

4-[(*E*)-(5-Chloro-2-hydroxybenzylidene)-amino]benzenesulfonamideZahid H. Chohan,^a Hazoor A. Shad^a and M. Nawaz Tahir^{b*}^aDepartment of Chemistry, Bahauddin Zakariya University, Multan-60800, Pakistan, and ^bDepartment of Physics, University of Sargodha, Sargodha, Pakistan
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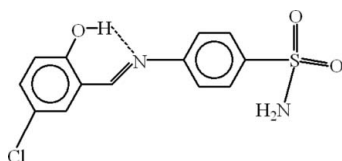
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.056; wR factor = 0.132; data-to-parameter ratio = 16.4.

In the molecule of title compound, $\text{C}_{13}\text{H}_{11}\text{ClN}_2\text{O}_3\text{S}$, the aromatic rings are oriented at a dihedral angle of $12.27(3)^\circ$. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond results in the formation of a planar (mean deviation 0.0083 Å) six-membered ring, which is nearly coplanar with the adjacent ring at a dihedral angle of $2.36(13)^\circ$. In the sulfonamide group, the S atom is $0.457(3)$ Å from the plane through the O and N atoms. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules.

Related literature

For general background, see: Chohan (2008); Chohan & Shad (2008); Chohan & Supuran (2008); Nishimori *et al.* (2005). For related structures, see: Chohan *et al.* (2008*a,b*); Shad *et al.* (2008); Gelbrich *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{11}\text{ClN}_2\text{O}_3\text{S}$
 $M_r = 310.76$
 Monoclinic, $P2_1$
 $a = 6.1936(9)$ Å
 $b = 4.6002(7)$ Å
 $c = 23.252(3)$ Å
 $\beta = 95.699(7)^\circ$

$V = 659.22(16)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.46$ mm⁻¹
 $T = 296(2)$ K
 $0.25 \times 0.18 \times 0.15$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.904$, $T_{\max} = 0.935$

7850 measured reflections
 3172 independent reflections
 1842 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.053$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.132$
 $S = 1.02$
 3172 reflections
 193 parameters
 4 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.36$ e Å⁻³
 Absolute structure: Flack (1983), 1125 Friedel pairs
 Flack parameter: 0.09 (13)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.87	2.603 (5)	148
$\text{N2}-\text{H21}\cdots\text{O3}^i$	0.87 (4)	2.16 (4)	2.986 (6)	160 (5)

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2593).

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supplementary materials

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4-[(*E*)-(5-Chloro-2-hydroxybenzylidene)amino]benzenesulfonamide

Z. H. Chohan, H. A. Shad and M. N. Tahir

Comment

Sulfonamides have gained much attention due to their extensive use in medicine. Many novel sulfonamide derived compounds have been synthesized and reported (Chohan, 2008; Chohan & Shad, 2008; Chohan & Supuran, 2008; Nishimori *et al.*, 2005) that are expected to attack the selective targets. This approach is supportive in controlling undesirable effects and producing distinctive pharmacological and clinical responses. In continuation to synthesize Schiff base ligands of 5-chlorosalicylaldehyde with different sulfonamides (Chohan *et al.*, 2008*a*, 2008*b*; Shad *et al.*, 2008), we have synthesized the title compound having the sulfanilamide, which is also a member of sulfonamides, and reported herein its crystal structure. The crystal structures of the individual moieties of δ -sulfanilamide have also been reported (Gelbrich *et al.*, 2008).

In the molecule of title compound (Fig 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C1-C6) and B (C8-C13) are, of course, planar, and the dihedral angle between them is A/B = 12.27 (3)°. The intramolecular O-H...N hydrogen bond (Table 1) results in the formation of a planar six-membered ring C (O1/N1/C1/C2/C7/H1), which is oriented with respect to rings A and B at dihedral angles of A/C = 2.36 (13)° and B/C = 13.22 (13)°. So, rings A and C are also nearly coplanar. In the sulfonamide group, the S1 atom is 0.457 (3) Å away from the plane of (O2/O3/N2).

In the crystal structure, intermolecular N-H...O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, sulfanilamide (344.4 mg, 2 mmol) in ethanol (20 ml) was mixed with 5-chlorosalicylaldehyde (313.1 mg, 2 mmol) in ethanol (10 ml). The resultant mixture was refluxed for 3 h by monitoring through TLC. During refluxing the solution turned from colorless to bright orange. After completion of reaction, it was cooled to room temperature, filtered and volume reduced to about one-third using rotary evaporator. It was then allowed to stand for 6 d at room temperature. After which, a crystallized product was formed that was filtered, washed with ethanol (2x5 ml), dried and recrystallized in a mixture of methanol/ethanol (1:1) to afford the orange crystals of the title compound (m.p. 469-471 K).

