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4-Amino-3,5-dichlorobenzene-sulfonamide

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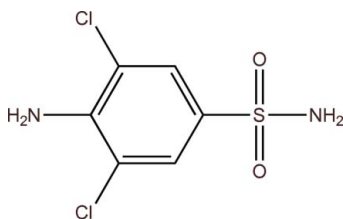
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Key indicators: single-crystal X-ray study; $T = 93$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.029; wR factor = 0.074; data-to-parameter ratio = 15.0.

In the title compound, $\text{C}_6\text{H}_6\text{Cl}_2\text{N}_2\text{O}_2\text{S}$, the O atoms of the sulfonamide group lie on one side of the benzene ring and the amino group lies on the opposite side. An intermolecular $\text{N}-\text{H}\cdots\text{Cl}$ interaction occurs. In the crystal, adjacent molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a three-dimensional structure with supporting $\pi-\pi$ stacking interactions [centroid-centroid distance = $3.7903(12)$ Å]. A short $\text{Cl}\cdots\text{Cl}$ contact [$3.3177(10)$ Å] also occurs.

Related literature

For the preparation, see: Qiu & Lv (2005). For $\text{Cl}\cdots\text{Cl}$ contacts, see: Sakurai *et al.* (1963); Stone *et al.* (1994); Qin *et al.* (2008).



Experimental

Crystal data

$\text{C}_6\text{H}_6\text{Cl}_2\text{N}_2\text{O}_2\text{S}$

$M_r = 241.09$

Monoclinic, $P2_1/c$

$a = 8.9544(17)$ Å

$b = 13.387(3)$ Å

$c = 7.5673(15)$ Å

$\beta = 95.809(2)^\circ$

$V = 902.4(3)$ Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.92$ mm⁻¹

$T = 93$ K

$0.30 \times 0.27 \times 0.13$ mm

Data collection

Rigaku SPIDER diffractometer

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.771$, $T_{\max} = 0.888$

5918 measured reflections

2008 independent reflections

1759 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.074$

$S = 1.00$

2008 reflections

134 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.45$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.36$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl1}$	0.80 (2)	2.60 (2)	2.9793 (18)	111 (2)
$\text{N1}-\text{H1B}\cdots\text{O2}^{\text{ii}}$	0.83 (3)	2.40 (3)	3.199 (2)	160 (2)
$\text{N2}-\text{H2A}\cdots\text{O1}^{\text{i}}$	0.81 (2)	2.14 (2)	2.935 (2)	167 (2)
$\text{N2}-\text{H2B}\cdots\text{O2}^{\text{iii}}$	0.86 (3)	2.08 (3)	2.934 (2)	172 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: NK2060).

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supplementary materials

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4-Amino-3,5-dichlorobenzenesulfonamide

X.-Y. Qin, H.-F. Liu and J.-X. Lin

Comment

4-Amino-3,5-dichloro-benzenesulfonamide is a primary substance used to synthesize Diclofenac sodium. Although the synthetic methods of the title compound has been reported (Qiu & Lv, 2005), no crystal structure data have been published so far. Our experimental aim was to obtain a Co(II) complex by preparing the potassium salt of 4-amino-3,5-dichloro-benzenesulfonamide modified by salicylaldehyde, and then reacting with $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$. To our surprise, we obtained yellow crystals which were identified as the title compound. Here, we report the synthesis and crystal structure of the title compound.

In the title compound, $\text{C}_6\text{H}_6\text{Cl}_2\text{N}_2\text{O}_2\text{S}$, the amino and chlorine substituent groups are essentially co-planar (maximum r.m.s. deviation of fitted atoms is -0.0747 \AA) with the benzene ring to which they are bonded. The O atoms of the sulfonamide group lie on one side of the benzene ring and the amino group lies on the opposite side (Scheme I, Fig. 1)

In the crystal, molecules are linked by intermolecular hydrogen bonds $\text{N}—\text{H} \cdots \text{O}$ (Fig.2., Table 2) and π – π stacking interactions [3.790 \AA , the distance of the centroids between the two benzene rings, symmetry code as $x, -y + 1/2, z + 1/2$ and $x, -y + 1/2, z - 1/2$], and a short $\text{Cl} \cdots \text{Cl}$ contact [3.318 \AA , symmetry code as $-x, -y - 1, -z$] occurs (Fig. 2). The $\text{Cl} \cdots \text{Cl}$ contact is stronger than that of the $[\text{Zn}(\text{II})(\text{C}_8\text{H}_7\text{O}_4\text{NCl}_2\text{S})(\text{Phen})(\text{H}_2\text{O})_3]$ (Qin *et al.*, 2008).

