Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Chloro-2-hydroxybenzene-1,3-dicarbaldehyde

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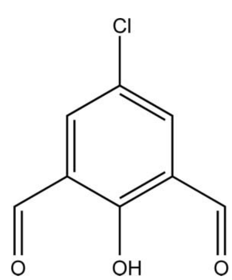
Received 23 June 2008; accepted 2 July 2008

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.092; data-to-parameter ratio = 10.8.

In the crystal structure of the title compound, $C_8H_5ClO_3$, both formyl groups are in the plane of the chlorophenyl unit and the molecule is stabilized by intramolecular O—H···O hydrogen bonding. The molecules are connected *via* intermolecular O—H···O hydrogen bonding into chains and are stacked into columns with a centroid–centroid distance between adjacent aromatic rings of 3.914 (2) Å.

Related literature

For related compounds, see: Huang *et al.* (2000, 2006); Chu *et al.* (2005); Chu & Huang (2006).



Experimental

Crystal data

$C_8H_5ClO_3$
M_r = 184.57
Monoclinic, $P2_1/c$
a = 7.5554 (15) Å
b = 3.9144 (8) Å
c = 25.676 (5) Å
β = 97.921 (3)°
V = 752.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.46 mm^{-1}
T = 291 (2) K
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.913, T_{max} = 0.930
3517 measured reflections
1353 independent reflections
1070 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
S = 0.97
1353 reflections
125 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O1^{i}	0.82	2.48	2.9581 (19)	118
O1—H1···O3	0.82	1.90	2.6204 (18)	146

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

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*.

WH acknowledges the Major State Basic Research Development Programs (grant Nos. 2006CB806104 and 2007CB925101) and the National Natural Science Foundation of China (grant No. 20301009) for financial aid.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2109).

References

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

supporting information

Acta Cryst. (2008). E**64**, o1426 [doi:10.1107/S1600536808020217]

5-Chloro-2-hydroxybenzene-1,3-dicarbaldehyde

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S1. Comment

Hydroxyisophthalaldehyde and its derivatives are an important class of intermediates used in synthesizing macrocyclic compounds. In recent years, a continuing attention has been drawn to them and their metal complexes (Huang *et al.*, 2006). In this paper, we report the X-ray single-crystal structure of 2,6-diformyl-4-chlorophenol prepared from 4-chloro-2,6-bis(hydroxymethyl)phenol.

The molecule of the title compound is essentially planar and all structural parameters (Fig. 1) are in good agreement with those found in similar compounds (Chu *et al.*, 2005; Chu & Huang, 2006). There is one weak intramolecular O-H···O hydrogen bond between the hydroxyl group at O1 and the carbonyl group O3.

In the crystal structure of the title compound the molecules are connected into chains by intermolecular O-H···O hydrogen bonding (Fig. 2 and Table 1). The molecules are stacked into columns in the direction of the crystallographic a-axis in order that π–π stacking interactions are maximized. The dihedral angle between two adjacent rings amount to 63.5 (2)° and the centroid-centroid separation is 3.914 (2) Å (Fig. 3).

S2. Experimental

4-Chloro-2,6-diformylphenol was prepared by an improved oxidation method using activated manganese (IV) dioxide (Huang *et al.*, 2000) from 4-chloro-2,6-bis(hydroxymethyl)phenol (Chu *et al.*, 2005). Single crystals suitable for X-ray diffraction measurement were grown from a chloroform solution by slow evaporation of the solvent at room temperature.

S3. Refinement

The C-H H atoms were located in difference map and were refined with varying coordinates isotropic. The O-H H atom was placed with idealized geometry allowed to rotata but not to tip O—H = 0.82 Å) and was refined using a riding model with U_{iso}(H) = 1.5U_{eq}(O).

