

## 1,20-Dibromoicosane

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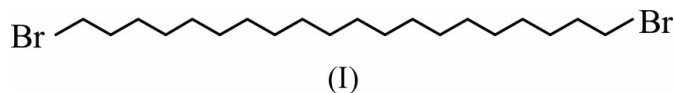
## Key indicators

Single-crystal X-ray study  
 $T = 296\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.038  
 $wR$  factor = 0.116  
Data-to-parameter ratio = 16.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of the title compound,  $\text{C}_{20}\text{H}_{40}\text{Br}_2$ , is centrosymmetric and the skeleton has an all-*trans* conformation including both terminal Br atoms. In the crystal structure, the molecules form a layer in which the long axes of the molecules are inclined to the basal plane of the Br atoms. The layers are arranged in a zigzag manner between the neighboring layers, forming a herringbone motif, just like the smectic  $C_A$  phase of liquid crystals.

## Comment

Normal long-chain aliphatic compounds, for example *n*-alkanes, have been studied to elucidate the principles of organic chemical crystallography and basic polymer science, because the molecular skeleton consists of a simple *trans* zigzag straight hydrocarbon chain. The molecular shape of these compounds can be regarded as rod-like, and the molecules in the crystalline state form a layered structure similar to those of the smectic liquid crystalline phase. Moreover, some of these compounds exhibit a high-temperature rotator phase just below their melting points, in which molecules have motional freedom to some degree as well as that in liquid crystals. As a result, normal long-chain aliphatic compounds have also been investigated as models for smectic liquid crystals.



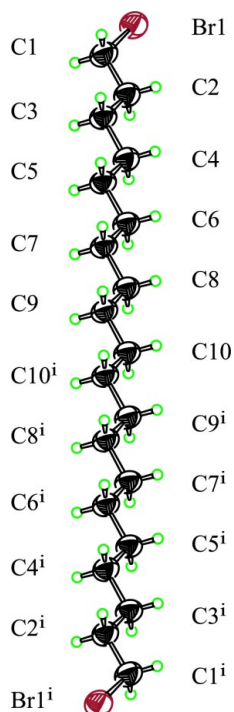
In these investigations, it is important to obtain detailed crystallographic data. Many researchers have analyzed the crystal structures of many different kinds of normal long-chain aliphatic compounds, for example, *n*-alkanes (*e.g.* Nyburg & Gerson, 1992), *n*-primary alcohols (*e.g.* Michaud *et al.*, 2000), and  $\alpha,\omega$ -disubstituted *n*-alkanes, such as 1,12-dibromododecane (Kulpe *et al.*, 1981) and 11-bromoundecan-1-ol (Rosen & Hybl, 1972). Recently, we have systematically analysed the crystal structures of the alkane- $\alpha,\omega$ -diols containing 10–19 and 21–23 C atoms (Nakamura *et al.*, 2001; Uno *et al.*, 2002), and we have studied the phase-transition phenomena of the series of the alkane- $\alpha,\omega$ -diols containing 13–24 C atoms (Ogawa & Nakamura, 1999). In the present paper, we report the crystal structure analysis of the title compound, (I), in order to elucidate the effect of the terminal groups in normal long-chain aliphatic compounds.

The molecular structure of (I) is shown in Fig. 1. The molecule is centrosymmetric and all torsion angles are close to  $\pm 180^\circ$ , that is, the molecular skeleton including both terminal

