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3,5-Dichlorosalicylaldehyde

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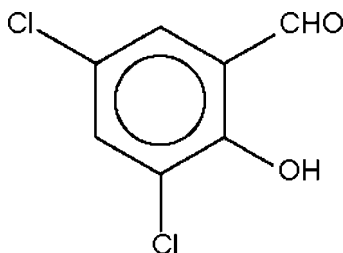
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.037; wR factor = 0.101; data-to-parameter ratio = 16.1.

The title compound (systematic name: 3,5-dichloro-2-hydroxybenzaldehyde), $\text{C}_7\text{H}_4\text{Cl}_2\text{O}_2$, crystallizes as discrete molecules, the conformation of which may be influenced by an intramolecular hydroxy-carbonyl $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond.

Related literature

For the crystal structure of 3',5'-dichloroacetophenone, see: Filarowski *et al.* (2004).



Experimental

Crystal data

$\text{C}_7\text{H}_4\text{Cl}_2\text{O}_2$
 $M_r = 191.00$
 Monoclinic, $P2_1/c$
 $a = 8.2823$ (2) Å
 $b = 13.7412$ (3) Å
 $c = 7.0973$ (2) Å
 $\beta = 115.185$ (2)°

$V = 730.95$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.82$ mm⁻¹
 $T = 100$ (2) K
 $0.25 \times 0.15 \times 0.05$ mm

Data collection

Bruker SMART APEX
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.701$, $T_{\max} = 0.960$

8436 measured reflections
 1672 independent reflections
 1303 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.101$
 $S = 1.05$
 1672 reflections
 104 parameters
 1 restraint

H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.59$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.49$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}$	0.84 (1)	1.87 (2)	2.628 (3)	149 (3)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We thank the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2606).

References

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supplementary materials

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3,5-Dichlorosalicylaldehyde

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Comment

The intramolecular hydrogen bonds in small molecules such as *ortho*-hydroxyacetophenone and its derivatives has been extensively studied, both theoretically and crystallographically. Such compounds can exist in a keto-enol equilibrium. For 3',5'-dichloroacetophenone, geometry-optimization calculations suggest that the presence of two chlorine substituents raises the acidity of the hydroxyl proton and decreases the basicity of the carbonyl function. The O...O distance in the hydrogen bond is 2.567 (3) Å (Filarowski *et al.*, 2004).

The hydrogen bond in the title molecule (I) is longer with an O...O distance of 2.628 (3) Å. 3,5-Dichlorosalicylaldehyde (I) exists as a monomeric compound (Fig. 1); the molecule is flat and all bond dimensions are normal.

Experimental

The compound was purchased from Aldrich Chemical Company; the chemical exists as colorless prismatic crystals. The bulk chemical has a yellow color.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to $1.2U(\text{C})$. The oxygen-bound H atom was located in a difference Fourier map, and was refined with a distance restraint of O—H 0.84±0.01 Å; its temperature factor was freely refined.

Figures

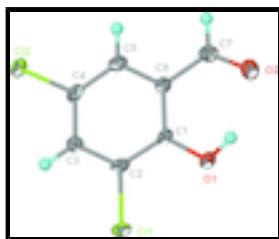


Fig. 1. 70% Probability thermal ellipsoid plot of 3,5-dichlorosalicylaldehyde. Hydrogen atoms are drawn as spheres of arbitrary radius.

3,5-dichloro-2-hydroxybenzaldehyde

Crystal data

$\text{C}_7\text{H}_4\text{Cl}_2\text{O}_2$

$M_r = 191.00$

Monoclinic, $P2_1/c$

$F(000) = 384$

$D_x = 1.736 \text{ Mg m}^{-3}$

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

supplementary materials

Hall symbol: -P 2ybc

$a = 8.2823$ (2) Å

$b = 13.7412$ (3) Å

$c = 7.0973$ (2) Å

$\beta = 115.185$ (2)°

$V = 730.95$ (3) Å³

$Z = 4$

Cell parameters from 3128 reflections

$\theta = 3.0$ – 28.2 °

$\mu = 0.82$ mm⁻¹

$T = 100$ K

Block, colorless

$0.25 \times 0.15 \times 0.05$ mm

Data collection

Bruker SMART APEXII
diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.701$, $T_{\max} = 0.960$

8436 measured reflections

1672 independent reflections

1303 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.058$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.7$ °

$h = -10$ → 10

$k = -17$ → 17

$l = -9$ → 9

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.101$

$S = 1.05$

1672 reflections

104 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

$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.8939P]$

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

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

$\Delta\rho_{\max} = 0.59$ e Å⁻³

$\Delta\rho_{\min} = -0.49$ e Å⁻³

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.17794 (9)	0.14230 (4)	0.48280 (10)	0.01972 (18)
C12	0.15345 (9)	0.53148 (4)	0.39904 (11)	0.02166 (19)
O1	0.5328 (3)	0.17615 (13)	0.8080 (3)	0.0200 (4)
H1	0.631 (3)	0.192 (3)	0.905 (4)	0.038 (10)*
O2	0.7916 (3)	0.28892 (14)	1.0580 (3)	0.0252 (4)
C1	0.4488 (3)	0.25960 (17)	0.7232 (4)	0.0155 (5)
C2	0.2766 (3)	0.25497 (17)	0.5629 (4)	0.0164 (5)
C3	0.1851 (4)	0.33804 (17)	0.4659 (4)	0.0168 (5)
H3	0.0680	0.3339	0.3572	0.020*
C4	0.2680 (4)	0.42815 (17)	0.5306 (4)	0.0177 (5)

