organic compounds

3,5-Dichloro-2-hydroxybenzaldehyde

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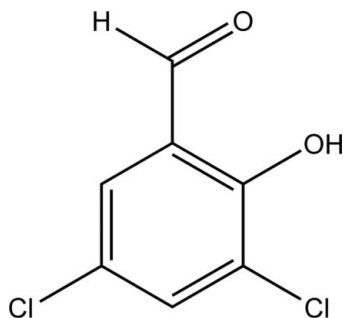
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The title compound, $C_7H_4Cl_2O_2$, exhibits a layer crystal structure; molecules within each layer are linked by weak C—H···O intermolecular hydrogen bonds. There is also an intramolecular O—H···O hydrogen bond.

Related literature

For a related compound, see: Fan *et al.* (2008).

Experimental

Crystal data

$C_7H_4Cl_2O_2$
$M_r = 191.00$
Monoclinic, $P2_1/c$
$a = 8.3359$ (16) Å
$b = 13.884$ (3) Å
$c = 7.2341$ (14) Å
$\beta = 114.519$ (2)°
$V = 761.7$ (3) Å^3
$Z = 4$

Mo $K\alpha$ radiation
$\mu = 0.79$ mm^{-1}
$T = 291$ (2) K
$0.14 \times 0.12 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.897$, $T_{max} = 0.925$

4063 measured reflections
1487 independent reflections
1181 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.097$
$S = 0.99$
1487 reflections

101 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1	0.82	1.92	2.630 (2)	145
C4—H4···O1[i]	0.93	2.51	3.428 (3)	168
C6—H6···O2[ii]	0.93	2.56	3.394 (3)	149

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2566).

References

Bruker (2000). *SMART, SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Fan, Y., You, W., Qian, H.-F., Liu, J.-L. & Huang, W. (2008). *Acta Cryst.* E**64**, o799.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

3,5-Dichloro-2-hydroxybenzaldehyde

Y. Fan, W. You, J.-L. Liu, H.-F. Qian and W. Huang

Comment

We have newly reported the X-ray single-crystal structure of 3,5-dibromo-2-hydroxybenzaldehyde (Fan *et al.*, 2008). In this paper, we report the X-ray single-crystal structure of 3,5-dichloro-2- hydroxybenzaldehyde.

The molecular structure of (I) is illustrated in Fig. 1. The selected bond distances and bond angles are normal. Different from 3,5-dibromo-2-hydroxybenzaldehyde, there is only one crystallographically independent molecule in the asymmetric unit. The molecular geometry of slicylaldehyde unit of (I) is comparable with that of 3,5-dibromo-2-hydroxybenzaldehyde.

In the crystal packing of (I), there are two sets of molecules with the dihedral angle of 6.52 (2) ° and molecules in every layer are linked by intermolecular CO—H⋯O hydrogen bondings (Fig. 2). A layer packing structure is formed with the mean interlayer separation of 3.428 (2) Å (Fig. 3.). However, no π–π stacking interactions can be observed in (I), which is different from those in 3,5-dibromo-2-hydroxybenzaldehyde.

Experimental

The title compound was obtained as received. Single crystals suitable for X-ray diffraction measurement were formed after 6 days in methanol by slow evaporation at room temperature in air. Analysis calculated for $C_7H_4O_2Cl_2$: C 44.02, H 2.11%. Found: C 44.18, H, 2.24%. FT—IR (KBr pellets, cm^{-1}): 3066(*versus*), 2856(*s*), 1666(*versus*), 1604(*m*), 1428(*s*), 1375(*versus*), 1276(*s*), 1208(*s*), 1171(*s*), 1103(*m*), 935(*s*), 891(*versus*), 735(*s*), 703(*s*), 566(*m*), and 515(*m*).

Refinement

The H atoms were placed in geometrically idealized positions (C—H = 0.93 Å and O—H = 0.82 Å) and refined as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.
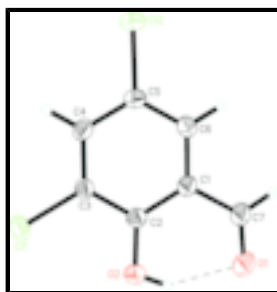
Figures

Fig. 1. An *ORTEP* drawing of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
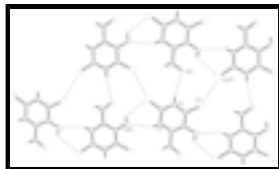
supplementary materials

Fig. 2. A perspective view of the intralayer intermolecular hydrogen-bond contacts among molecules in the title compound. Hydrogen bonds and Cl···Cl interactions are shown as dashed lines. [Symmetry codes: (i) $x+1$, y, z; (ii) $-x + 1$, $y+1/2$, $-z+1/2$; (iii) $-x+2$, $y - 1/2$, $-z + 1/2$].

Fig. 3. A perspective view of the layer packing structure of (I) together with the unit cell.

