3—H3	119.9	C11—C12—H12	119.3
C2—C3—H3	119.9	C13—C12—H12	119.3
C3—C4—C5	121.1 (5)	C8—C13—C12	121.2 (3)
C3—C4—H4	119.5	C8—C13—H13	119.4
C5—C4—H4	119.5	C12—C13—H13	119.4
C4—C5—C6	119.6 (5)	C11—C14—H14A	109.5
C4—C5—H5	120.2	C11—C14—H14B	109.5
C6—C5—H5	120.2	H14A—C14—H14B	109.5
C1—C6—C5	119.5 (4)	C11—C14—H14C	109.5
C1—C6—H6	120.2	H14A—C14—H14C	109.5
C5—C6—H6	120.2	H14B—C14—H14C	109.5
O3—C7—N1	119.8 (3)	C7—N1—S1	122.6 (2)
O3—C7—C8	123.5 (3)	C7—N1—H1N	122 (3)
N1—C7—C8	116.7 (3)	S1—N1—H1N	115 (3)
C9—C8—C13	117.9 (3)	O1—S1—O2	119.06 (17)
C9—C8—C7	117.3 (3)	O1—S1—N1	109.85 (16)
C13—C8—C7	124.8 (3)	O2—S1—N1	104.63 (14)
C8—C9—C10	120.2 (4)	O1—S1—C1	107.63 (15)
C8—C9—H9	119.9	O2—S1—C1	109.23 (16)
C10—C9—H9	119.9	N1—S1—C1	105.67 (14)
C6—C1—C2—C3	-0.4 (5)	C9—C10—C11—C12	-2.8 (9)
S1—C1—C2—C3	177.8 (3)	C9—C10—C11—C14	177.9 (6)

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C6—C1—C2—C11	179.5 (3)	C10—C11—C12—C13	2.5 (7)
S1—C1—C2—C11	-2.3 (4)	C14—C11—C12—C13	-178.2 (5)
C1—C2—C3—C4	-0.3 (7)	C9—C8—C13—C12	-3.1 (6)
C11—C2—C3—C4	179.8 (4)	C7—C8—C13—C12	176.1 (4)
C2—C3—C4—C5	1.3 (8)	C11—C12—C13—C8	0.4 (7)
C3—C4—C5—C6	-1.6 (8)	O3—C7—N1—S1	6.5 (5)
C2—C1—C6—C5	0.1 (5)	C8—C7—N1—S1	-171.8 (3)
S1—C1—C6—C5	-178.2 (3)	C7—N1—S1—O1	-55.4 (3)
C4—C5—C6—C1	0.9 (6)	C7—N1—S1—O2	175.7 (3)
O3—C7—C8—C9	10.5 (6)	C7—N1—S1—C1	60.4 (3)
N1—C7—C8—C9	-171.3 (4)	C6—C1—S1—O1	-4.7 (3)
O3—C7—C8—C13	-168.8 (4)	C2—C1—S1—O1	177.0 (3)
N1—C7—C8—C13	9.5 (5)	C6—C1—S1—O2	125.9 (2)
C13—C8—C9—C10	2.8 (8)	C2—C1—S1—O2	-52.4 (3)
C7—C8—C9—C10	-176.5 (5)	C6—C1—S1—N1	-122.0 (2)
C8—C9—C10—C11	0.2 (10)	C2—C1—S1—N1	59.7 (3)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1N\cdots O2^i$	0.84 (2)	2.14 (2)	2.970 (4)	169 (4)

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

Fig. 1

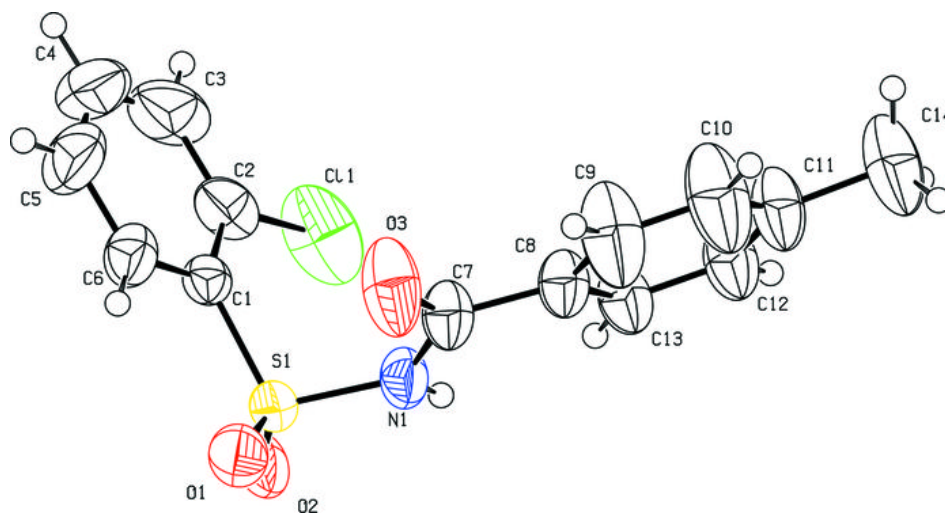


Fig. 2

