C4	0.3402 (11)	0.7681 (4)	0.5973 (4)	0.043 (2)
C5	0.3692 (10)	0.8099 (4)	0.6703 (4)	0.039 (2)
C6	0.5525 (9)	0.8486 (4)	0.6755 (4)	0.047 (2)
C7	0.5704 (10)	0.9275 (4)	0.6344 (4)	0.039 (2)
C8	0.4260 (8)	0.9860 (4)	0.6568 (4)	0.033 (2)
C9	0.2438 (9)	0.9489 (4)	0.6450 (4)	0.034 (2)
C10	0.2225 (9)	0.8694 (4)	0.6900 (4)	0.034 (2)
C11	0.0938 (8)	1.0089 (3)	0.6562 (4)	0.040 (2)
C12	0.1232 (10)	1.0917 (4)	0.6207 (4)	0.043 (2)
C13	0.2983 (8)	1.1281 (4)	0.6412 (3)	0.031 (2)
C14	0.4389 (9)	1.0672 (4)	0.6193 (4)	0.031 (2)
C15	0.6122 (9)	1.1128 (4)	0.6268 (4)	0.044 (2)
C16	0.5611 (10)	1.2019 (4)	0.6098 (4)	0.042 (2)
C17	0.3611 (10)	1.2031 (4)	0.5987 (4)	0.033 (2)
C18	0.3033 (10)	1.1468 (4)	0.7229 (3)	0.049 (2)
C19	0.2311 (10)	0.8881 (4)	0.7720 (3)	0.050 (2)
C20	0.2786 (11)	1.2869 (4)	0.6154 (4)	0.040 (2)
C21	0.0806 (10)	1.2873 (4)	0.6023 (5)	0.052 (2)
C22	0.3696 (11)	1.3523 (3)	0.5721 (4)	0.044 (2)
C23	0.3055 (10)	1.4383 (4)	0.5887 (4)	0.051 (2)
C24	0.4218 (11)	1.5018 (4)	0.5590 (5)	0.046 (2)
C25	0.6982 (11)	1.5633 (5)	0.5753 (4)	0.072 (3)
O1	0.5659 (7)	0.9138 (3)	0.5588 (3)	0.0533 (15)
O2	0.5676 (8)	1.5074 (3)	0.5986 (3)	0.062 (2)
O3	0.3988 (7)	1.5419 (3)	0.5067 (3)	0.059 (2)

Table 2. *Selected geometric parameters (Å, °)*

Br—C3	1.990 (7)	C3—C4	1.487 (11)
C1—C2	1.513 (10)	C4—C5	1.538 (9)
C1—C10	1.531 (9)	C5—C10	1.535 (9)
C2—C3	1.513 (10)		
C2—C1—C10	115.2 (6)	C3—C4—C5	113.8 (7)
C3—C2—C1	111.6 (7)	C10—C5—C4	113.1 (7)
C4—C3—C2	111.3 (6)	C10—C5—C6	112.5 (5)
C4—C3—Br	110.4 (6)	C4—C5—C6	111.9 (7)
C2—C3—Br	109.5 (5)		
C10—C1—C2—C3	53.7 (8)	C3—C4—C5—C10	−52.4 (8)
C1—C2—C3—C4	−53.3 (8)	C2—C1—C10—C5	−50.5 (8)
C2—C3—C4—C5	53.6 (9)	C4—C5—C10—C1	48.3 (7)
Br—C3—C4—C5	−68.2 (7)		

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL*93 and *PARST* (Nardelli, 1983).

The X-ray diffraction experiments were carried out at the Diffraction National Laboratory (LANADI), La Plata, Argentina.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1305). Services for accessing these data are described at the back of the journal.

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1,12-Dodecanediol

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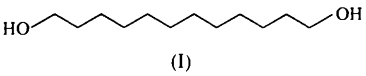
Abstract

The crystal structure analysis of 1,12-dodecanediol, $C_{12}H_{26}O_2$, has been carried out by X-ray diffraction. Both hydroxyl groups show a *trans* conformation with respect to the hydrocarbon skeleton. The structure, which adopts a herring-bone motif, appears to be common to α,ω-disubstituted long-chain alkanes with even numbers of C atoms. These structures could be regarded as model structures of the smectic C liquid crystals.

Comment

There are a few reports of the crystal structures of α,ω-alkanediols. Recently, one of the present authors analyzed the crystal structure of two long-chain α,ω-alkanediols. The structural feature of the compounds with an even number of C atoms (1,16-hexadecanediol) was a herring-bone motif with both hydroxyl groups located at the ends of the molecular skeleton having a *trans* conformation (Nakamura & Yamamoto, 1994). The homolog with an odd number of C atoms (1,13-tridecanediol) showed a parallel arrangement of the molecules similar to that of the smectic A liquid crystals, with one of the hydroxyl groups *gauche* with respect to the hydrocarbon skeleton, the other being *trans* (Nakamura, Tanihara & Takayama, 1997). These compounds have been of great interest recently as liquid crystalline model materials. In the present work, the crystal structure of the title compound, 1,12-dodecanediol, (I), has been determined.