3,5-Dichloro-2-hydroxybenzaldehyde

Crystal data

$C_7H_4Cl_2O_2$	$F_{000} = 384$
$M_r = 191.00$	$D_x = 1.666$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1691 reflections
$a = 8.3359$ (16) Å	$\theta = 2.7$–$26.8°$
$b = 13.884$ (3) Å	$\mu = 0.79$ mm^{-1}
$c = 7.2341$ (14) Å	$T = 291$ (2) K
$\beta = 114.519$ (2)°	Block, yellow
$V = 761.7$ (3) Å^3	$0.14 \times 0.12 \times 0.10$ mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	1487 independent reflections
Radiation source: fine-focus sealed tube	1181 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.055$
$T = 291(2)$ K	$\theta_{max} = 26.0°$
φ and ω scans	$\theta_{min} = 2.7°$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -10 \rightarrow 10$
$T_{min} = 0.898$, $T_{max} = 0.925$	$k = -16 \rightarrow 16$
4063 measured reflections	$l = -7 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} < 0.001$

1487 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
101 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. The structure was solved by direct methods (Bruker, 2000) and successive difference Fourier syntheses.

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.4748 (2)	0.35190 (14)	0.2605 (3)	0.0419 (5)
C2	0.5509 (2)	0.26110 (14)	0.2721 (3)	0.0383 (4)
C3	0.7213 (2)	0.25643 (14)	0.2822 (3)	0.0379 (4)
C4	0.8119 (2)	0.33830 (13)	0.2774 (3)	0.0422 (5)
H4	0.9261	0.3341	0.2858	0.051*
C5	0.7313 (2)	0.42742 (15)	0.2599 (3)	0.0422 (5)
C6	0.5658 (3)	0.43474 (15)	0.2547 (3)	0.0445 (5)
H6	0.5145	0.4949	0.2474	0.053*
C7	0.2987 (3)	0.35981 (17)	0.2606 (3)	0.0536 (6)
H7	0.2525	0.4210	0.2579	0.064*
Cl1	0.81885 (7)	0.14449 (4)	0.29976 (9)	0.0542 (2)
Cl2	0.84604 (7)	0.52989 (4)	0.24617 (10)	0.0612 (2)
O1	0.21042 (19)	0.29062 (13)	0.2640 (3)	0.0678 (5)
O2	0.46796 (18)	0.17843 (10)	0.2737 (2)	0.0519 (4)
H2	0.3694	0.1904	0.2670	0.078*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0364 (10)	0.0424 (12)	0.0505 (11)	0.0021 (8)	0.0216 (9)	0.0031 (9)
C2	0.0405 (10)	0.0354 (11)	0.0423 (10)	−0.0011 (8)	0.0206 (8)	0.0018 (8)
C3	0.0389 (10)	0.0360 (11)	0.0428 (10)	0.0048 (8)	0.0209 (8)	0.0000 (8)
C4	0.0357 (10)	0.0470 (14)	0.0483 (12)	0.0007 (8)	0.0218 (9)	0.0033 (9)
C5	0.0410 (11)	0.0383 (11)	0.0492 (11)	−0.0052 (8)	0.0209 (9)	0.0034 (9)
C6	0.0432 (11)	0.0367 (11)	0.0568 (12)	0.0050 (8)	0.0240 (10)	0.0045 (9)
C7	0.0427 (12)	0.0502 (14)	0.0746 (15)	0.0042 (10)	0.0310 (11)	0.0076 (11)
Cl1	0.0576 (4)	0.0406 (3)	0.0719 (4)	0.0126 (2)	0.0344 (3)	0.0019 (2)

Cl2	0.0515 (3)	0.0438 (4)	0.0914 (5)	−0.0092 (2)	0.0327 (3)	0.0102 (3)
O1	0.0463 (8)	0.0642 (12)	0.1052 (13)	−0.0007 (8)	0.0437 (8)	0.0073 (9)
O2	0.0475 (8)	0.0370 (8)	0.0773 (10)	−0.0056 (6)	0.0319 (8)	0.0020 (7)

Geometric parameters (Å, °)

C1—C6	1.388 (3)	C4—H4	0.9300
C1—C2	1.398 (3)	C5—C6	1.369 (3)
C1—C7	1.472 (3)	C5—Cl2	1.740 (2)
C2—O2	1.342 (2)	C6—H6	0.9300
C2—C3	1.393 (2)	C7—O1	1.217 (3)
C3—C4	1.373 (3)	C7—H7	0.9300
C3—Cl1	1.7340 (19)	O2—H2	0.8200
C4—C5	1.388 (3)		
C6—C1—C2	120.59 (18)	C5—C4—H4	120.3
C6—C1—C7	119.71 (18)	C6—C5—C4	120.85 (18)
C2—C1—C7	119.68 (18)	C6—C5—Cl2	120.63 (16)
O2—C2—C3	118.51 (17)	C4—C5—Cl2	118.52 (14)
O2—C2—C1	123.29 (16)	C5—C6—C1	119.68 (18)
C3—C2—C1	118.20 (17)	C5—C6—H6	120.2
C4—C3—C2	121.30 (17)	C1—C6—H6	120.2
C4—C3—Cl1	119.86 (13)	O1—C7—C1	123.6 (2)
C2—C3—Cl1	118.84 (15)	O1—C7—H7	118.2
C3—C4—C5	119.34 (17)	C1—C7—H7	118.2
C3—C4—H4	120.3	C2—O2—H2	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.82	1.92	2.630 (2)	145
C4—H4···O1[i]	0.93	2.51	3.428 (3)	168
C6—H6···O2[ii]	0.93	2.56	3.394 (3)	149

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

Fig. 1

Fig. 2

Fig. 3