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## Structure Reports

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## 2,3-Dichlorobenzene-1,4-diol

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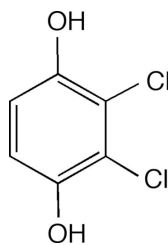
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Key indicators: single-crystal X-ray study;  $T = 294$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  
 $R$  factor = 0.034;  $wR$  factor = 0.050; data-to-parameter ratio = 14.0.

The achiral title compound,  $\text{C}_6\text{H}_4\text{Cl}_2\text{O}_2$ , crystallizes with  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonding linking molecules into layers. Between layers there are chains of  $\text{Cl}\cdots\text{Cl}\cdots\text{Cl}$  interactions with alternating distances of 3.274 (2) and 3.742 (2) Å. Augmenting this arrangement there are also  $\text{C}-\text{H}\cdots\text{Cl}$  (2.97 and 3.17 Å) and  $\text{Cl}\cdots\pi$  (shortest distances 3.40 and 3.54 Å) interactions.

## Related literature

For the structures of related dichloronaphthalenediols, see: Ahn *et al.* (1995, 1996). For the preparation of the title compound, see: Beddoes *et al.* (1981).



## Experimental

## Crystal data

$\text{C}_6\text{H}_4\text{Cl}_2\text{O}_2$   
 $M_r = 179.0$   
Monoclinic,  $P2_1/c$   
 $a = 4.831$  (1) Å  
 $b = 11.347$  (2) Å  
 $c = 12.962$  (3) Å  
 $\beta = 105.94$  (1)°

$V = 683.2$  (2) Å<sup>3</sup>  
 $Z = 4$   
Cu  $K\alpha$  radiation  
 $\mu = 8.02$  mm<sup>-1</sup>  
 $T = 294$  K  
 $0.15 \times 0.15 \times 0.06$  mm

## Data collection

Enraf–Nonius CAD-4 diffractometer  
Absorption correction: analytical (de Meulenaer & Tompa, 1965)  
 $T_{\min} = 0.33$ ,  $T_{\max} = 0.63$   
1449 measured reflections

1290 independent reflections  
1187 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.041$   
1 standard reflections  
frequency: 30 min  
intensity decay: 21%

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.050$   
 $S = 1.87$   
1290 reflections

92 parameters  
H-atom parameters not refined  
 $\Delta\rho_{\max} = 0.42$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.31$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1O1}\cdots\text{O2}^{\text{i}}$	1.00	1.84	2.794 (2)	158
$\text{O2}-\text{H1O2}\cdots\text{O1}^{\text{ii}}$	1.00	1.78	2.778 (2)	172

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

Data collection: *CAD-4 Manual* (Schagen *et al.*, 1989); cell refinement: *CAD-4 Manual*; data reduction: local program; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *RAELS* (Rae, 2000); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CrystalMaker* (CrystalMaker Software, 2005); software used to prepare material for publication: local programs.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2117).

## References

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**supplementary materials**

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## 2,3-Dichlorobenzene-1,4-diol

P. D. Ahn, R. Bishop, D. C. Craig and M. L. Scudder

### Comment

Crystal structures of related dichloronaphthalenediols have been previously reported by us (Ahn *et al.*, 1995, 1996). In the title compound (Fig 1), each molecule participates in four hydrogen bonds, two as donor and two as acceptor, creating a layer structure in the *ac* plane with O1-H1O1...O2-H1O2...O1-H1O1... chains parallel to *a* (Fig 2, Table 1). Aromatic offset face-face stacking at a distance of 3.5 Å takes place within the layer. Chains of C11...C11...C11 interactions (alternating distances 3.274 (2) and 3.742 (2) Å) which also run parallel to *a* link the layers into a three-dimensional array. In addition there are C5H...C12 and C6H...C12 (2.97 and 3.17 Å) and C12... $\pi$  interactions (shortest distances 3.40 and 3.54 Å).

### Experimental

2,3-Dichlorobenzene-1,4-diol was prepared as described (Beddoes *et al.*, 1981).  $^1\text{H}$  NMR (300 MHz,  $d_6$ -DMSO)  $\delta$  6.79 (s, 2H), 9.71 (s, 2H, -OH);  $^{13}\text{C}$  NMR (75 MHz,  $d_6$ -DMSO)  $\delta$  115.1 (CH), 119.2 (C), 147.1 (C). X-ray quality solvent-free crystals were obtained from benzene solution.