C5	0.4355 (4)	0.43592 (17)	0.6906 (4)	0.0179 (5)
H5	0.4890	0.4980	0.7336	0.021*
C6	0.5266 (3)	0.35155 (17)	0.7897 (4)	0.0160 (5)
C7	0.7028 (4)	0.35892 (18)	0.9634 (4)	0.0206 (6)
H7	0.7517	0.4220	1.0058	0.025*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0225 (3)	0.0110 (3)	0.0228 (3)	-0.0036 (2)	0.0068 (3)	-0.0026 (2)
C12	0.0204 (3)	0.0122 (3)	0.0276 (4)	0.0029 (2)	0.0056 (3)	0.0051 (2)
O1	0.0193 (10)	0.0111 (8)	0.0243 (11)	0.0025 (7)	0.0042 (9)	0.0025 (7)
O2	0.0211 (10)	0.0197 (9)	0.0272 (11)	0.0004 (8)	0.0030 (9)	0.0033 (8)
C1	0.0182 (13)	0.0111 (10)	0.0180 (12)	0.0028 (9)	0.0083 (11)	0.0016 (9)
C2	0.0216 (13)	0.0106 (10)	0.0186 (12)	-0.0017 (9)	0.0100 (11)	-0.0016 (9)
C3	0.0163 (13)	0.0162 (12)	0.0172 (13)	-0.0004 (9)	0.0063 (11)	-0.0007 (9)
C4	0.0201 (14)	0.0114 (11)	0.0213 (13)	0.0038 (9)	0.0086 (12)	0.0032 (9)
C5	0.0202 (14)	0.0108 (11)	0.0228 (14)	-0.0024 (9)	0.0093 (12)	0.0000 (9)
C6	0.0151 (13)	0.0123 (11)	0.0195 (13)	-0.0004 (9)	0.0064 (11)	0.0003 (9)
C7	0.0212 (14)	0.0149 (12)	0.0229 (14)	-0.0029 (10)	0.0068 (12)	-0.0003 (10)

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

C11—C2	1.730 (2)	C3—C4	1.395 (3)
C12—C4	1.742 (2)	C3—H3	0.9500
O1—C1	1.343 (3)	C4—C5	1.373 (4)
O1—H1	0.840 (10)	C5—C6	1.399 (3)
O2—C7	1.223 (3)	C5—H5	0.9500
C1—C2	1.397 (4)	C6—C7	1.459 (4)
C1—C6	1.406 (3)	C7—H7	0.9500
C2—C3	1.381 (3)		
C1—O1—H1	107 (3)	C5—C4—C12	120.53 (19)
O1—C1—C2	118.7 (2)	C3—C4—C12	117.9 (2)
O1—C1—C6	122.7 (2)	C4—C5—C6	119.4 (2)
C2—C1—C6	118.5 (2)	C4—C5—H5	120.3
C3—C2—C1	121.5 (2)	C6—C5—H5	120.3
C3—C2—C11	119.6 (2)	C5—C6—C1	120.3 (2)
C1—C2—C11	118.96 (18)	C5—C6—C7	119.9 (2)
C2—C3—C4	118.7 (2)	C1—C6—C7	119.8 (2)
C2—C3—H3	120.6	O2—C7—C6	124.1 (2)
C4—C3—H3	120.6	O2—C7—H7	118.0
C5—C4—C3	121.6 (2)	C6—C7—H7	118.0
O1—C1—C2—C3	-178.3 (2)	C12—C4—C5—C6	-178.17 (19)
C6—C1—C2—C3	2.0 (4)	C4—C5—C6—C1	1.3 (4)
O1—C1—C2—C11	0.8 (3)	C4—C5—C6—C7	-178.4 (2)
C6—C1—C2—C11	-178.90 (18)	O1—C1—C6—C5	177.7 (2)
C1—C2—C3—C4	0.1 (4)	C2—C1—C6—C5	-2.7 (4)
C11—C2—C3—C4	-179.03 (19)	O1—C1—C6—C7	-2.6 (4)

supplementary materials

C2—C3—C4—C5	-1.5 (4)	C2—C1—C6—C7	177.1 (2)
C2—C3—C4—Cl2	177.52 (18)	C5—C6—C7—O2	-179.8 (3)
C3—C4—C5—C6	0.9 (4)	C1—C6—C7—O2	0.5 (4)

Hydrogen-bond geometry (Å, °)

<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 O2	0.84 (1)	1.87 (2)	2.628 (3)	149 (3)

Fig. 1

