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1,16-Dichlorohexadecane

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In the title compound, $C_{16}H_{32}Cl_2$, the molecule is centrosymmetric and the molecular skeleton including both terminal Cl atoms has an all-*trans* conformation. The molecules form layers with a thickness of *c*/2. These features are similar to the tilt smectic C phase of liquid crystals.

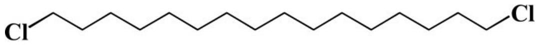
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.120
Data-to-parameter ratio = 16.3

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

Comment

The crystal structures of many long-chain aliphatic compounds, such as *n*-alkanes (*e.g.* Nyburg & Gerson, 1992), *n*-higher primary alcohols (*e.g.* Michaud *et al.*, 2000) and α,ω-disubstituted *n*-alkanes, have been studied not only from the view-point of basic polymer science but also as models for smectic liquid crystals. The reason is that the long-chain compounds have an extended rod-like hydrocarbon chain. In addition, the molecular arrangement in the crystalline state forms a layer structure, as in liquid crystals.



(I)

The crystal structures of 1,12-dibromododecane (Kulpe *et al.*, 1981) and 1,26-dibromohexacosane (Takamizawa *et al.*, 1992) have already been analyzed by other workers. In addition, we have also analysed 1,14-dibromotetradecane (Uno & Nakamura, 2003), 1,16-dibromohexadecane (Kobayashi *et al.*, 1995) and 1,18-dibromooctadecane (Nakamura *et al.*, 1993). The crystal system of each of these α,ω-dibromoalkanes is monoclinic, and the molecules are arranged in a herring-bone motif. For the α,ω-dichloroalkanes, only the lattice parameters of 1,20-dichloroicosane and 1,26-dichlorohexacosane have been determined (Takamizawa *et al.*, 1992). To obtain more extensive crystal data of the homologs with an even number of C atoms, the crystal structure analysis of 1,16-dichlorohexadecane, (I), has been carried out.

The molecular structure of (I) is shown in Fig. 1. The molecule is centrosymmetric and all torsion angles involving non-H atoms are close to ±180°, so that the molecular skeleton including both terminal Cl atoms has an all-*trans* conformation. The projection of the crystal structure of (I) along the *b* axis is shown in Fig. 2. The molecules form layers with a thickness of *c*/2. As shown in Fig. 3, the molecules are arranged in a zigzag manner between adjacent layers, forming a herring-bone motif. The molecular arrangement of (I) is similar to that of the tilt smectic C phase of liquid crystals. In the crystal structure, the shortest Cl···Cl distance is 3.697 (2) Å. The long axes of the molecules are inclined at 37.5 (1)° with respect to the *ab* plane. The dihedral angle between the *trans* zigzag planes of (I) in a layer and those in a

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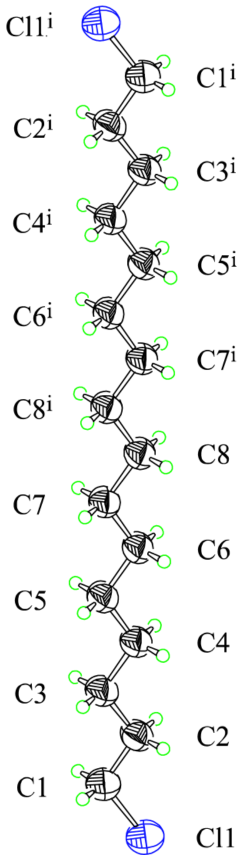

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $1 - x, 2 - y, 1 - z$.]

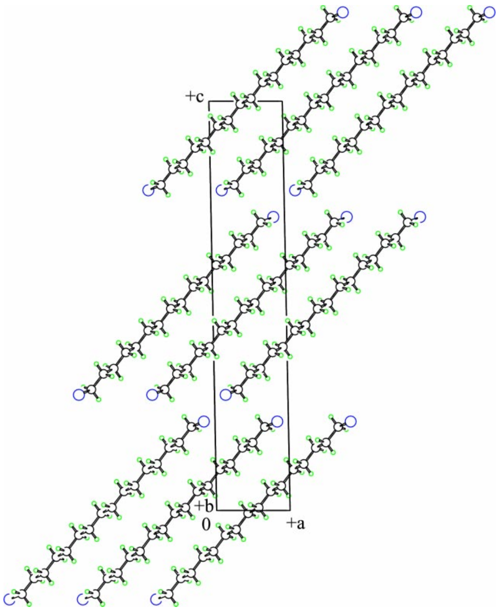

Figure 2
Projection of the crystal structure of (I) along the *b* axis.

neighbouring layer is 35.0 (2)°. In the crystalline state, molecules of alkane-α,ω-diols with an even number of C atoms also make a herring-bone motif. The crystal structure of (I) is similar to those of even-numbered alkane-α,ω-diols, but the

