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4-Chloro-*N*-(4-chlorobenzoyl)benzenesulfonamide

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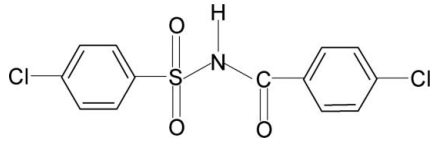
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.102; data-to-parameter ratio = 13.9.

In the title compound, $C_{13}H_9Cl_2NO_3S$, the conformation of the N—H bond in the C—SO_2—NH—C(O) segment is *anti* to the C=O bond. The molecule is twisted at the S atom with a torsion angle of 67.5 (3)°. The dihedral angle between the sulfonyl benzene ring and the —SO_2—NH—C—O segment is 79.0 (1)° and that between the sulfonyl and benzoyl benzene rings is 85.6 (1)°. In the crystal, molecules are linked by N—H···O(S) hydrogen bonds with graph-set descriptor $C(4)$ along the [010] direction.

Related literature

For background literature and related structures, see: Gowda *et al.* (2009); Suchetan *et al.* (2009, 2010*a*,*b*). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{13}H_9Cl_2NO_3S$
M_r = 330.17
Orthorhombic, *Pbca*
a = 13.6405 (9) Å
b = 9.6495 (8) Å
c = 21.116 (2) Å
V = 2779.4 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.62 mm^{-1}
T = 299 K
0.34 × 0.30 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.816, T_{max} = 0.886
10651 measured reflections
2550 independent reflections
1946 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.102
S = 1.07
2550 reflections
184 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 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—H1*N*···O3^{i}	0.85 (1)	2.23 (1)	3.074 (3)	172 (3)

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, z$.

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: BX2279).

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supporting information

Acta Cryst. (2010). E**66**, o1253 [https://doi.org/10.1107/S160053681001559X]

4-Chloro-*N*-(4-chlorobenzoyl)benzenesulfonamide

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S1. Comment

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.*, 2009; Suchetan *et al.*,2009, 2010*a,b*), the structure of *N*-(4-chlorobenzoyl)-4-chlorobenzene-sulfonamide (I) has been determined. The conformation of the N—H bond in the C—SO_2—NH—C(O) segment is *anti* to the C=O bond (Fig.1), similar to those observed in *N*-(benzoyl)benzenesulfonamide (II) (Gowda *et al.*, 2009), *N*-(4-chlorobenzoyl)-benzenesulfonamide (III)(Suchetan *et al.*, 2009), *N*-(benzoyl)-4-chlorobenzenesulfonamide (IV) (Suchetan *et al.*, 2010*a*) and *N*-(2-chlorobenzoyl)- 2-chlorobenzenesulfonamide (V)(Suchetan *et al.*, 2010*b*).

The molecules are twisted at the *S* atoms with the torsional angles of 67.5 (3)°, compared to the values of -66.9 (3)° in (II), 69.4 (2)° in (III), -70.0 (2)°, 61.3 (2)° in the two independent molecules of (IV) and 66.5 (2)° in (V).

The dihedral angle between the sulfonyl and the benzoyl benzene rings is 85.6 (1)°, compared to the vlues of 80.3 (1) in (II), 68.6 (1)° in (III), 62.8 (1)° (molecule 1) and 78.6 (1)° (molecule 2) of (IV) and 76.9 (1)° in (V).

The molecules are linked by of N—H···O(S) hydrogen bonds with graph-set descriptor C(4) along [010] direction, (Bernstein *et al.*, 1995) (Table 1), Fig. 2.

S2. Experimental

The title compound was prepared by refluxing a mixture of 4-chlorobenzoic acid, 4-chlorobenzenesulfonamide and phosphorous oxy chloride for 3 h on a water bath. The resultant mixture was cooled and poured into ice cold water. The solid 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. It was filtered, dried and recrystallized.

Prism like colourless single crystals of the title compound used in X-ray diffraction studies were obtained by slow evaporation of its toluene solution at room temperature.

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to N—H = 0.86 (1) %A. The other H atoms were positioned with idealized geometry using a riding model with C—H = 0.93 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