Experimental

Powdered 4-amino-3,5-dichloro-benzenesulfonamide (2.671 g, 10.08 mmol), KOH(0.504 g, 9.0 mmol) and salicylaldehyde (0.53 ml, 5.01 mmol) were mixed in methanol (40 ml). The mixture was stirred at 323 K for 2 h. Then, 1.30 ml solution was taken out from the mixed solution was added into a solution of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (0.060 g, 0.25 mmol) in methanol (25 ml). This mixture has been stirred at 323 K for 8 h. Subsequently, the reagents were filtrated. The resulting solution was left at room temperature, orange-yellow crystals were obtained after some days.

Refinement

All H atoms were geometrically positioned and refined using a mixed model, with distance restraints of $\text{C}—\text{H} = 0.9500$ and $\text{N}—\text{H} = 0.80$ (2)– 0.86 (3) \AA , and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

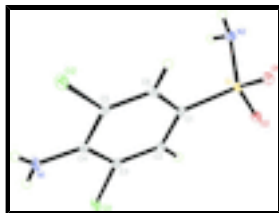


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids.

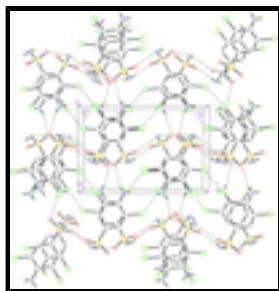


Fig. 2. The packing of (I), viewed down the *c* axis, showing layers of molecules conneted by N—H···O hydrogen bonds, Cl···Cl contact and π – π stacking interactions.

4-Amino-3,5-dichlorobenzenesulfonamide

Crystal data

$C_6H_6Cl_2N_2O_2S$

$M_r = 241.09$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.9544$ (17) Å

$b = 13.387$ (3) Å

$c = 7.5673$ (15) Å

$\beta = 95.809$ (2)°

$V = 902.4$ (3) Å³

$Z = 4$

$F(000) = 488$

$D_x = 1.774$ Mg m⁻³

Melting point = 207.2–207.5 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2008 reflections

$\theta = 3.0$ – 27.5 °

$\mu = 0.92$ mm⁻¹

$T = 93$ K

Block, orange-yellow

$0.30 \times 0.27 \times 0.13$ mm

Data collection

Rigaku SPIDER
diffractometer

Radiation source: rotating anode
graphite

ω scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.771$, $T_{\max} = 0.888$

5918 measured reflections

2008 independent reflections

1759 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.0$ °

$h = -10 \rightarrow 10$

$k = -17 \rightarrow 10$

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.074$

$S = 1.00$

2008 reflections

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

$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.260P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

