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1,2-Bis(2-chlorophenyl)-1,2-bis(3,4-dimethylphenyl)ethane-1,2-diol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.076
wR factor = 0.175
Data-to-parameter ratio = 16.1

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

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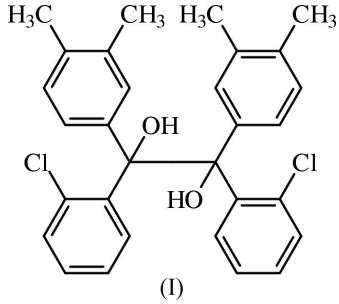
The asymmetric unit of the title compound, $C_{30}H_{28}Cl_2O_2$, contains a half molecule with the other half generated by the crystallographic twofold symmetry. The hydroxyl groups are in the *trans* configuration. The molecular structure is stabilized by O—H···Cl and C—H···O hydrogen bonds, and C—H···π interactions stabilize the crystal packing.

Comment

The X-ray crystal structure analysis of the title compound, (I), was carried out to study its molecular conformation and hydrogen-bonding characteristics.



(I)

The asymmetric unit contains one half-molecule of (I) with the other half being generated by the twofold rotation symmetry operation $(1-x, y, \frac{1}{2}-z)$ (Fig. 1). The C1—C1^{i}

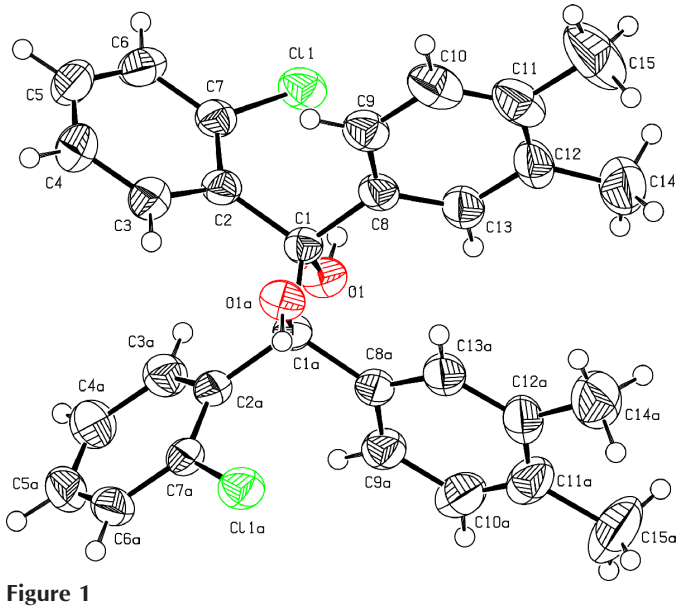


Figure 1
ZORTEP (Zsolnai, 1998) plot of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The suffix 'a' corresponds to symmetry code i in Table 1.

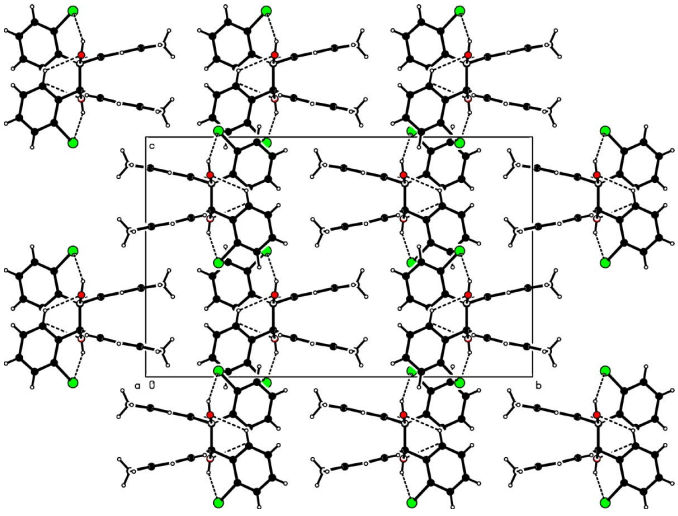


Figure 2
Packing of (I), viewed down the *a* axis. Dashed lines represent intramolecular hydrogen bonds.