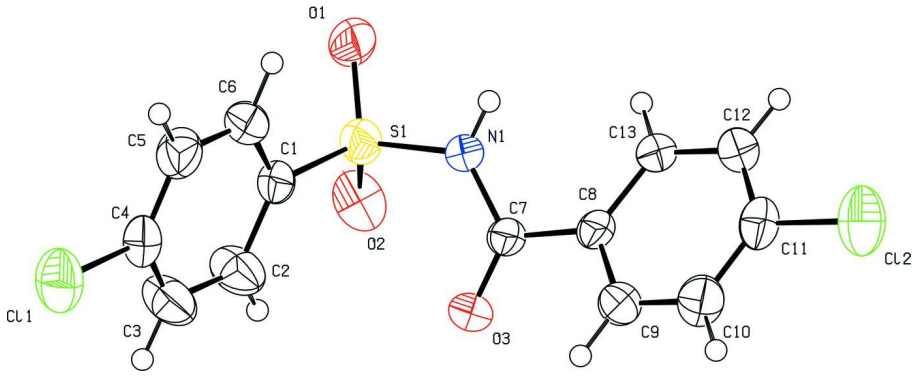

Figure 1

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

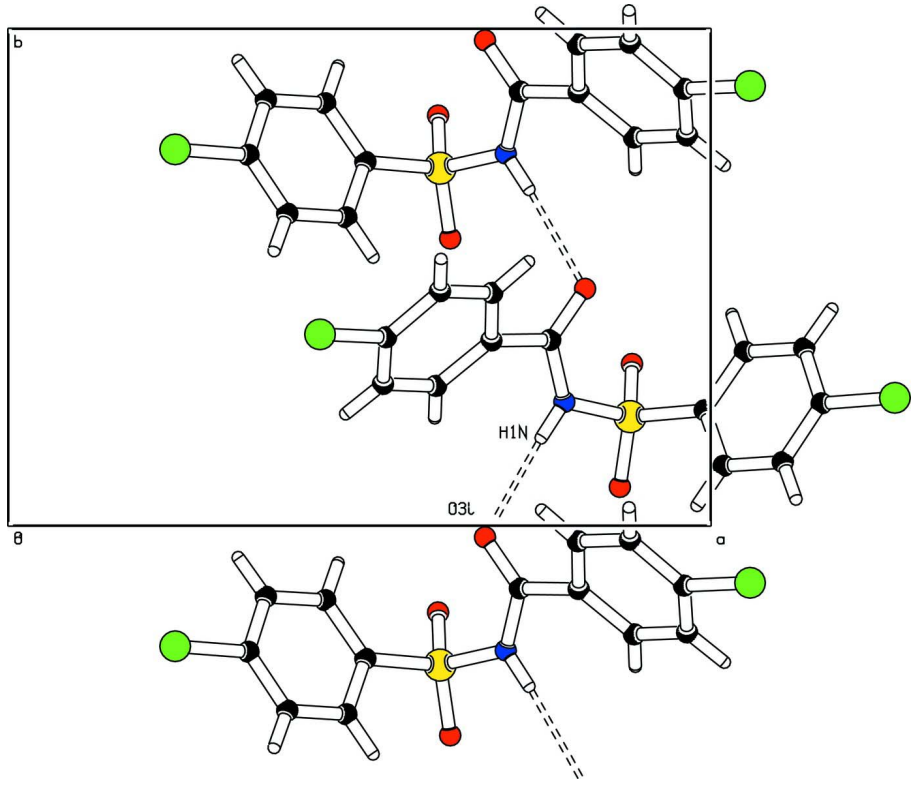

Figure 2

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

4-Chloro-*N*-(4-chlorobenzoyl)benzenesulfonamide

Crystal data

$C_{13}H_9Cl_2NO_3S$
$M_r = 330.17$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 13.6405$ (9) Å
$b = 9.6495$ (8) Å
$c = 21.116$ (2) Å
$V = 2779.4$ (4) Å^3
$Z = 8$
$F(000) = 1344$
$D_x = 1.578$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6170 reflections
θ = 2.6–27.9°
μ = 0.62 mm^{-1}
T = 299 K
Prism, colourless
0.34 × 0.30 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω and phi scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.816, T_{max} = 0.886
10651 measured reflections
2550 independent reflections
1946 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.3°, θ_{min} = 2.8°
h = −15→16
k = −11→8
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.102
S = 1.07
2550 reflections
184 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 2.7251P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.99158 (19)	0.2323 (3)	0.55735 (12)	0.0379 (6)
C2	1.0488 (2)	0.3501 (3)	0.55627 (16)	0.0589 (9)
H2	1.0305	0.4251	0.5313	0.071*
C3	1.1332 (2)	0.3571 (3)	0.59208 (17)	0.0623 (9)
H3	1.1726	0.4358	0.5911	0.075*
C4	1.15804 (19)	0.2462 (3)	0.62912 (14)	0.0453 (7)
C5	1.1027 (2)	0.1281 (3)	0.63045 (15)	0.0532 (8)
H5	1.1214	0.0536	0.6556	0.064*
C6	1.0187 (2)	0.1205 (3)	0.59412 (14)	0.0478 (7)