134 parameters

$$\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$$

0 restraints

$$\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$$

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.24595 (5)	0.01031 (3)	0.45203 (6)	0.01881 (13)
Cl2	0.00000 (5)	0.37179 (3)	0.29728 (7)	0.02268 (13)
S1	0.57265 (5)	0.34844 (3)	0.58837 (6)	0.01325 (12)
O1	0.53529 (14)	0.43437 (9)	0.68889 (17)	0.0171 (3)
O2	0.67408 (13)	0.27471 (9)	0.67149 (18)	0.0190 (3)
N1	0.00496 (18)	0.15006 (13)	0.3074 (2)	0.0198 (4)
N2	0.64946 (18)	0.39096 (12)	0.4206 (2)	0.0166 (3)
C1	0.40449 (19)	0.28746 (13)	0.5127 (2)	0.0135 (3)
C2	0.39422 (19)	0.18356 (12)	0.5117 (2)	0.0137 (3)
H2	0.4778	0.1437	0.5551	0.016*
C3	0.26064 (19)	0.13958 (13)	0.4466 (2)	0.0145 (4)
C4	0.13407 (19)	0.19420 (13)	0.3762 (2)	0.0143 (4)
C5	0.15166 (19)	0.29916 (13)	0.3806 (2)	0.0155 (4)
C6	0.28195 (19)	0.34575 (13)	0.4475 (2)	0.0151 (4)
H6	0.2885	0.4166	0.4493	0.018*
H1A	-0.001 (3)	0.0906 (19)	0.305 (3)	0.032 (7)*
H2A	0.604 (3)	0.4378 (17)	0.375 (3)	0.022 (6)*
H1B	-0.071 (3)	0.1841 (18)	0.277 (3)	0.033 (7)*
H2B	0.665 (3)	0.3444 (19)	0.346 (4)	0.039 (7)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0172 (2)	0.0123 (2)	0.0260 (2)	-0.00223 (16)	-0.00262 (17)	-0.00010 (16)
Cl2	0.0118 (2)	0.0194 (2)	0.0355 (3)	0.00248 (17)	-0.00420 (18)	0.00595 (19)
S1	0.0103 (2)	0.0123 (2)	0.0165 (2)	-0.00140 (15)	-0.00131 (16)	0.00062 (15)
O1	0.0175 (7)	0.0159 (6)	0.0177 (6)	-0.0018 (5)	0.0006 (5)	-0.0024 (5)
O2	0.0132 (7)	0.0156 (6)	0.0267 (7)	-0.0008 (5)	-0.0049 (5)	0.0048 (5)
N1	0.0123 (9)	0.0168 (8)	0.0287 (9)	-0.0024 (7)	-0.0059 (7)	0.0018 (7)
N2	0.0154 (8)	0.0138 (7)	0.0207 (8)	-0.0003 (6)	0.0027 (6)	-0.0002 (6)

supplementary materials

C1	0.0112 (9)	0.0153 (8)	0.0137 (8)	-0.0015 (6)	0.0005 (7)	-0.0005 (7)
C2	0.0120 (9)	0.0142 (8)	0.0146 (8)	0.0005 (7)	0.0003 (7)	0.0006 (7)
C3	0.0152 (9)	0.0123 (8)	0.0160 (8)	-0.0006 (7)	0.0017 (7)	0.0008 (6)
C4	0.0113 (9)	0.0174 (8)	0.0140 (8)	-0.0019 (7)	0.0003 (7)	0.0004 (7)
C5	0.0123 (9)	0.0168 (8)	0.0172 (9)	0.0036 (7)	0.0007 (7)	0.0036 (7)
C6	0.0138 (9)	0.0120 (8)	0.0195 (9)	0.0004 (6)	0.0020 (7)	0.0008 (7)