### Refinement

H atoms attached to C were included at calculated positions (C—H = 1.0 Å). The hydroxy hydrogen atoms were located on a difference map, and were then fixed at a position along the OH vector with O—H = 1.0 Å. All hydrogen atoms were refined with isotropic thermal parameters equivalent to those of the atom to which they were bonded.

### Figures

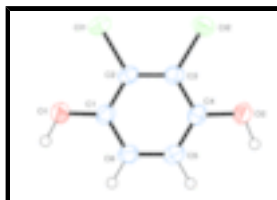


Fig. 1. A molecule of the title compound, showing atom labelling. Thermal ellipsoids are drawn at the 50% level.

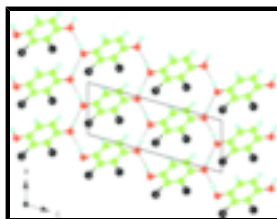


Fig. 2. A hydrogen bonded layer. Each molecule participates in two donor and two acceptor hydrogen bonds which are represented as dashed lines.

## 2,3-Dichlorobenzene-1,4-diol

### Crystal data

$C_6H_4Cl_2O_2$	$F_{000} = 360.0$
$M_r = 179.0$	$D_x = 1.74 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
$a = 4.831 (1) \text{ \AA}$	$\lambda = 1.54184 \text{ \AA}$
$b = 11.347 (2) \text{ \AA}$	Cell parameters from 10 reflections
$c = 12.962 (3) \text{ \AA}$	$\theta = 25\text{--}30^\circ$
$\beta = 105.94 (1)^\circ$	$\mu = 8.02 \text{ mm}^{-1}$
$V = 683.2 (2) \text{ \AA}^3$	$T = 294 \text{ K}$
$Z = 4$	Tabular, colourless
	$0.15 \times 0.15 \times 0.06 \text{ mm}$

### Data collection

Enraf-Nonius CAD-4 diffractometer	$\theta_{\text{max}} = 70^\circ$
$\omega$ - $2\theta$ scans	$h = -5 \rightarrow 0$
Absorption correction: analytical (de Meulenaer & Tompa, 1965)	$k = -13 \rightarrow 0$
$T_{\text{min}} = 0.33$ , $T_{\text{max}} = 0.63$	$l = -15 \rightarrow 15$
1449 measured reflections	1 standard reflections
1290 independent reflections	every 30 min
1187 reflections with $I > 2\sigma(I)$	intensity decay: 21%
$R_{\text{int}} = 0.041$	

### Refinement

Refinement on $F$	H-atom parameters not refined
$R[F^2 > 2\sigma(F^2)] = 0.034$	$w = 1/[\sigma^2(F) + 0.0004F^2]$
$wR(F^2) = 0.050$	$(\Delta/\sigma)_{\text{max}} = 0.008$
$S = 1.87$	$\Delta\rho_{\text{max}} = 0.42 \text{ e \AA}^{-3}$
1290 reflections	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
92 parameters	Extinction correction: none

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.78228 (10)	0.11149 (4)	0.49607 (4)	0.0444 (2)
Cl2	0.54566 (11)	0.00422 (4)	0.26478 (4)	0.0439 (2)
O1	0.4715 (3)	0.3170 (1)	0.5364 (1)	0.0463 (4)
O2	0.0408 (3)	0.1224 (1)	0.1331 (1)	0.0404 (4)
C1	0.3548 (4)	0.2698 (2)	0.4364 (2)	0.0340 (4)
C2	0.4868 (4)	0.1726 (2)	0.4063 (1)	0.0328 (4)

C3	0.3797 (4)	0.1238 (2)	0.3049 (2)	0.0319 (4)
C4	0.1366 (4)	0.1719 (2)	0.2334 (1)	0.0334 (4)
C5	0.0028 (4)	0.2679 (2)	0.2647 (2)	0.0363 (4)
C6	0.1126 (4)	0.3174 (2)	0.3651 (2)	0.0363 (4)
H1O1	0.3196	0.3578	0.5622	0.046
H1O2	-0.1619	0.1499	0.1026	0.040
HC5	-0.1745	0.3018	0.2143	0.036
HC6	0.0168	0.3879	0.3864	0.036