H6	0.9806	0.0406	0.5944	0.057*
C7	0.77151 (18)	0.3738 (2)	0.58811 (13)	0.0358 (6)
C8	0.68782 (18)	0.3726 (2)	0.63338 (13)	0.0361 (6)
C9	0.6890 (2)	0.4687 (3)	0.68266 (14)	0.0450 (7)
H9	0.7406	0.5313	0.6858	0.054*
C10	0.6147 (2)	0.4719 (3)	0.72667 (14)	0.0504 (7)
H10	0.6162	0.5358	0.7596	0.061*
C11	0.5378 (2)	0.3794 (3)	0.72143 (14)	0.0464 (7)
C12	0.5335 (2)	0.2852 (3)	0.67218 (15)	0.0482 (7)
H12	0.4805	0.2252	0.6684	0.058*
C13	0.60898 (18)	0.2814 (3)	0.62879 (14)	0.0431 (7)
H13	0.6072	0.2171	0.5960	0.052*
N1	0.79169 (16)	0.2491 (2)	0.55934 (11)	0.0395 (5)
H1N	0.7586 (18)	0.1768 (19)	0.5681 (13)	0.047*
O1	0.87074 (15)	0.0780 (2)	0.49305 (11)	0.0610 (6)
O2	0.88803 (15)	0.3255 (2)	0.46352 (9)	0.0595 (6)
O3	0.82124 (13)	0.47670 (18)	0.57873 (10)	0.0485 (5)
Cl1	1.26227 (6)	0.25736 (10)	0.67639 (4)	0.0679 (3)
Cl2	0.44389 (6)	0.38508 (11)	0.77714 (4)	0.0710 (3)
S1	0.88532 (5)	0.21948 (7)	0.51034 (3)	0.0437 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0323 (13)	0.0400 (15)	0.0415 (15)	0.0010 (11)	0.0068 (12)	−0.0043 (13)
C2	0.0546 (19)	0.0498 (18)	0.072 (2)	−0.0097 (15)	−0.0159 (17)	0.0228 (16)
C3	0.0489 (18)	0.0569 (19)	0.081 (2)	−0.0157 (15)	−0.0158 (17)	0.0160 (18)
C4	0.0324 (13)	0.0561 (17)	0.0474 (16)	0.0058 (13)	0.0016 (13)	−0.0011 (14)
C5	0.0475 (17)	0.0484 (17)	0.064 (2)	0.0084 (14)	0.0035 (15)	0.0128 (15)
C6	0.0418 (16)	0.0391 (15)	0.0624 (19)	−0.0035 (13)	0.0076 (14)	0.0040 (14)
C7	0.0299 (13)	0.0294 (13)	0.0482 (16)	0.0023 (11)	−0.0065 (11)	−0.0014 (12)
C8	0.0308 (13)	0.0295 (13)	0.0480 (16)	0.0057 (10)	−0.0052 (12)	−0.0011 (12)
C9	0.0364 (15)	0.0432 (15)	0.0555 (18)	−0.0008 (13)	−0.0073 (13)	−0.0093 (14)
C10	0.0463 (17)	0.0595 (19)	0.0454 (17)	0.0041 (15)	−0.0051 (14)	−0.0129 (15)
C11	0.0368 (15)	0.0548 (18)	0.0475 (17)	0.0106 (13)	0.0015 (13)	0.0041 (15)
C12	0.0360 (15)	0.0419 (16)	0.067 (2)	−0.0017 (12)	0.0044 (14)	−0.0003 (15)
C13	0.0357 (14)	0.0327 (14)	0.0608 (18)	0.0006 (12)	0.0007 (14)	−0.0091 (13)
N1	0.0327 (12)	0.0294 (11)	0.0564 (15)	−0.0013 (9)	0.0065 (11)	−0.0053 (11)
O1	0.0491 (12)	0.0582 (13)	0.0758 (15)	0.0005 (10)	0.0050 (11)	−0.0327 (12)
O2	0.0550 (13)	0.0814 (15)	0.0421 (11)	−0.0084 (12)	−0.0030 (10)	0.0079 (11)
O3	0.0399 (10)	0.0333 (10)	0.0721 (14)	−0.0061 (9)	0.0041 (10)	−0.0047 (10)
Cl1	0.0442 (4)	0.0885 (6)	0.0709 (6)	0.0080 (4)	−0.0124 (4)	0.0004 (5)
Cl2	0.0533 (5)	0.1024 (7)	0.0574 (5)	0.0095 (5)	0.0137 (4)	0.0039 (5)
S1	0.0380 (4)	0.0479 (4)	0.0452 (4)	−0.0015 (3)	0.0034 (3)	−0.0091 (3)

Geometric parameters (Å, º)

