Data collection

Kuma KM-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1603 measured reflections
1603 independent reflections
1436 reflections with
  $I > 2\sigma(I)$

$\theta_{\max} = 68.10°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 19$
2 standard reflections
  every 100 reflections
  intensity decay: <2.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.101$
$S = 1.010$
1603 reflections
200 parameters
H atoms fixed
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 0.2880P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.169$ e Å^{-3}
$\Delta\rho_{\min} = -0.166$ e Å^{-3}
Extinction correction:
  *SHELX*97 (Sheldrick, 1997)
Extinction coefficient:
  0.0053 (7)
Scattering factors from
  International Tables for Crystallography (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H12···O6^{i}	0.97	2.68	3.340 (3)	125
C2—H21···O17^{ii}	0.97	2.50	3.464 (3)	172
C2—H22···O6^{i}	0.97	2.74	3.409 (4)	127
C16—H161···O17^{iii}	0.97	2.67	3.434 (4)	136
C16—H162···O3^{iv}	0.97	2.63	3.525 (4)	154
C18—H181···O17^{iii}	0.96	2.61	3.514 (4)	156
C19—H192···O17^{ii}	0.96	2.71	3.515 (4)	141

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

Data collection: *KM*-4 *Software* (Kuma Diffraction, 1989). Cell refinement: *KM*-4 *Software*. Data reduction: *KM*-4 *Software*. Program(s) used to solve structure: *SHELX*97 (Sheldrick, 1997). Program(s) used to refine structure: *SHELX*97. Molecular graphics: *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELX*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1312). Services for accessing these data are described at the back of the journal.

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1,11-Undecanediol

Naotake Nakamura, Shigetaka Setodoi and
Tatsuhiro Ikeya

Department of Chemistry, Faculty of Science and Engineering, Ritsumeikan University, 1-1-1, Nojihigashi, Kusatsu, Shiga 525-8577, Japan. E-mail: nakamura@se.ritsumei.ac.jp

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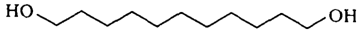
Abstract

The crystal structure analysis of 1,11-undecanediol, $C_{11}H_{24}O_2$, has been carried out by X-ray diffraction. The hydrocarbon skeleton has an all-*trans* conformation. One of the hydroxyl groups, which are located at both ends of the hydrocarbon chain, has a *gauche* conformation with respect to the skeleton, whereas the other has a *trans* conformation. The molecules lie parallel to the *b* axis and layers are formed with a thickness of *b*/2. The molecules are arranged in an antiparallel fashion along the *a* axis in these layers. These features are similar to those of the homologues with an odd number of C atoms, but different from those with an even number.

Comment

Generally, long-chain aliphatic compounds have been studied as basic models of polymers. These compounds have a simple chemical structure whose molecular skeleton is a straight hydrocarbon chain. Thus, the molecular shape could be regarded as a rod-like one. These compounds also show a layer structure just like the smectic one of liquid crystals. Recently, these compounds have been of great interest as model compounds of liquid crystals (*e.g.* Nakamura & Yamamoto, 1994; Kobayashi & Nakamura, 1995). Up to the present, crystal structures of many different kinds of long-chain compounds, for example, *n*-alkanes, α-monosubstituted *n*-alkanes such as *n*-higher alcohols, and α-monohalogenoalkanes have been studied (*e.g.* Watanabe, 1961; Seto, 1962). However, there are few reports about α,ω-disubstituted *n*-alkanes. Quite recently, we reported crystal structures of three α,ω-alkanediols: 1,12-dodecanediol (Nakamura & Setodoi, 1997), 1,13-tridecanediol (Nakamura *et al.*, 1997) and 1,16-hexadecanediol (Nakamura & Yamamoto, 1994).