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cl1	0.0340 (3)	0.0503 (3)	0.0427 (3)	0.0103 (2)	-0.0001 (2)	0.0039 (2)
Cl2	0.0407 (3)	0.0389 (3)	0.0521 (4)	0.0050 (2)	0.0128 (2)	-0.0066 (2)
O1	0.0360 (8)	0.0620 (9)	0.0367 (7)	0.0060 (6)	0.0030 (6)	-0.0131 (7)
O2	0.0365 (7)	0.0492 (8)	0.0324 (7)	-0.0001 (6)	0.0044 (6)	-0.0040 (6)
C1	0.0291 (9)	0.0403 (9)	0.0316 (9)	-0.0004 (7)	0.0068 (7)	-0.0004 (7)
C2	0.0251 (8)	0.0374 (9)	0.0352 (9)	0.0018 (7)	0.0072 (7)	0.0040 (7)
C3	0.0281 (9)	0.0318 (8)	0.037 (1)	0.0001 (6)	0.0101 (7)	0.0009 (7)
C4	0.0302 (9)	0.0386 (9)	0.0306 (9)	-0.0049 (7)	0.0070 (7)	0.0018 (7)
C5	0.0314 (9)	0.040 (1)	0.036 (1)	0.0038 (7)	0.0061 (7)	0.0051 (8)
C6	0.031 (1)	0.039 (1)	0.039 (1)	0.0056 (7)	0.0097 (8)	0.0020 (8)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Cl1—C2	1.721 (2)	C1—C6	1.386 (2)
Cl2—C3	1.727 (2)	C2—C3	1.389 (3)
O1—C1	1.372 (2)	C3—C4	1.392 (3)
O1—H1O1	1.000	C4—C5	1.383 (3)
O2—C4	1.374 (2)	C5—C6	1.383 (3)
O2—H1O2	1.000	C5—HC5	1.000
C1—C2	1.383 (3)	C6—HC6	1.000
C1—O1—H1O1	110.4	C2—C3—C4	120.1 (2)
C4—O2—H1O2	106.6	O2—C4—C3	118.2 (2)
O1—C1—C2	118.4 (2)	O2—C4—C5	122.4 (2)
O1—C1—C6	122.1 (2)	C3—C4—C5	119.4 (2)
C2—C1—C6	119.5 (2)	C4—C5—C6	120.4 (2)
Cl1—C2—C1	119.5 (1)	C4—C5—HC5	119.8
Cl1—C2—C3	120.3 (1)	C6—C5—HC5	119.8
C1—C2—C3	120.3 (2)	C1—C6—C5	120.3 (2)
Cl2—C3—C2	121.0 (1)	C1—C6—HC6	119.8
Cl2—C3—C4	118.9 (1)	C5—C6—HC6	119.8

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1O1 $\cdots$ O2 <sup>i</sup>	1.00	1.84	2.794 (2)	158
O2—H1O2 $\cdots$ O1 <sup>ii</sup>	1.00	1.78	2.778 (2)	172

# supplementary materials

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

Fig. 1

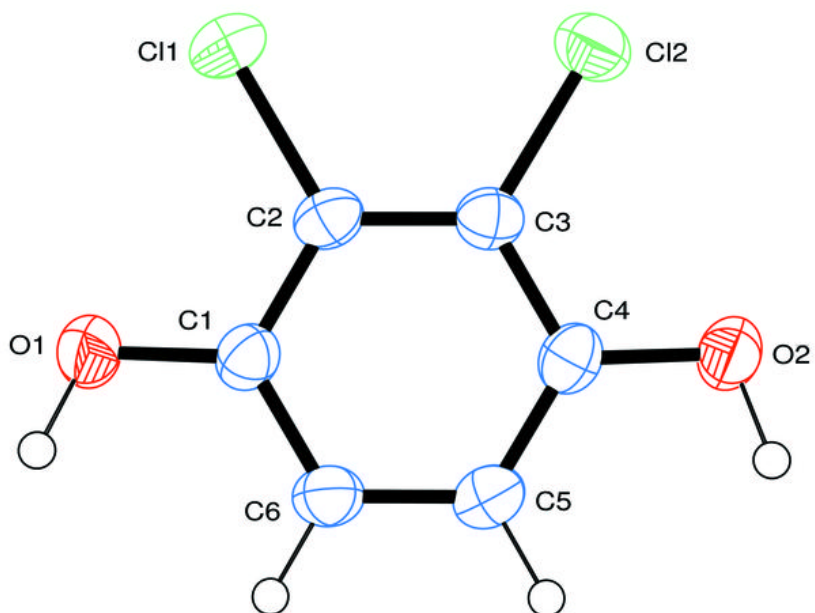


Fig. 2

