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1,4-Bis[3-chloro-2-(chloromethyl)-propyl]benzene

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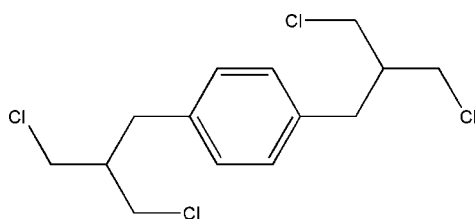
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.040; wR factor = 0.113; data-to-parameter ratio = 20.5.

The title molecule, $\text{C}_{14}\text{H}_{18}\text{Cl}_4$, possesses a crystallographically imposed inversion centre, which coincides with the centre of benzene ring. In the absence of classical intermolecular interactions, van der Waals forces help the molecules to pack in the crystal.

Related literature

For related crystal structures, see: Chen *et al.* (2005); Gao *et al.* (2009). For general background, see Amabilino & Stoddart (1995).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{18}\text{Cl}_4$
 $M_r = 328.08$
 Monoclinic, $P2_1/c$
 $a = 6.518$ (3) Å
 $b = 14.680$ (6) Å
 $c = 8.433$ (4) Å
 $\beta = 106.335$ (5)°

$V = 774.3$ (6) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.75$ mm⁻¹
 $T = 291$ (2) K
 $0.30 \times 0.26 \times 0.24$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.807$, $T_{\max} = 0.842$

4423 measured reflections
 1679 independent reflections
 1275 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.066$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.113$
 $S = 1.09$
 1679 reflections

82 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2502).

References

- Amabilino, D. B. & Stoddart, J. F. (1995). *Chem. Rev.* **95**, 2725–2737.
 Bruker (2000). SMART, SAINT and SADABS. Bruker AXS Inc. Madison, Wisconsin, USA.
 Chen, A.-H., Wang, Z.-G., Yin, G.-D. & Wu, A.-X. (2005). *Acta Cryst.* **E61**, o3240–o3241.
 Gao, Y., Xi, H., Sun, X., Fu, Y. & Liu, L. (2009). *Acta Cryst.* **E65**, o170.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2009). E65, o473 [doi:10.1107/S1600536809000609]

1,4-Bis[3-chloro-2-(chloromethyl)propyl]benzene

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Comment

The molecular recognition between a π -electron-rich hydroquinone ring and π -electron-deficient cyclophane has provided the inspiration for the self-assembly of a large number of catenanes (Amabilino & Stoddart, 1995). In our study of the applications of fused bipyridine cyclophane compounds in the self-assembly of supramolecular systems, we obtained tetraethyl 2,2'-(*p*-phenylenedimethylene)dimalonate (Chen *et al.*, 2005), which was used in the synthesis of 2,2'-(*p*-phenylenedimethylene)bis(propane-1,3-diol) (Gao *et al.*, 2009). The title compound, (I), was obtained by the chlorination of the diol. Herewith we present the crystal structure of (I) (Fig. 1).

Experimental

The 2,2'-(*p*-phenylenedimethylene)bis(propane-1,3-diol), used in this study, was obtained in accordance with the Gao *et al.* (2005). In a flame-dried, round-bottomed flask was placed SOCl_2 (5 mL) and *p*- $\text{C}_6\text{H}_4[\text{CH}_2\text{CH}(\text{CH}_2\text{OH})_2]_2$ (0.508 g, 2 mmol) was slowly added under stirring. The mixture was heated up to 333 K. The solvent was evaporated and the resulting oil was chromatographed on a silica-gel column, yielding the title compound (0.51 g, 77%). M.p. 353–354 K.

Refinement

All H atoms were geometrically positioned (C–H 0.93–0.98%Å) and treated as riding, with $\text{Uiso}(\text{H}) = 1.2\text{Ueq}(\text{C})$.

Figures

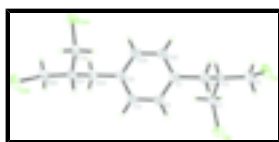


Fig. 1. The molecular structure of (I) showing the atom-labelling scheme and 30% probability displacement ellipsoids [symmetry code: (A) $-x, -y + 2, -z + 1$].

1,4-Bis[3-chloro-2-(chloromethyl)propyl]benzene

Crystal data

$\text{C}_{14}\text{H}_{18}\text{Cl}_4$

$M_r = 328.08$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 6.518$ (3) Å

$b = 14.680$ (6) Å

$c = 8.433$ (4) Å

$F_{000} = 340$

$D_x = 1.407$ Mg m $^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1607 reflections

$\theta = 2.8$ – 26.1°

$\mu = 0.75$ mm $^{-1}$

$T = 291$ K

supplementary materials

$\beta = 106.335 (5)^\circ$
 $V = 774.3 (6) \text{ \AA}^3$
 $Z = 2$

Block, colourless
 $0.30 \times 0.26 \times 0.24 \text{ mm}$

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: sealed tube
Monochromator: graphite
 $T = 291 \text{ K}$
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.807$, $T_{\max} = 0.842$
4423 measured reflections

1679 independent reflections
1275 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.066$
 $\theta_{\max} = 27.0^\circ$
 $\theta_{\min} = 2.8^\circ$
 $h = -8 \rightarrow 7$
 $k = -18 \rightarrow 14$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.113$
 $S = 1.09$
1679 reflections
82 parameters
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.0345P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.32 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.32 \text{ e \AA}^{-3}$
Extinction correction: none

Special details

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.