The molecular structure of 1,11-undecanediol, (I), is shown in Fig. 1. The hydrocarbon skeleton has an all-*trans* conformation. The terminal C1—O1 bond is *gauche* with respect to the skeleton [O1—C1—C2—C3 −63.3 (3)°], whereas the other terminal C11—O2 bond is *trans* [O2—C11—C10—C9 179.3 (2)°]. The *gauche* conformation of the hydroxyl group has been observed in 1-heptadecanol (Seto, 1962). The molecules lie parallel to the *b* axis and layers are formed with a thickness of *b*/2. The molecules are arranged in an antiparallel fashion along the *a* axis in these layers, as can be seen in Fig. 2. This packing is very similar to the smectic A structure of liquid crystals. In this structure, the molecules form two different types of hydrogen bond, *i.e.* interlayer and intralayer hydrogen bonds. These features are quite similar to those of 1,13-tridecanediol. The interlayer hydrogen-bond distance O1···O2 is 2.710 (2) Å and the O2—H2O···O1 angle is 172.0°. The intralayer hydrogen-bond distance O1···O2 is 2.775 (3) Å and the O1—H1O···O2 angle is 173.9°. These values are in good agreement with those of 1,13-tridecanediol [2.713 (2) and 2.776 (4) Å].



(I)

On the other hand, we reported in our previous papers that the homologues with an even number of C atoms (1,12-dodecanediol and 1,16-hexadecanediol) had different molecular and crystal structures. The hydrocarbon skeleton had the all-*trans* conformation, and both terminal C—O bonds also showed the *trans* conformation. The centrosymmetric molecules are arranged in a zigzag manner to make a herring-bone motif, and could be regarded as model structures of the smectic C liquid crystals. 1,12-Dibromododecane (Kuple *et al.*, 1981) and 1,16-dibromohexadecane (Kobayashi *et al.*, 1995) also show the herring-bone motif. In these structures, the molecules have only interlayer hydrogen bonds.

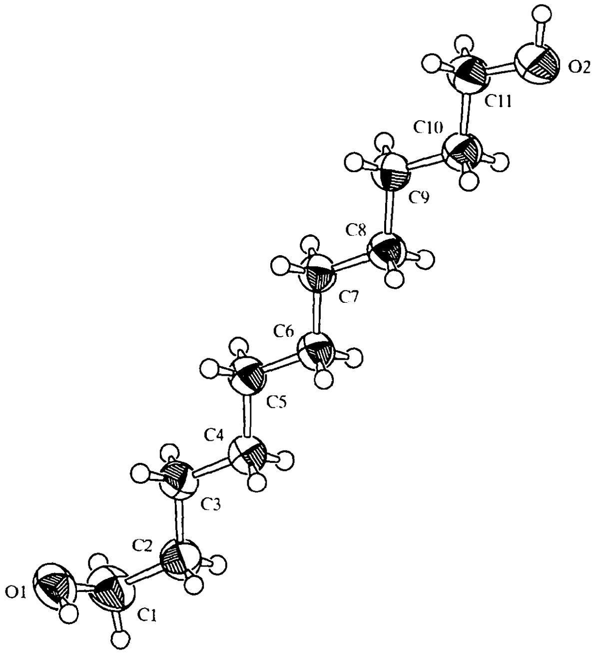


Fig. 1. An *ORTEP*II (Johnson, 1976) view of the title molecule showing the crystallographic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

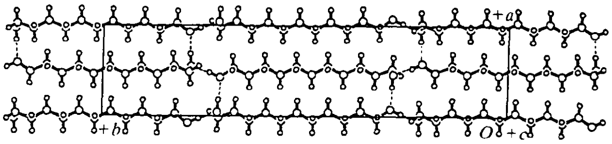


Fig. 2. The projection of the crystal structure along the *c* axis. Dotted lines indicate the hydrogen bonds.

Experimental

According to the conventional method, the title compound was synthesized from commercially available undecanedioic acid (Tokyo Kasei Kogyo Co., Ltd) by esterification and reduction with $LiAlH_4$. The single crystal used for analysis was grown by slow evaporation from a solution with a mixed solvent of methanol and hexane (1:1).