Refinement

H7 (for CH) and H21, H22 (for NH₂) atoms were located in difference syntheses and refined isotropically [C-H = 0.97 (4) Å, U_{iso}(H) = 0.040 (13) Å²; N-H = 0.87 (4) and 0.87 (5) Å; U_{iso}(H) = 0.07 (2) and 0.08 (2) Å²]. The remaining H atoms were positioned geometrically, with O-H = 0.82 Å (for OH) and C-H = 0.93 Å for aromatic H and constrained to ride on their parent atoms with U_{iso}(H) = xU_{eq}(C,O), where x = 1.5 for OH H and x = 1.2 for aromatic H atoms.

Figures

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bond is shown as dashed line.

Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

4-[(E)-(5-Chloro-2-hydroxybenzylidene)amino]benzenesulfonamide

Crystal data

C₁₃H₁₁ClN₂O₃S

M_r = 310.76

Monoclinic, *P*2₁

Hall symbol: P 2yb

a = 6.1936 (9) Å

b = 4.6002 (7) Å

c = 23.252 (3) Å

β = 95.699 (7)°

V = 659.22 (16) Å³

Z = 2

*F*₀₀₀ = 320

D_x = 1.566 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 1848 reflections

θ = 0.9–26.4°

μ = 0.46 mm⁻¹

T = 296 (2) K

Prism, orange

0.25 × 0.18 × 0.15 mm

Data collection

Bruker Kappa APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 7.6 pixels mm⁻¹

T = 296(2) K

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2005)

*T*_{min} = 0.904, *T*_{max} = 0.935

7850 measured reflections

3172 independent reflections

1842 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.053

θ_{max} = 28.5°

θ_{min} = 0.9°

h = -8→8

k = -6→6

l = -31→29

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.056

wR(*F*²) = 0.132

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2 + 0.3674P]$$

where *P* = (*F*_o² + 2*F*_c²)/3

(Δ/σ)_{max} = 0.001

$S = 1.02$	$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$
3172 reflections	$\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$
193 parameters	Extinction correction: none
4 restraints	Absolute structure: Flack (1983), 1125 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.09 (13)
Secondary atom site location: difference Fourier map	

Special details

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 $F^2 > 2\sigma(F^2)$ 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	0.76662 (18)	1.3639 (2)	0.57243 (4)	0.0351 (3)
Cl1	0.7848 (2)	-0.1726 (4)	0.96987 (6)	0.0659 (4)
O1	0.1496 (5)	0.3015 (9)	0.79117 (14)	0.0554 (10)
H1	0.2062	0.4241	0.7719	0.083*
O2	0.9594 (5)	1.5110 (7)	0.59629 (14)	0.0498 (9)
O3	0.5902 (5)	1.5306 (7)	0.54481 (14)	0.0472 (9)
N1	0.4614 (6)	0.6117 (9)	0.75542 (16)	0.0365 (9)
N2	0.8345 (8)	1.1435 (9)	0.52410 (19)	0.0431 (11)
H21	0.726 (6)	1.068 (12)	0.5029 (19)	0.07 (2)*
H22	0.929 (8)	1.022 (12)	0.541 (2)	0.08 (2)*
C1	0.5139 (7)	0.2913 (9)	0.83587 (19)	0.0344 (11)
C2	0.2983 (8)	0.1959 (11)	0.8319 (2)	0.0394 (12)
C3	0.2389 (8)	-0.0151 (12)	0.8696 (2)	0.0490 (13)
H3	0.0970	-0.0837	0.8659	0.059*
C4	0.3850 (8)	-0.1254 (15)	0.91213 (19)	0.0501 (12)
H4	0.3418	-0.2638	0.9378	0.060*
C5	0.5997 (8)	-0.0272 (11)	0.91650 (19)	0.0430 (12)
C6	0.6617 (8)	0.1767 (11)	0.8793 (2)	0.0420 (12)
H6	0.8048	0.2409	0.8827	0.050*
C7	0.5892 (8)	0.4971 (11)	0.7953 (2)	0.0391 (12)
H7	0.742 (7)	0.545 (10)	0.8017 (17)	0.040 (13)*
C8	0.5381 (7)	0.8040 (10)	0.71469 (17)	0.0335 (11)
C9	0.7445 (7)	0.9293 (10)	0.72084 (19)	0.0425 (13)
H9	0.8379	0.8935	0.7539	0.051*
C10	0.8097 (8)	1.1053 (10)	0.67816 (19)	0.0385 (12)