The structures were solved with direct methods and refined with full-matrix least-squares techniques using the *SHELXTL*. The coordinates of the non-hydrogen atoms were refined anisotropically, and the positions of the H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2 \text{ times } U_{\text{eq}}(\text{C})$.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1873 (3)	0.97482 (12)	0.6208 (2)	0.0415 (4)
C2	0.0205 (4)	1.01687 (13)	0.6634 (2)	0.0482 (5)
H2	0.0327	1.0284	0.7741	0.058*
C3	-0.1637 (3)	1.04196 (13)	0.5447 (3)	0.0477 (5)
H3	-0.2733	1.0705	0.5764	0.057*
C4	0.3901 (3)	0.94730 (13)	0.7505 (3)	0.0489 (5)
H7A	0.3999	0.9812	0.8511	0.059*
H7B	0.5123	0.9638	0.7122	0.059*
C5	0.4008 (3)	0.84467 (13)	0.7898 (2)	0.0410 (4)
H8	0.3984	0.8123	0.6877	0.049*
C6	0.6155 (3)	0.82501 (15)	0.9136 (3)	0.0543 (5)
H9A	0.6174	0.8515	1.0194	0.065*
H9B	0.7275	0.8539	0.8764	0.065*
C7	0.2135 (3)	0.81085 (13)	0.8444 (2)	0.0444 (5)
H10A	0.2273	0.7456	0.8629	0.053*
H10B	0.0828	0.8218	0.7571	0.053*
Cl1	0.67066 (10)	0.70596 (4)	0.94037 (8)	0.0736 (3)
Cl2	0.19547 (9)	0.86582 (4)	1.03025 (7)	0.0635 (2)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0465 (11)	0.0298 (9)	0.0488 (11)	-0.0015 (8)	0.0141 (9)	0.0050 (8)
C2	0.0586 (13)	0.0437 (11)	0.0440 (11)	0.0046 (9)	0.0172 (10)	-0.0002 (8)
C3	0.0535 (12)	0.0389 (11)	0.0560 (12)	0.0063 (9)	0.0241 (10)	0.0036 (9)
C4	0.0424 (11)	0.0438 (11)	0.0570 (12)	-0.0062 (8)	0.0082 (9)	0.0026 (9)
C5	0.0389 (10)	0.0396 (10)	0.0443 (10)	0.0017 (8)	0.0114 (8)	-0.0017 (8)
C6	0.0368 (11)	0.0513 (12)	0.0718 (14)	0.0052 (9)	0.0101 (10)	0.0022 (10)
C7	0.0397 (11)	0.0414 (11)	0.0480 (11)	-0.0012 (8)	0.0058 (9)	0.0009 (8)
Cl1	0.0629 (4)	0.0603 (4)	0.0915 (5)	0.0227 (3)	0.0119 (3)	0.0086 (3)
Cl2	0.0614 (4)	0.0795 (4)	0.0538 (4)	0.0101 (3)	0.0228 (3)	0.0004 (3)

Geometric parameters (\AA , $^\circ$)

C1—C3 ⁱ	1.382 (3)	C5—C7	1.504 (3)
C1—C2	1.383 (3)	C5—C6	1.520 (3)
C1—C4	1.515 (3)	C5—H8	0.9800
C2—C3	1.380 (3)	C6—Cl1	1.786 (2)
C2—H2	0.9300	C6—H9A	0.9700
C3—C1 ⁱ	1.382 (3)	C6—H9B	0.9700
C3—H3	0.9300	C7—Cl2	1.796 (2)
C4—C5	1.540 (3)	C7—H10A	0.9700
C4—H7A	0.9700	C7—H10B	0.9700
C4—H7B	0.9700		

supplementary materials

C3 ⁱ —C1—C2	118.01 (19)	C6—C5—C4	108.10 (16)
C3 ⁱ —C1—C4	120.55 (19)	C7—C5—H8	107.2
C2—C1—C4	121.44 (18)	C6—C5—H8	107.2
C3—C2—C1	121.17 (19)	C4—C5—H8	107.2
C3—C2—H2	119.4	C5—C6—C11	112.74 (15)
C1—C2—H2	119.4	C5—C6—H9A	109.0
C2—C3—C1 ⁱ	120.82 (19)	C11—C6—H9A	109.0
C2—C3—H3	119.6	C5—C6—H9B	109.0
C1 ⁱ —C3—H3	119.6	C11—C6—H9B	109.0
C1—C4—C5	113.20 (15)	H9A—C6—H9B	107.8
C1—C4—H7A	108.9	C5—C7—C12	112.11 (13)
C5—C4—H7A	108.9	C5—C7—H10A	109.2
C1—C4—H7B	108.9	C12—C7—H10A	109.2
C5—C4—H7B	108.9	C5—C7—H10B	109.2
H7A—C4—H7B	107.8	C12—C7—H10B	109.2
C7—C5—C6	113.40 (16)	H10A—C7—H10B	107.9
C7—C5—C4	113.42 (15)		
C3 ⁱ —C1—C2—C3	-0.4 (3)	C1—C4—C5—C6	176.91 (17)
C4—C1—C2—C3	179.85 (17)	C7—C5—C6—C11	64.6 (2)
C1—C2—C3—C1 ⁱ	0.4 (3)	C4—C5—C6—C11	-168.74 (14)
C3 ⁱ —C1—C4—C5	-77.5 (2)	C6—C5—C7—C12	62.71 (19)
C2—C1—C4—C5	102.2 (2)	C4—C5—C7—C12	-61.10 (19)
C1—C4—C5—C7	-56.4 (2)		

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

Fig. 1