C1—C2	1.379 (4)	C8—C13	1.393 (3)
C1—C6	1.380 (4)	C8—C9	1.394 (4)
C1—S1	1.761 (3)	C9—C10	1.376 (4)
C2—C3	1.379 (4)	C9—H9	0.9300
C2—H2	0.9300	C10—C11	1.381 (4)
C3—C4	1.368 (4)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.383 (4)
C4—C5	1.367 (4)	C11—Cl2	1.740 (3)
C4—Cl1	1.740 (3)	C12—C13	1.379 (4)
C5—C6	1.380 (4)	C12—H12	0.9300
C5—H5	0.9300	C13—H13	0.9300
C6—H6	0.9300	N1—S1	1.668 (2)
C7—O3	1.219 (3)	N1—H1N	0.851 (10)
C7—N1	1.376 (3)	O1—S1	1.427 (2)
C7—C8	1.489 (4)	O2—S1	1.423 (2)
C2—C1—C6	120.1 (3)	C10—C9—C8	120.7 (3)
C2—C1—S1	121.0 (2)	C10—C9—H9	119.7
C6—C1—S1	118.9 (2)	C8—C9—H9	119.6
C3—C2—C1	120.3 (3)	C9—C10—C11	119.4 (3)
C3—C2—H2	119.9	C9—C10—H10	120.3
C1—C2—H2	119.9	C11—C10—H10	120.3
C4—C3—C2	118.8 (3)	C12—C11—C10	121.1 (3)
C4—C3—H3	120.6	C12—C11—Cl2	119.9 (2)
C2—C3—H3	120.6	C10—C11—Cl2	119.0 (2)
C3—C4—C5	121.8 (3)	C13—C12—C11	119.1 (3)
C3—C4—Cl1	118.8 (2)	C13—C12—H12	120.5
C5—C4—Cl1	119.4 (2)	C11—C12—H12	120.5
C4—C5—C6	119.4 (3)	C12—C13—C8	120.9 (3)
C4—C5—H5	120.3	C12—C13—H13	119.6
C6—C5—H5	120.3	C8—C13—H13	119.6
C5—C6—C1	119.6 (3)	C7—N1—S1	125.21 (18)
C5—C6—H6	120.2	C7—N1—H1N	121 (2)
C1—C6—H6	120.2	S1—N1—H1N	113.7 (19)
O3—C7—N1	122.0 (2)	O2—S1—O1	120.90 (14)
O3—C7—C8	122.5 (2)	O2—S1—N1	109.13 (12)
N1—C7—C8	115.5 (2)	O1—S1—N1	102.45 (12)
C13—C8—C9	118.8 (3)	O2—S1—C1	108.63 (12)
C13—C8—C7	123.5 (2)	O1—S1—C1	109.05 (13)
C9—C8—C7	117.7 (2)	N1—S1—C1	105.57 (12)
C6—C1—C2—C3	−0.3 (5)	C9—C10—C11—Cl2	−179.7 (2)
S1—C1—C2—C3	−178.3 (3)	C10—C11—C12—C13	1.9 (4)
C1—C2—C3—C4	−0.9 (5)	Cl2—C11—C12—C13	−179.5 (2)
C2—C3—C4—C5	1.4 (5)	C11—C12—C13—C8	−1.3 (4)
C2—C3—C4—Cl1	−177.8 (3)	C9—C8—C13—C12	−0.3 (4)

C3—C4—C5—C6	−0.8 (5)	C7—C8—C13—C12	180.0 (2)
Cl1—C4—C5—C6	178.5 (2)	O3—C7—N1—S1	1.5 (4)
C4—C5—C6—C1	−0.4 (4)	C8—C7—N1—S1	−175.99 (18)
C2—C1—C6—C5	0.9 (4)	C7—N1—S1—O2	−49.1 (3)
S1—C1—C6—C5	179.0 (2)	C7—N1—S1—O1	−178.3 (2)
O3—C7—C8—C13	154.8 (3)	C7—N1—S1—C1	67.5 (2)
N1—C7—C8—C13	−27.7 (4)	C2—C1—S1—O2	15.4 (3)
O3—C7—C8—C9	−24.9 (4)	C6—C1—S1—O2	−162.6 (2)
N1—C7—C8—C9	152.5 (2)	C2—C1—S1—O1	149.0 (3)
C13—C8—C9—C10	1.2 (4)	C6—C1—S1—O1	−29.0 (3)
C7—C8—C9—C10	−179.1 (2)	C2—C1—S1—N1	−101.5 (3)
C8—C9—C10—C11	−0.5 (4)	C6—C1—S1—N1	80.5 (2)
C9—C10—C11—C12	−1.1 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O3^{i}	0.85 (1)	2.23 (1)	3.074 (3)	172 (3)

Symmetry code: (i) −*x*+3/2, *y*−1/2, *z*.