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

C11—C3	1.7363 (18)	N2—H2B	0.86 (3)
C12—C5	1.7360 (17)	C1—C2	1.394 (2)
S1—O1	1.4370 (13)	C1—C6	1.395 (2)
S1—O2	1.4419 (13)	C2—C3	1.379 (2)
S1—N2	1.6078 (16)	C2—H2	0.9500
S1—C1	1.7571 (18)	C3—C4	1.407 (2)
N1—C4	1.355 (2)	C4—C5	1.414 (2)
N1—H1A	0.80 (2)	C5—C6	1.374 (2)
N1—H1B	0.83 (3)	C6—H6	0.9500
N2—H2A	0.81 (2)		
O1—S1—O2	119.15 (8)	C3—C2—C1	118.85 (16)
O1—S1—N2	106.00 (8)	C3—C2—H2	120.6
O2—S1—N2	106.61 (8)	C1—C2—H2	120.6
O1—S1—C1	107.86 (8)	C2—C3—C4	123.34 (16)
O2—S1—C1	107.78 (8)	C2—C3—C11	118.83 (13)
N2—S1—C1	109.15 (8)	C4—C3—C11	117.83 (13)
C4—N1—H1A	119.6 (18)	N1—C4—C3	122.82 (17)
C4—N1—H1B	120.7 (17)	N1—C4—C5	122.09 (16)
H1A—N1—H1B	119 (2)	C3—C4—C5	115.08 (15)
S1—N2—H2A	112.2 (16)	C6—C5—C4	123.25 (16)
S1—N2—H2B	111.9 (17)	C6—C5—C12	118.92 (14)
H2A—N2—H2B	113 (2)	C4—C5—C12	117.83 (13)
C2—C1—C6	120.47 (16)	C5—C6—C1	118.99 (16)
C2—C1—S1	121.28 (13)	C5—C6—H6	120.5
C6—C1—S1	118.21 (13)	C1—C6—H6	120.5
O1—S1—C1—C2	-140.05 (14)	C11—C3—C4—N1	-2.6 (2)
O2—S1—C1—C2	-10.20 (17)	C2—C3—C4—C5	-1.5 (3)
N2—S1—C1—C2	105.21 (15)	C11—C3—C4—C5	178.10 (13)
O1—S1—C1—C6	42.09 (16)	N1—C4—C5—C6	-179.13 (17)
O2—S1—C1—C6	171.94 (13)	C3—C4—C5—C6	0.2 (3)
N2—S1—C1—C6	-72.65 (16)	N1—C4—C5—C12	1.0 (2)
C6—C1—C2—C3	-0.5 (3)	C3—C4—C5—C12	-179.65 (13)
S1—C1—C2—C3	-178.35 (13)	C4—C5—C6—C1	0.8 (3)
C1—C2—C3—C4	1.7 (3)	C12—C5—C6—C1	-179.29 (13)
C1—C2—C3—C11	-177.91 (13)	C2—C1—C6—C5	-0.7 (3)
C2—C3—C4—N1	177.82 (17)	S1—C1—C6—C5	177.19 (13)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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N1—H1A···C11	0.80 (2)	2.60 (2)	2.9793 (18)	111 (2)
N2—H2A···O1 ⁱ	0.81 (2)	2.14 (2)	2.935 (2)	167 (2)
N1—H1B···O2 ⁱⁱ	0.83 (3)	2.40 (3)	3.199 (2)	160 (2)
N2—H2B···O2 ⁱⁱⁱ	0.86 (3)	2.08 (3)	2.934 (2)	172 (2)

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

Fig. 1

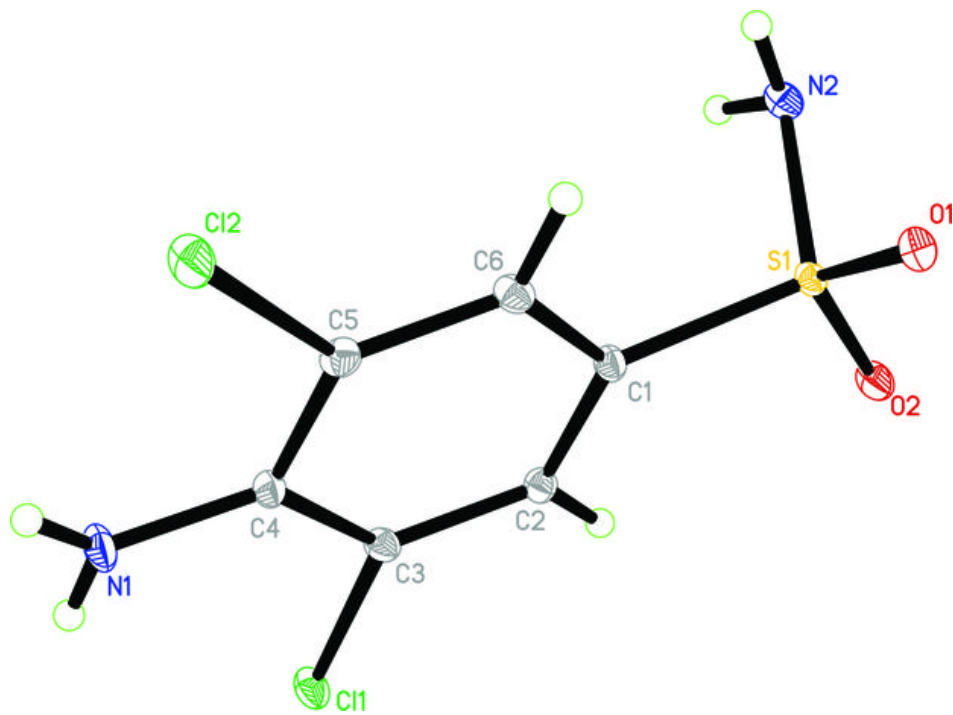


Fig. 2