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H10	0.9476	1.1875	0.6824	0.046*
C11	0.6728 (7)	1.1609 (9)	0.62919 (18)	0.0312 (10)
C12	0.4655 (7)	1.0440 (11)	0.6241 (2)	0.0422 (12)
H12	0.3702	1.0855	0.5916	0.051*
C13	0.4006 (7)	0.8677 (14)	0.66654 (18)	0.0411 (11)
H13	0.2612	0.7903	0.6627	0.049*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0380 (7)	0.0288 (5)	0.0380 (6)	-0.0026 (6)	0.0009 (5)	-0.0002 (6)
C11	0.0711 (9)	0.0698 (10)	0.0545 (8)	0.0074 (9)	-0.0056 (7)	0.0153 (8)
O1	0.0333 (18)	0.065 (3)	0.066 (2)	-0.0079 (19)	-0.0044 (17)	0.012 (2)
O2	0.049 (2)	0.048 (2)	0.051 (2)	-0.0232 (18)	-0.0069 (17)	0.0015 (17)
O3	0.050 (2)	0.0369 (19)	0.053 (2)	0.0081 (17)	-0.0039 (17)	0.0064 (16)
N1	0.035 (2)	0.037 (2)	0.038 (2)	-0.0014 (18)	0.0037 (19)	0.0012 (18)
N2	0.047 (3)	0.040 (3)	0.043 (3)	-0.003 (2)	0.011 (2)	-0.010 (2)
C1	0.031 (3)	0.033 (3)	0.040 (3)	0.002 (2)	0.007 (2)	-0.003 (2)
C2	0.032 (3)	0.040 (3)	0.046 (3)	-0.002 (2)	0.007 (2)	-0.003 (2)
C3	0.042 (3)	0.052 (3)	0.056 (3)	-0.007 (3)	0.018 (3)	-0.002 (3)
C4	0.060 (3)	0.052 (3)	0.042 (3)	-0.006 (4)	0.021 (2)	0.000 (3)
C5	0.054 (3)	0.044 (3)	0.031 (3)	0.007 (3)	0.005 (2)	-0.001 (2)
C6	0.037 (3)	0.042 (3)	0.047 (3)	0.001 (2)	0.003 (2)	-0.004 (2)
C7	0.028 (3)	0.038 (3)	0.052 (3)	-0.004 (2)	0.005 (3)	-0.002 (2)
C8	0.033 (3)	0.035 (3)	0.034 (2)	0.003 (2)	0.008 (2)	-0.003 (2)
C9	0.034 (3)	0.054 (4)	0.038 (3)	-0.003 (2)	-0.006 (2)	0.007 (2)
C10	0.030 (3)	0.045 (3)	0.039 (3)	-0.006 (2)	-0.002 (2)	0.002 (2)
C11	0.028 (3)	0.030 (2)	0.036 (3)	0.000 (2)	0.001 (2)	-0.002 (2)
C12	0.029 (3)	0.049 (3)	0.046 (3)	0.000 (2)	-0.008 (2)	0.008 (2)
C13	0.024 (2)	0.049 (3)	0.050 (3)	-0.003 (3)	0.005 (2)	0.008 (3)

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

C11—C5	1.738 (5)	C6—C5	1.358 (7)
S1—O2	1.435 (3)	C6—H6	0.9300
S1—O3	1.434 (3)	C7—N1	1.272 (6)
S1—N2	1.600 (4)	C7—C1	1.446 (6)
S1—C11	1.762 (5)	C7—H7	0.97 (4)
O1—H1	0.8200	C8—N1	1.412 (5)
N2—H21	0.87 (4)	C9—C8	1.397 (6)
N2—H22	0.87 (5)	C9—C10	1.372 (6)
C2—O1	1.344 (5)	C9—H9	0.9300
C2—C3	1.381 (7)	C10—H10	0.9300
C2—C1	1.400 (6)	C11—C10	1.375 (6)
C3—C4	1.370 (7)	C11—C12	1.386 (6)
C3—H3	0.9300	C12—H12	0.9300
C4—H4	0.9300	C13—C8	1.370 (6)
C5—C4	1.398 (7)	C13—C12	1.369 (7)
C6—C1	1.397 (6)	C13—H13	0.9300