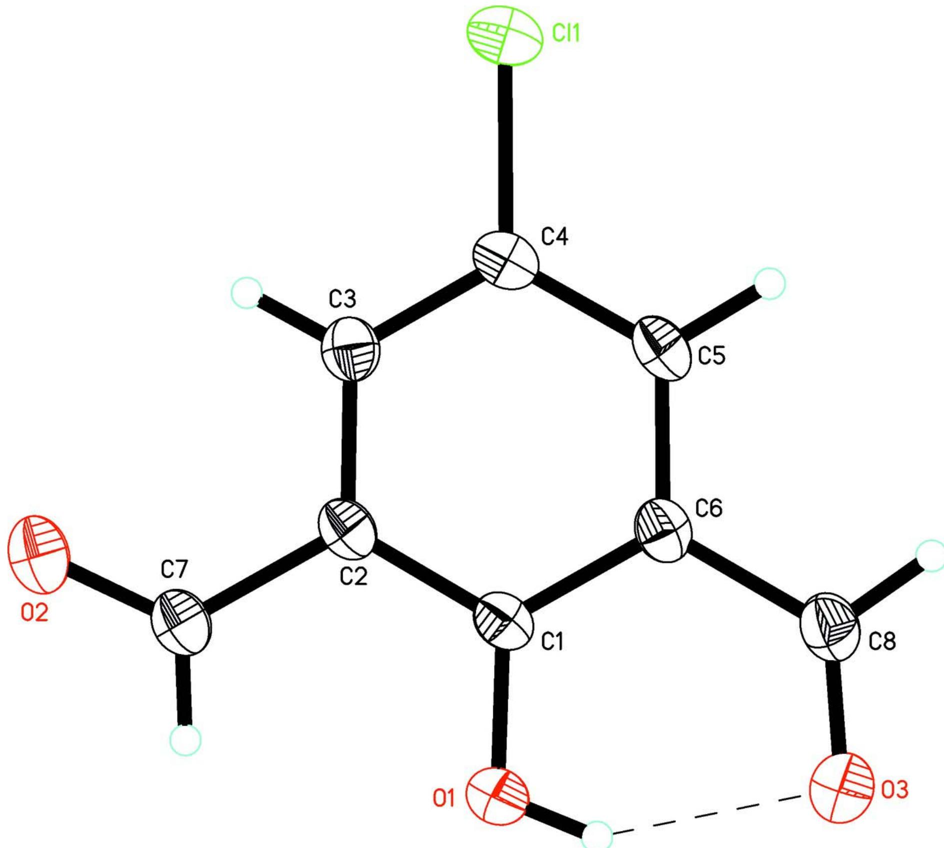

Figure 1

An *ORTEP* drawing of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and intramolecular hydrogen bonds is shown as dashed lines.

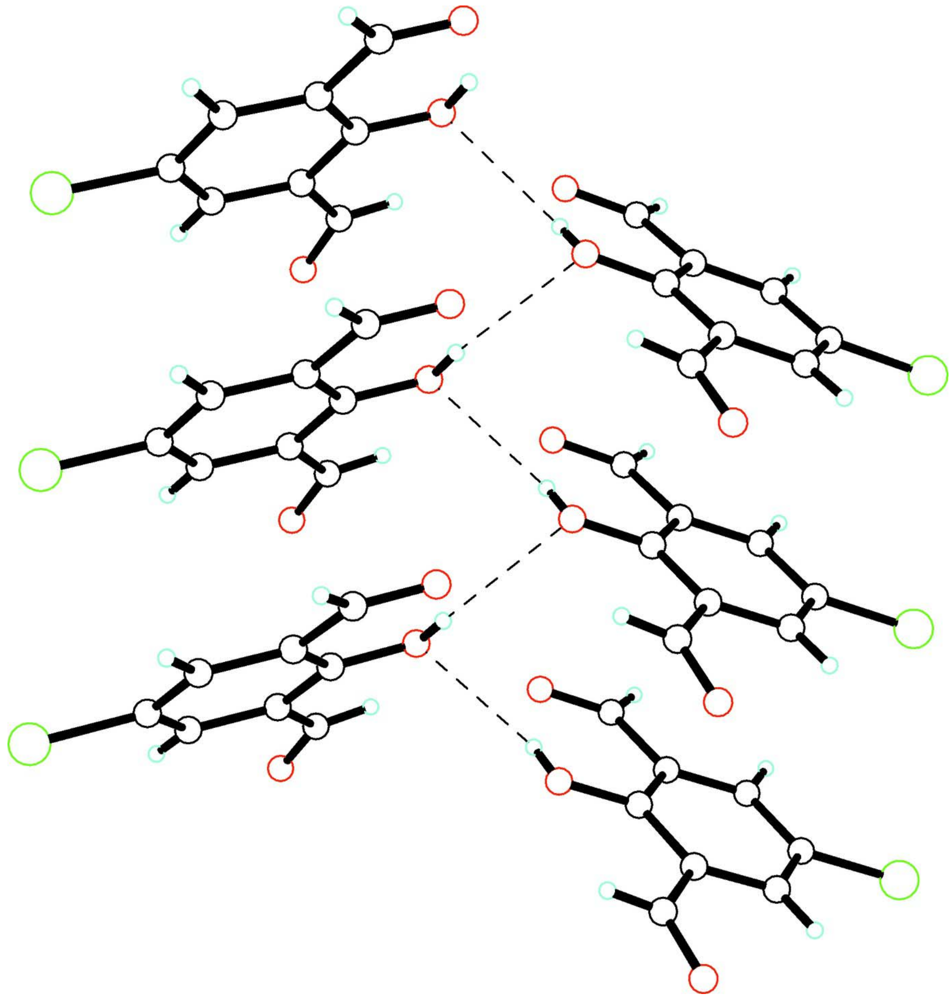

Figure 2

A perspective view of the one-dimensional hydrogen-bonded chain of the title compound.