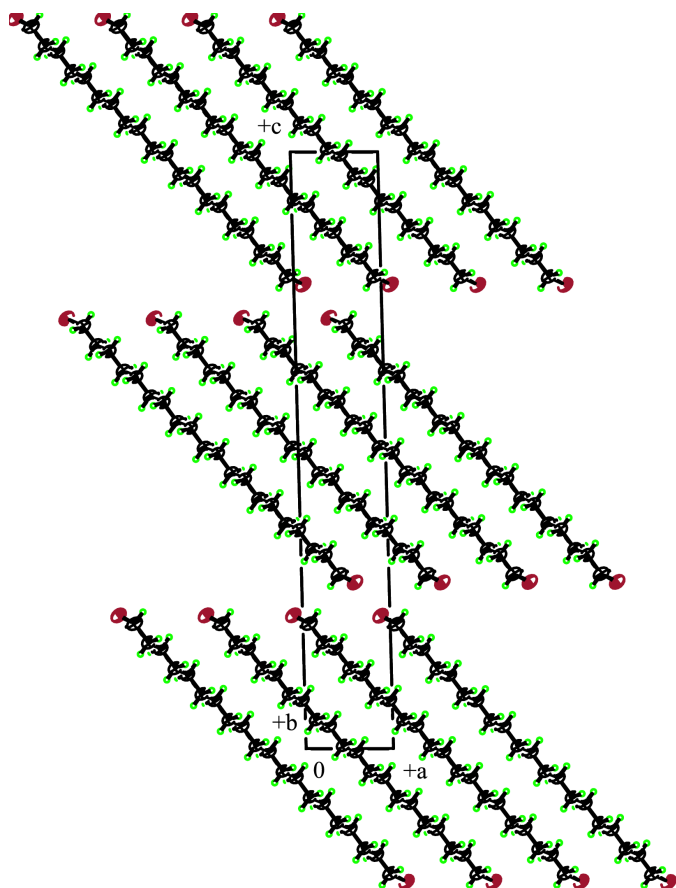
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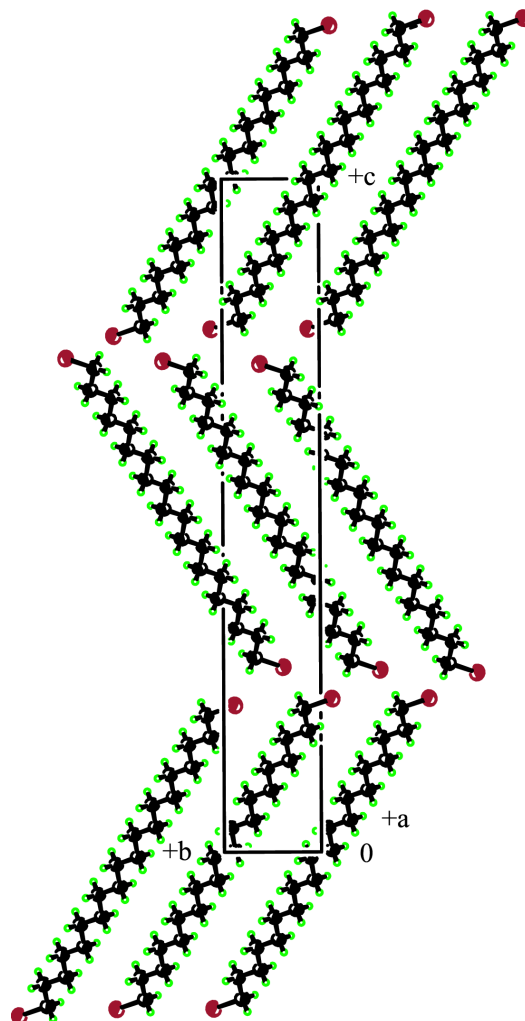
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**Figure 1**  
The molecular structure of (I), showing the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i)  $2 - x, -y, -z$ .]



**Figure 2**  
The projection of the crystal structure of (I) along the  $b$  axis.



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

Br atoms has an all-*trans* conformation. Fig. 2 shows the projection of the crystal structure of (I) along the  $b$  axis. The molecules form layers with a thickness of  $c/2$ . In the layer, the long axes of the molecules are inclined to the  $ab$  plane. This layer structure is similar to that of the triclinic structure of the even-numbered  $n$ -alkanes containing 6–24 C atoms. It is considered that the arrangement in the layer is influenced by the steric and electrostatic repulsion of Br atoms at both ends. As a result, a molecular position in the layer slides along the direction of the long axis of the neighboring molecule.

Moreover, the layers are arranged in a zigzag manner between the neighboring layers, forming a herringbone motif similar to the tilt-smectic C phase and the smectic  $C_A$  phase of liquid crystals, as shown in Fig. 3. The layers are stacked closely in such a way that the  $\alpha$ -CH<sub>2</sub> groups are allowed to fit into the grooves formed by Br atoms, with nearest contacts of 3.752 (3) Å, agreeing closely with the van der Waals contacts of 3.75 Å (Rowland & Taylor, 1996). Such a close packing is observed in the even-numbered alkane- $\alpha,\omega$ -diols containing 4–19 and 21–23 C atoms (*e.g.* Thalladi *et al.*, 2000) and the  $\alpha,\omega$ -dichloroalkanes containing 16, 20 and 26 C atoms (Takami-zawa *et al.*, 1992).