O2—S1—N2	107.7 (2)	C6—C5—C4	120.3 (5)
O2—S1—C11	106.48 (19)	C1—C6—H6	119.5
O3—S1—O2	119.3 (2)	C5—C6—C1	121.0 (5)
O3—S1—N2	105.4 (2)	C5—C6—H6	119.5
O3—S1—C11	109.0 (2)	N1—C7—C1	121.9 (4)
N2—S1—C11	108.6 (2)	N1—C7—H7	123 (3)
C2—O1—H1	109.5	C1—C7—H7	115 (3)
C7—N1—C8	121.4 (4)	C9—C8—N1	123.7 (4)
S1—N2—H21	114 (4)	C13—C8—C9	118.9 (4)
S1—N2—H22	108 (4)	C13—C8—N1	117.3 (4)
H21—N2—H22	117 (6)	C8—C9—H9	119.9
C2—C1—C7	122.0 (4)	C10—C9—C8	120.1 (4)
C6—C1—C2	118.8 (4)	C10—C9—H9	119.9
C6—C1—C7	119.2 (4)	C9—C10—C11	120.5 (4)
O1—C2—C1	121.0 (4)	C9—C10—H10	119.8
O1—C2—C3	119.6 (5)	C11—C10—H10	119.8
C3—C2—C1	119.4 (5)	C10—C11—C12	119.3 (4)
C2—C3—H3	119.3	C10—C11—S1	119.8 (4)
C4—C3—C2	121.3 (5)	C12—C11—S1	120.8 (3)
C4—C3—H3	119.3	C11—C12—H12	119.9
C3—C4—C5	119.2 (5)	C13—C12—C11	120.2 (4)
C3—C4—H4	120.4	C13—C12—H12	119.9
C5—C4—H4	120.4	C8—C13—H13	119.6
C4—C5—C11	118.9 (4)	C12—C13—C8	120.9 (4)
C6—C5—C11	120.9 (4)	C12—C13—H13	119.6
O2—S1—C11—C10	-17.2 (4)	C1—C6—C5—C4	-0.3 (7)
O2—S1—C11—C12	165.9 (4)	C1—C6—C5—C11	179.2 (4)
O3—S1—C11—C10	-147.1 (4)	N1—C7—C1—C6	-179.0 (5)
O3—S1—C11—C12	36.0 (4)	N1—C7—C1—C2	3.1 (7)
N2—S1—C11—C10	98.5 (4)	C1—C7—N1—C8	-177.6 (4)
N2—S1—C11—C12	-78.4 (4)	C9—C8—N1—C7	-13.3 (7)
O1—C2—C1—C6	179.5 (4)	C13—C8—N1—C7	166.4 (5)
O1—C2—C1—C7	-2.6 (7)	C10—C9—C8—N1	177.4 (4)
C3—C2—C1—C6	-2.1 (7)	C10—C9—C8—C13	-2.2 (7)
C3—C2—C1—C7	175.8 (4)	C8—C9—C10—C11	0.3 (7)
O1—C2—C3—C4	-179.1 (5)	S1—C11—C10—C9	-175.1 (4)
C1—C2—C3—C4	2.4 (8)	C12—C11—C10—C9	1.8 (7)
C2—C3—C4—C5	-1.6 (8)	S1—C11—C12—C13	174.8 (4)
C6—C5—C4—C3	0.5 (8)	C10—C11—C12—C13	-2.0 (7)
C11—C5—C4—C3	-179.0 (4)	C12—C13—C8—N1	-177.6 (5)
C5—C6—C1—C2	1.1 (7)	C12—C13—C8—C9	2.0 (8)
C5—C6—C1—C7	-176.9 (4)	C8—C13—C12—C11	0.1 (8)

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.82	1.87	2.603 (5)	148
N2—H21 \cdots O3 ⁱ	0.87 (4)	2.16 (4)	2.986 (6)	160 (5)

supplementary materials

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

Fig. 1

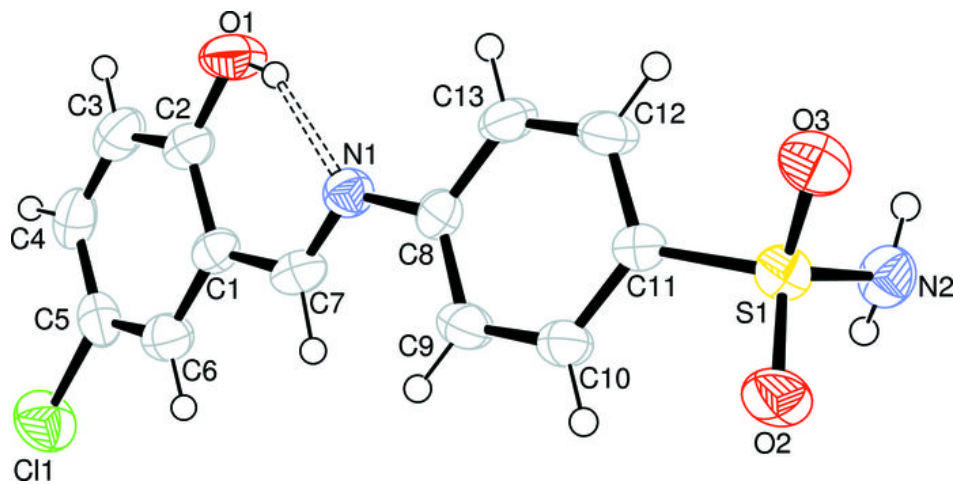


Fig. 2