Crystal data

$C_{11}H_{24}O_2$
$M_r = 188.31$
Orthorhombic
$P2_12_12_1$
$a = 7.145\,(4)$ Å
$b = 32.419\,(9)$ Å
$c = 5.142\,(2)$ Å
$V = 1191.1\,(9)$ Å^3
$Z = 4$
$D_x = 1.050$ Mg m^{-3}
$D_m = 1.049$ Mg m^{-3}
D_m measured by flotation in aqueous KI

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 5.1$–$13.2°$
$\mu = 0.543$ mm^{-1}
$T = 298.2$ K
Plate-like
$0.8 \times 0.6 \times 0.1$ mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North *et al.*, 1968)
$T_{\min} = 0.661$, $T_{\max} = 0.947$
3541 measured reflections
1736 independent reflections
1341 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.035$
$\theta_{\max} = 70°$
$h = -5 \rightarrow 8$
$k = 0 \rightarrow 39$
$l = -3 \rightarrow 6$
3 standard reflections every 150 reflections intensity decay: 3.8%

Refinement

Refinement on F
$R = 0.05$
$wR = 0.070$
$S = 1.380$
1341 reflections
118 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.00141|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.0004$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C1	1.417 (3)	C5—C6	1.519 (3)
O2—C11	1.421 (3)	C6—C7	1.512 (3)
C1—C2	1.502 (3)	C7—C8	1.511 (3)
C2—C3	1.517 (3)	C8—C9	1.516 (3)
C3—C4	1.518 (3)	C9—C10	1.511 (3)
C4—C5	1.508 (3)	C10—C11	1.500 (3)
O1—C1—C2	112.7 (2)	C6—C7—C8	113.6 (2)
C1—C2—C3	114.6 (2)	C7—C8—C9	115.1 (2)
C2—C3—C4	112.7 (2)	C8—C9—C10	112.8 (2)
C3—C4—C5	114.7 (2)	C9—C10—C11	114.4 (2)
C4—C5—C6	113.7 (2)	O2—C11—C10	109.0 (2)
C5—C6—C7	114.8 (2)		
O1—C1—C2—C3	−63.3 (3)	O2—C11—C10—C9	179.3 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2O···O1$^{\text{i}}$	0.96	1.76	2.710 (2)	172.0
O1—H1O···O2$^{\text{ii}}$	0.97	1.81	2.775 (3)	173.9

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

All non-H atoms were refined anisotropically by full-matrix least-squares methods. All H atoms including hydroxyl H atoms were fixed in idealized positions.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SAPI91* (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1004). Services for accessing these data are described at the back of the journal.

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Tautomerism in a 2,4-dichlorobenzenesulfonamide derived from 2-amino-4-(2,5-dimethoxyphenyl)thiazole

Pierre Beuchet,[a] Jean-Michel Léger,[b] Martine Varache-Lembège[a] and Alain Nuhrich[a]

[a]*Laboratoire de Pharmacie Chimique et Chimie Thérapeutique, UFR des Sciences Pharmaceutiques, 3 place de la Victoire, 33076 Bordeaux CEDEX, France, and* [b]*Laboratoire de Chimie Analytique, UFR des Sciences Pharmaceutiques, 3 place de la Victoire, 33076 Bordeaux CEDEX, France. E-mail: pbeuchet@rockefeller.univ-lyon1.fr*

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Abstract

2,4-Dichloro-*N*-[2,3-dihydro-4-(2,5-dimethoxyphenyl)thiazol-2-ylidene]benzenesulfonamide, $C_{17}H_{14}Cl_2N_2O_4S_2$, crystallizes in space group $P\bar{1}$ with two independent molecules. The structural parameters do not indicate a tautomeric equilibrium but a single imino form. The main differences between the two crystalline forms lie in the intramolecular hydrogen bonding and the relative orientation of the methoxy groups. Attractive intermolecular interactions occur and are responsible for the crystalline cohesion.

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