bond distance [1.585 (6) Å; symmetry code as in Table 1] of the ethane group is comparable with the mean value of 1.588 Å reported by Allen *et al.* (1987) and those observed in related structures (Mak *et al.*, 1998; Pozharskii *et al.*, 2000; Bond *et al.*, 1989). The C—Cl bond length of 1.756 (3) Å agrees with the mean value of 1.734 Å (Allen *et al.*, 1987). The O1—C1—C1^{i}—O1^{i} torsion angle [173.0 (2)°] indicates that the hydroxyl groups are in the *trans* configuration. The C2–C7 and C8–C13 benzene rings are oriented approximately perpendicular to each other [dihedral angle = 87.3 (1)°].

The molecular structure of (I) is stabilized by O—H···Cl and C—H···O hydrogen bonds (Table 2 and Fig. 2). The molecular packing in the crystal is stabilized by a weak C—H···π interaction, C14—H14*A*···*Cg*1, where *Cg*1 is the C8–C13 ring centroid (Desiraju, 1989).

Experimental

Compound (I) was prepared following the procedure adopted by Matsukawa & Hinakubo (2003). To a mixture of Sm powder (450 mg, 3 mmol) and $SmCl_3$ (364 mg, 1 mmol) in water was added 3,4-dimethoxyphenylbenzoyl chloride (244.7 mg, 1 mmol). After 36 h, the resultant yellow–green suspension was treated with 2 *M* HCl (10 ml) and extracted with ether. The organic layer was washed with $NaHCO_3$ (twice) and brine, then dried and concentrated *in vacuo*. The crude product was subjected to flash column chromatography and the coupling product obtained was then recrystallized from methanol.

Crystal data

$C_{30}H_{28}Cl_2O_2$
M_r = 491.42
Monoclinic, *C*2/*c*
a = 7.667 (2) Å
b = 22.862 (6) Å
c = 14.654 (4) Å
β = 104.478 (4)°
V = 2487.0 (11) Å^3
Z = 4
D_x = 1.312 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2531 reflections
θ = 1.8–27.1°
μ = 0.29 mm^{-1}
T = 293 (2) K
Needle, colourless
0.52 × 0.24 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
9108 measured reflections
2531 independent reflections
1904 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.1°
h = −9 → 9
k = −28 → 28
l = −18 → 18

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.175
S = 1.17
2531 reflections
157 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 2.2226P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C7	1.756 (3)	C1—C1^{i}	1.585 (6)
O1—C1	1.435 (4)	C11—C15	1.532 (5)
C1—C8	1.535 (4)	C12—C14	1.468 (5)
C1—C2	1.554 (4)		
O1—C1—C1^{i}	102.8 (3)	C2—C1—C1^{i}	114.91 (19)
C8—C1—C1^{i}	109.4 (2)		
O1—C1—C2—C7	52.6 (4)	O1—C1—C8—C13	26.2 (4)
C1^{i}—C1—C2—C7	167.8 (3)	C1^{i}—C1—C8—C13	−86.5 (4)
O1—C1—C2—C3	−128.7 (3)	O1—C1—C8—C9	−157.1 (3)
C1^{i}—C1—C2—C3	−13.4 (4)	C1^{i}—C1—C8—C9	90.2 (3)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1	0.82	2.35	3.010 (2)	138
C13—H13···O1	0.93	2.48	2.817 (4)	102
C3—H3···O1^{i}	0.93	2.34	2.943 (4)	122
C14—H14*A*···*Cg*1^{ii}	0.96	2.90	3.751 (5)	148

Symmetry codes: (i) $1-x, y, \frac{1}{2}-z$; (ii) $\frac{1}{2}+x, \frac{1}{2}+y, 1+z$. Note: *Cg*1 is the C8–C13 ring centroid

All H atoms were positioned geometrically (O—H = 0.82 Å and C—H = 0.93 or 0.96 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1998) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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