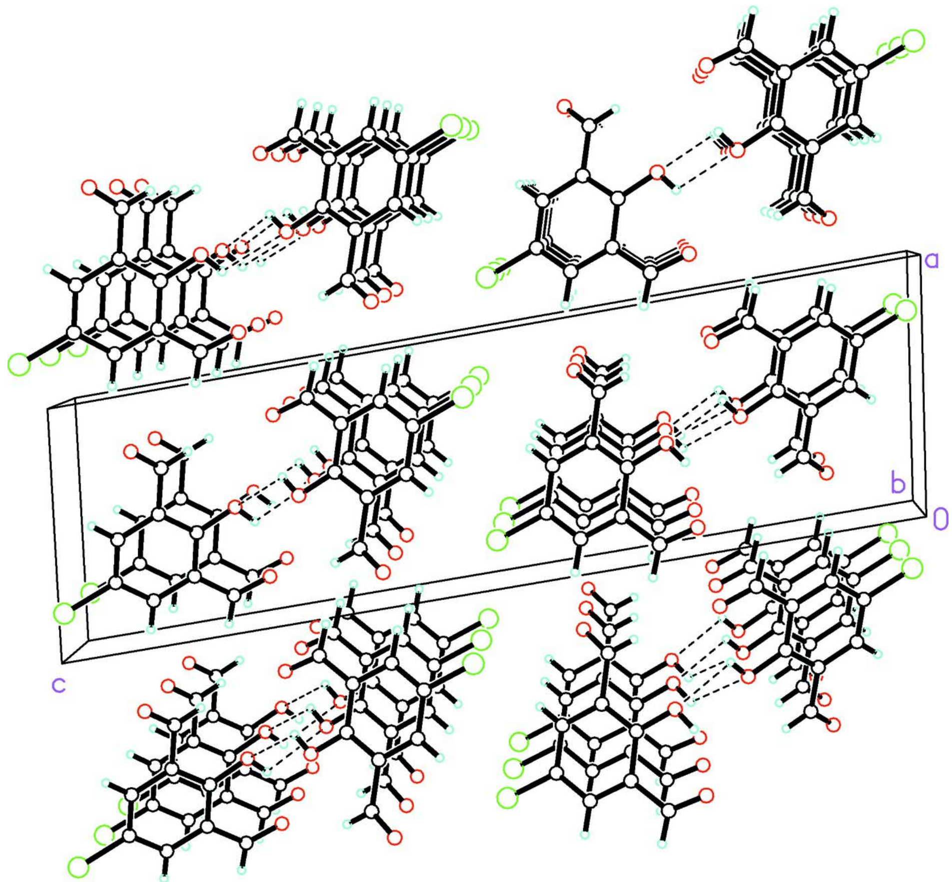

Figure 3

Crystal structure of the title compound with view along the a-axis (intermolecular O-H···O hydrogen bonding is shown as dashed lines).

5-Chloro-2-hydroxybenzene-1,3-dicarbaldehyde

Crystal data

$C_8H_5ClO_3$
M_r = 184.57
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.5554 (15) Å
b = 3.9144 (8) Å
c = 25.676 (5) Å
β = 97.921 (3)°
V = 752.1 (3) Å^3
Z = 4
F(000) = 376
D_x = 1.630 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1370 reflections
θ = 2.7–28.1°
μ = 0.46 mm^{-1}
T = 291 K
Block, yellow
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.913, T_{max} = 0.930
3517 measured reflections
1353 independent reflections
1070 reflections with $I > 2\sigma(I)$