The features in the molecular and crystal structure of (I) are similar to those of the homologous series with an even number of C atoms, *viz.* 1,12-dibromododecane (Kulpe *et al.*, 1981), 1,14-dibromotetradecane (Uno & Nakamura, 2003), 1,16-dibromohexadecane (Kobayashi *et al.*, 1995), 1,18-dibromooctadecane (Nakamura *et al.*, 1993), and 1,26-dibromohexadecane (Takamizawa *et al.*, 1992).

### Experimental

Thin plate-like crystals of (I) were grown by slow evaporation of a solution containing a mixture of *n*-hexane and ethanol (1:1). The well developed face of the crystal is (001).

#### Crystal data

C <sub>20</sub> H <sub>40</sub> Br <sub>2</sub>	$D_x = 1.325 \text{ Mg m}^{-3}$
$M_r = 440.34$	Cu $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 24 reflections
$a = 5.482 (4) \text{ \AA}$	$\theta = 9.5\text{--}15.5^\circ$
$b = 5.383 (2) \text{ \AA}$	$\mu = 4.61 \text{ mm}^{-1}$
$c = 37.412 (2) \text{ \AA}$	$T = 296 (1) \text{ K}$
$\beta = 91.50 (2)^\circ$	Plate, colorless
$V = 1103.6 (9) \text{ \AA}^3$	$0.59 \times 0.47 \times 0.04 \text{ mm}$
$Z = 2$	

#### Data collection

Rigaku AFC-5R diffractometer	$R_{\text{int}} = 0.028$
$\omega$ - $2\theta$ scans	$\theta_{\text{max}} = 70.1^\circ$
Absorption correction: by integration (NUMABS; Higashi, 1999)	$h = -1 \rightarrow 6$
$T_{\text{min}} = 0.325$ , $T_{\text{max}} = 0.997$	$k = 0 \rightarrow 6$
3004 measured reflections	$l = -45 \rightarrow 45$
2008 independent reflections	3 standard reflections
1667 reflections with $F^2 > 2\sigma(F^2)$	every 150 reflections
	intensity decay: 6.8%

#### Refinement

Refinement on $F^2$	$w = (4F_o^2)/[0.001F_o^2 + 5.3\sigma^2(F_o) + 0.52]$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$wR(F^2) = 0.116$	$\Delta\rho_{\text{max}} = 0.38 \text{ e \AA}^{-3}$
$S = 1.00$	$\Delta\rho_{\text{min}} = -0.62 \text{ e \AA}^{-3}$
2008 reflections	Extinction correction: (Larson, 1970)
121 parameters	Extinction coefficient: 21.3 (3)
H-atom parameters constrained	

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Br1—C1	1.946 (4)	C6—C7	1.510 (5)
C1—C2	1.489 (5)	C7—C8	1.514 (4)
C2—C3	1.514 (5)	C8—C9	1.509 (5)
C3—C4	1.526 (5)	C9—C10	1.518 (4)
C4—C5	1.514 (5)	C10—C10 <sup>ii</sup>	1.507 (5)
C5—C6	1.522 (4)		
Br1—C1—C2—C3	-179.6 (2)	C5—C6—C7—C8	179.2 (3)
C1—C2—C3—C4	179.4 (3)	C6—C7—C8—C9	179.8 (3)
C2—C3—C4—C5	179.7 (3)	C7—C8—C9—C10	-179.8 (3)
C3—C4—C5—C6	-179.7 (3)	C8—C9—C10—C10 <sup>ii</sup>	179.7 (3)
C4—C5—C6—C7	178.8 (3)	C9—C10—C10 <sup>ii</sup> —C9 <sup>ii</sup>	180.0 (3)

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

**Table 2**

Intermolecular contacts ( $\text{\AA}$ ).

Br1 $\cdots$ Br1 <sup>iii</sup>	3.752 (3)	Br1 $\cdots$ Br1 <sup>iv</sup>	3.752 (3)
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Symmetry codes: (iii)  $\frac{y}{2} - x, y - \frac{1}{2}, \frac{z}{2} - z$ ; (iv)  $\frac{y}{2} - x, \frac{1}{2} + y, \frac{z}{2} - z$ .

H atoms were positioned geometrically and treated as riding, with C—H distances of 0.95  $\text{\AA}$  and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$ .

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Molecular Structure Corporation & Rigaku, 2001); program(s) used to solve structure: *SIR92* (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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