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4-Chloro-2,6-bis(hydroxymethyl)phenol

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.089
Data-to-parameter ratio = 10.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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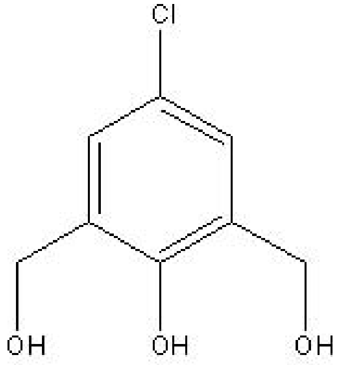
In the title compound, $C_8H_9ClO_3$, intermolecular O—H···O hydrogen bonds and π–π interactions are highly effective in forming the three-dimensional supramolecular network, thereby stabilizing the crystal structure.

Comment

The dihydroxymethylation reaction of phenol has been investigated widely to date. A series of 4-substituted 2,6-bis(hydroxymethyl)phenols have been obtained in high yields *via* phenol–formaldehyde condensation in alkaline solution (Oehler *et al.*, 1985; Perrin & Cherared, 1986; Perrin *et al.*, 1986; Crisp *et al.*, 2000; Masci & Thuéry, 2002). They are very useful intermediates in the preparation of macrocylcic Schiff bases and their metal complexes (Huang *et al.*, 2000, 2001, 2002).



(I)

In the title compound, (I) (Fig. 1), hydroxymethyl atoms O2 and O3 are at distances of 0.172 (1) and 1.302 (1) Å from the aromatic ring plane. The C—O bond lengths (Table 1) are in agreement with the corresponding ones in similar structures with different 4-substituent groups (Oehler *et al.*, 1985; Masci & Thuéry, 2002).

Intra- and intermolecular hydrogen bonds (Table 2) are highly effective in the formation of a three-dimensional network (Fig. 2). There are two sets of benzene rings, with a dihedral angle of 32.5 (1)°, each set packing in a parallel fashion by means of weak offset head-to-tail π–π stacking interactions.

The molecules form centrosymmetric dimers held together by two complementary hydrogen bonds between hydroxymethyl groups. The centroid–centroid separation between them is 3.919 (2) Å. Moreover, π–π packing interactions between the two adjacent aromatic rings belonging to different dimers are also observed, with a centroid–centroid separation of 3.803 (2) Å (Fig. 3).

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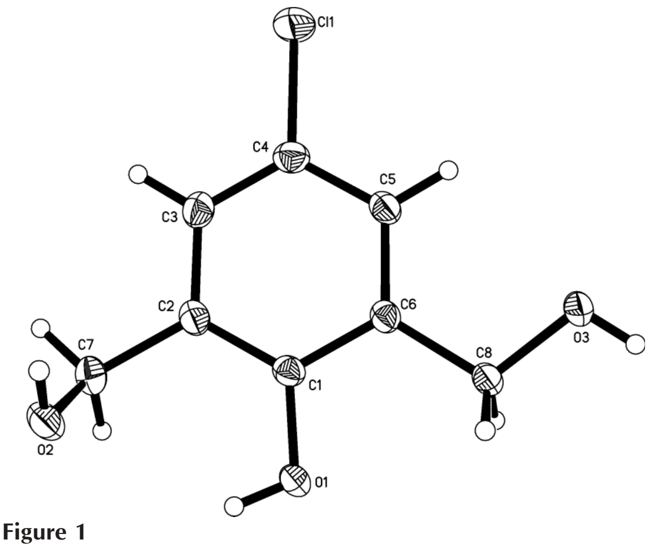

Figure 1
Drawing of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

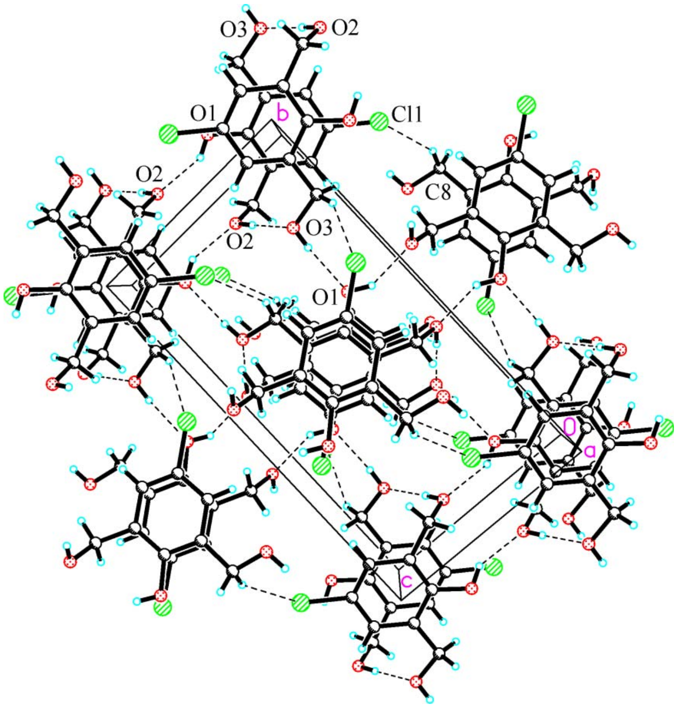

Figure 2
A packing diagram of (I). Dashed lines indicate hydrogen bonds.