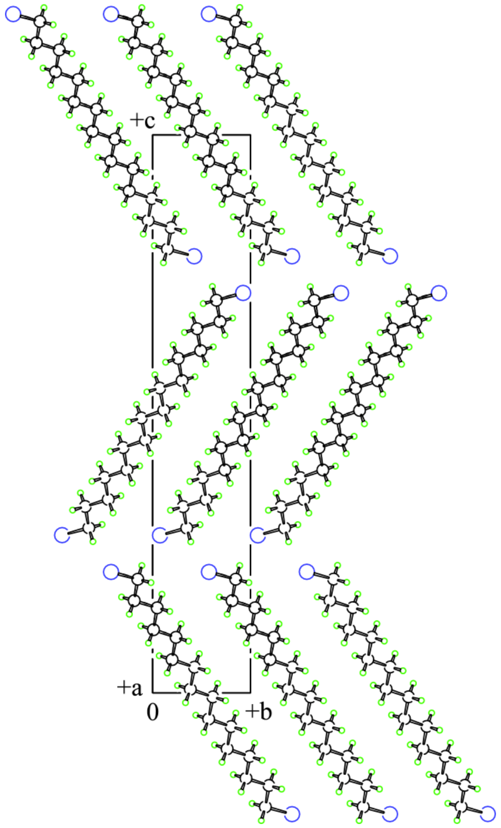

Figure 3
The projection of the crystal structure of (I) along the *a* axis.

dihedral angle of (I) mentioned above is larger than those of even-numbered alkane-α,ω-diols [*e.g.* 1,16-hexadecanediol: 4.1 (2)°; Nakamura & Yamamoto, 1994]. This difference of dihedral angles is influenced by interlayer interactions, *i.e.* the molecules of even-numbered alkane-α,ω-diols form hydrogen bonds but those of (I) do not. As a result, the crystal structure of (I) is similar to those of α,ω-dibromoalkanes and alkane-α,ω-diols with an even number of C atoms and these could be regarded as model compounds for the tilt smectic C phase of liquid crystals.

Experimental

The title compound, (I), was synthesized from commercially available 1,16-hexadecanedioic acid (Tokyo Kasei Kogyo Co. Ltd) by esterification, reduction and chlorination. The pure compound was obtained through fractional distillation and recrystallization. The single-crystal of (I) used for the analysis was obtained by slow evaporation of a solution in a mixture of 1,2-dichloroethane and 2-propanol (1:1).

Crystal data

$C_{16}H_{32}Cl_2$
$M_r = 295.32$
Monoclinic, $P2_1/c$
$a = 5.458$ (2) Å
$b = 5.3326$ (19) Å
$c = 30.4527$ (12) Å
$\beta = 91.102$ (13)°
$V = 886.2$ (5) Å^3
$Z = 2$
$D_x = 1.107$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 20 reflections
$\theta = 9.4$–$19.7°$
$\mu = 3.15$ mm^{-1}
$T = 296$ (1) K
Plate, colorless
$0.68 \times 0.26 \times 0.01$ mm

Data collection

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min} = 0.622$, $T_{\max} = 0.975$
2419 measured reflections
1613 independent reflections
715 reflections with $F^2 > 2\sigma(F^2)$

$R_{\text{int}} = 0.035$
$\theta_{\max} = 70.2°$
$h = -1 \rightarrow 6$
$k = 0 \rightarrow 6$
$l = -37 \rightarrow 37$
3 standard reflections
every 150 reflections
intensity decay: 6.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.120$
$S = 1.00$
1613 reflections
99 parameters
H-atom parameters constrained

$w = 4F_o^2/[0.0001F_o^2 + \sigma^2(F_o) + 0.1]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.62$ e Å^{-3}
$\Delta\rho_{\min} = -0.72$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 35.6 (2)

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.771 (4)	C5—C6	1.529 (4)
C1—C2	1.492 (4)	C6—C7	1.507 (4)
C2—C3	1.512 (5)	C7—C8	1.517 (4)
C3—C4	1.511 (4)	C8—C8$^{\text{i}}$	1.514 (5)
C4—C5	1.502 (4)		
Cl1—C1—C2—C3	−177.6 (2)	C4—C5—C6—C7	179.1 (3)
C1—C2—C3—C4	179.1 (3)	C5—C6—C7—C8	−179.9 (3)
C2—C3—C4—C5	−179.8 (3)	C6—C7—C8—C8$^{\text{i}}$	−179.7 (3)
C3—C4—C5—C6	179.8 (3)	C7—C8—C8$^{\text{i}}$—C7$^{\text{i}}$	180.0

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

Table 2
Contact distances (Å).

Cl1···Cl1$^{\text{ii}}$	3.697 (2)	Cl1···Cl1$^{\text{iii}}$	3.697 (2)

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

All H atoms were placed at idealized positions and allowed to ride on their parent C atoms (C—H = 0.95 Å). The isotropic displacement parameters of the H atoms were fixed at $1.2U_{\text{eq}}$ of the parent C atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Rigaku/MSC, 2001); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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