R_{int} = 0.076
θ_{max} = 25.2°, θ_{min} = 2.7°
h = −9→8
k = −4→4
l = −30→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
S = 0.97
1353 reflections
125 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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	0.4171 (2)	0.4316 (4)	0.33617 (6)	0.0342 (4)
C2	0.5238 (2)	0.6213 (5)	0.37437 (7)	0.0355 (4)
C3	0.4574 (2)	0.7115 (5)	0.42026 (8)	0.0384 (4)
C4	0.2876 (2)	0.6140 (5)	0.42836 (7)	0.0382 (4)
C5	0.1802 (2)	0.4303 (5)	0.39068 (7)	0.0372 (4)
C6	0.2425 (2)	0.3385 (5)	0.34400 (7)	0.0348 (4)
C7	0.7038 (3)	0.7330 (5)	0.36545 (9)	0.0447 (5)
C8	0.1285 (3)	0.1496 (5)	0.30341 (8)	0.0426 (5)
Cl1	0.20868 (7)	0.73166 (14)	0.486064 (19)	0.0550 (2)
H3	0.532 (3)	0.831 (5)	0.4454 (7)	0.047 (5)*
H5	0.064 (3)	0.358 (5)	0.3977 (8)	0.052 (5)*
H7	0.747 (3)	0.653 (5)	0.3360 (8)	0.052 (6)*
H8	0.015 (2)	0.099 (5)	0.3119 (7)	0.044 (5)*
O1	0.48613 (16)	0.3399 (3)	0.29244 (5)	0.0467 (4)
H1	0.4106	0.2351	0.2726	0.070*
O2	0.79414 (18)	0.9339 (4)	0.39307 (6)	0.0606 (4)
O3	0.17345 (17)	0.0628 (4)	0.26168 (5)	0.0544 (4)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0313 (9)	0.0364 (10)	0.0359 (9)	0.0027 (8)	0.0079 (7)	0.0064 (8)
C2	0.0293 (9)	0.0375 (10)	0.0404 (10)	0.0016 (8)	0.0068 (7)	0.0059 (8)
C3	0.0349 (10)	0.0375 (11)	0.0424 (11)	−0.0001 (8)	0.0034 (8)	0.0017 (8)
C4	0.0372 (10)	0.0403 (10)	0.0388 (10)	0.0048 (8)	0.0110 (8)	0.0052 (8)
C5	0.0266 (9)	0.0412 (11)	0.0447 (10)	0.0026 (8)	0.0078 (8)	0.0096 (8)
C6	0.0295 (10)	0.0352 (10)	0.0392 (10)	0.0020 (7)	0.0026 (7)	0.0074 (8)
C7	0.0320 (11)	0.0524 (13)	0.0504 (12)	−0.0027 (9)	0.0083 (9)	−0.0016 (10)
C8	0.0329 (11)	0.0487 (12)	0.0456 (11)	−0.0010 (9)	0.0036 (8)	0.0062 (9)
Cl1	0.0548 (4)	0.0668 (4)	0.0475 (3)	−0.0021 (2)	0.0221 (2)	−0.0052 (2)
O1	0.0400 (7)	0.0626 (9)	0.0393 (7)	−0.0074 (6)	0.0120 (5)	−0.0060 (6)
O2	0.0404 (8)	0.0732 (10)	0.0689 (9)	−0.0158 (8)	0.0099 (7)	−0.0082 (8)
O3	0.0454 (8)	0.0680 (10)	0.0488 (8)	−0.0076 (7)	0.0036 (6)	−0.0092 (7)

Geometric parameters (Å, º)

C1—O1	1.3500 (19)	C5—C6	1.394 (2)
C1—C2	1.395 (2)	C5—H5	0.96 (2)
C1—C6	1.409 (2)	C6—C8	1.459 (3)
C2—C3	1.388 (2)	C7—O2	1.206 (2)
C2—C7	1.476 (2)	C7—H7	0.92 (2)
C3—C4	1.382 (2)	C8—O3	1.216 (2)
C3—H3	0.926 (19)	C8—H8	0.938 (18)
C4—C5	1.377 (3)	O1—H1	0.8200
C4—Cl1	1.7337 (17)		
O1—C1—C2	118.32 (15)	C4—C5—H5	119.1 (12)
O1—C1—C6	121.75 (15)	C6—C5—H5	120.6 (12)
C2—C1—C6	119.93 (15)	C5—C6—C1	119.33 (16)
C3—C2—C1	119.40 (16)	C5—C6—C8	120.57 (16)
C3—C2—C7	120.30 (17)	C1—C6—C8	120.10 (16)
C1—C2—C7	120.27 (16)	O2—C7—C2	124.11 (19)
C4—C3—C2	120.61 (18)	O2—C7—H7	118.0 (13)
C4—C3—H3	121.7 (12)	C2—C7—H7	117.8 (13)
C2—C3—H3	117.7 (12)	O3—C8—C6	124.18 (18)
C5—C4—C3	120.54 (16)	O3—C8—H8	121.5 (11)
C5—C4—Cl1	120.03 (13)	C6—C8—H8	114.3 (11)
C3—C4—Cl1	119.42 (15)	C1—O1—H1	109.5
C4—C5—C6	120.17 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1^{i}	0.82	2.48	2.9581 (19)	118
O1—H1···O3	0.82	1.90	2.6204 (18)	146

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