Intermolecular O—H$\cdots$O hydrogen bonds and π–π interactions are highly effective in the formation of the three-dimensional supramolecular network, thereby stabilizing the crystal structure.

Experimental

The title compound was prepared according to the method of Openshaw & Roinson (1946). Analysis calculated for (I): C 50.95, H 4.81%; found: C 50.92, H 4.77%. IR (KBr, cm^{-1}): 3413 (*s*), 3301 (*s*), 2965 (*m*), 2913 (*m*), 2886 (*m*), 1478 (*s*), 1459 (*s*), 1332 (*s*), 1255 (*s*), 1211 (*s*), 1069 (*s*), 1011 (*s*), 870 (*m*), 717 (*m*), 632 (*w*), 598 (*w*). The title compound was crystallized from ethanol by slow evaporation [yield 1.55 g, 82%; m.p. 330–331 K, literature m.p. 331–333 K (Moshfegh *et al.*, 1982)].

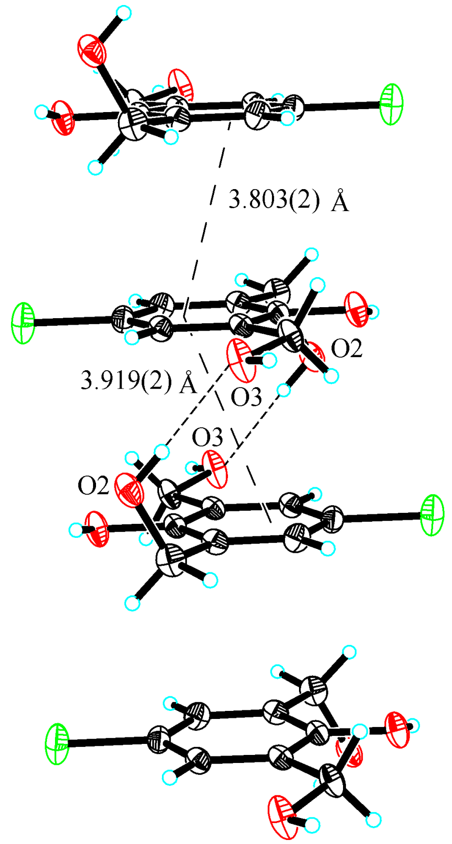

Figure 3
A perspective view of the π–π stacking in (I).

Crystal data

$C_8H_9ClO_3$
$M_r = 188.60$
Monoclinic, $P2_1/c$
$a = 7.346$ (2) Å
$b = 14.306$ (4) Å
$c = 8.396$ (2) Å
$\beta = 113.447$ (4)°
$V = 809.5$ (4) Å^3
$Z = 4$
$D_x = 1.548$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 634 reflections
$\theta = 3.0$–25.1°
$\mu = 0.43$ mm^{-1}
$T = 291$ (2) K
Block, colorless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.846$, $T_{\max} = 0.919$
4259 measured reflections
1585 independent reflections
1217 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 26.0°$
$h = -9 \rightarrow 9$
$k = -17 \rightarrow 11$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.089$
$S = 0.98$
1585 reflections
145 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.365 (2)	C6—C8	1.494 (3)
C2—C7	1.499 (3)	C7—O2	1.420 (3)
C4—Cl1	1.746 (2)	C8—O3	1.418 (2)
O2—C7—C2	112.26 (17)	O3—C8—C6	110.66 (16)
C3—C2—C7—O2	−108.5 (2)	C5—C6—C8—O3	8.3 (3)
C1—C2—C7—O2	68.8 (2)	C1—C6—C8—O3	−172.13 (17)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.80 (2)	1.91 (2)	2.666 (2)	158 (2)
O2—H2···O3^{ii}	0.81 (2)	1.98 (2)	2.787 (2)	178 (2)
O3—H3*A*···O1^{iii}	0.81 (2)	2.00 (2)	2.807 (2)	177 (2)
C8—H8*B*···Cl1^{iv}	0.99 (2)	2.81 (2)	3.729 (2)	155 (1)

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $-x+1, -y+2, -z+2$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x+2, y-\frac{1}{2}, -z+\frac{5}{2}$.

H atoms were located in a difference synthesis and were refined isotropically [C—H = 0.91 (2) Å, O—H = 0.80 (2)–0.81 (2) Å and CH_2 C—H = 0.96 (2)–0.99 (2) Å].

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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