(I)

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The molecule of (I) (Fig. 1) is centrosymmetric, with the terminal hydroxyl groups *trans*, similar to the case in 1,16-hexadecanediol (Nakamura & Yamamoto, 1994). The skeleton of the hydrocarbon chain packs in a zigzag fashion with the molecules linked by hydrogen-bonding $O1\cdots O1^{i}$ interactions of 2.823 (2) Å [symmetry code: (i) $2-x, -\frac{1}{2}+y, \frac{1}{2}-z$]. This value is in good agreement with that in 1,16-hexadecanediol for which the value is 2.839 Å (Yamamoto, 1993). This arrangement is different from that in 1,13-tridecanediol, in which one of the two hydroxyl groups adopts a *gauche* conformation as mentioned above. In 1,12-dodecanediol, the molecular arrangement along the longest axis is a zigzag, as shown in Fig. 2, and this is very similar to that of 1,16-hexadecanediol, but contrasts with that of 1,13-tridecanediol. As a result, the structure of α,ω-alkanediols with 12 and 16 C atoms closely resemble each other.

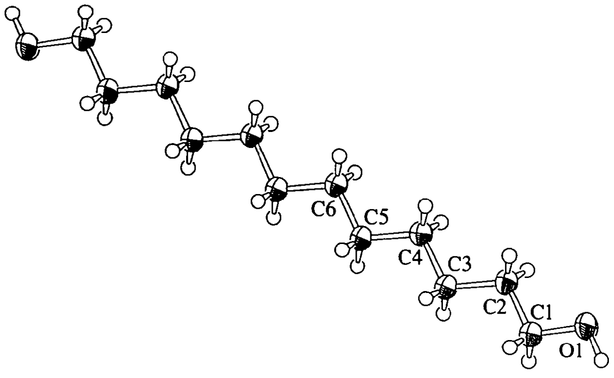

Fig. 1. An *ORTEP*II (Johnson, 1976) view of the title molecule, showing the crystallographic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

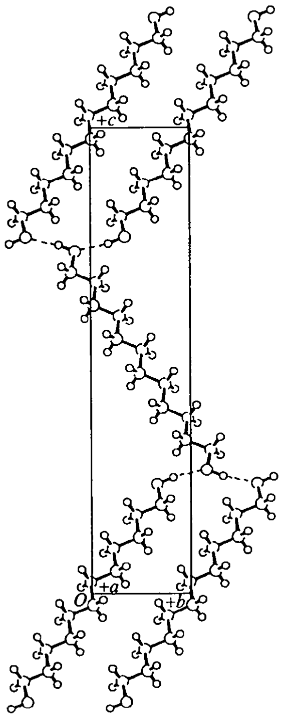

Fig. 2. Packing diagram viewed along the *a* axis. Dotted lines indicate the hydrogen-bonding interactions.

The crystal structures of 1,12-dibromododecane (Kulpe *et al.*, 1981) and 1,16-dibromohexadecane (Kobayashi, Yamamoto & Nakamura, 1995) both show a herring-bone motif and are very similar. These compounds closely resemble the structures of 1,12-dodecanediol and 1,16-hexadecanediol. Therefore, this kind of herring-bone-type structure may be common in α,ω-disubstituted long-chain compounds with even numbers of C atoms. It must be noted that these long-chain alkanes having the herring-bone motif may be regarded as model compounds of the smectic C liquid crystals.

Experimental

The single crystal of the title compound used for analysis was grown from a benzene solution by slow evaporation.

Crystal data

$C_{12}H_{26}O_2$
$M_r = 202.34$
Monoclinic
$P2_1/c$
$a = 4.964\ (3)$ Å
$b = 5.190\ (4)$ Å
$c = 24.497\ (3)$ Å
$\beta = 90.75\ (3)^\circ$
$V = 631.1\ (6)$ Å^3
$Z = 2$
$D_x = 1.065$ Mg m^{-3}
$D_m = 1.08\ (1)$ Mg m^{-3}
D_m measured by flotation in KI

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 13.9$–14.9°
$\mu = 0.069$ mm^{-1}
$T = 296.2$ K
Plate
$0.40 \times 0.30 \times 0.05$ mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.973$, $T_{\max} = 0.998$
1797 measured reflections
1620 independent reflections

748 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.053$
$\theta_{\max} = 27.49^\circ$
$h = 0 \rightarrow 6$
$k = -6 \rightarrow 0$
$l = -31 \rightarrow 31$
3 standard reflections every 150 reflections intensity decay: 0.20%

Refinement

Refinement on F
$R = 0.049$
$wR = 0.072$
$S = 1.270$
748 reflections
64 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.00235|F_o|^2]$

$(\Delta/\sigma)_{\max} = 0.0009$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($\AA^2$)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
O1	0.9148 (3)	1.1588 (2)	0.26522 (5)	0.0707 (4)
C1	0.7392 (3)	1.1986 (3)	0.30940 (6)	0.0500 (4)
C2	0.6462 (3)	0.9447 (3)	0.33149 (6)	0.0452 (4)
C3	0.4614 (3)	0.9713 (3)	0.37993 (6)	0.0443 (4)
C4	0.3659 (3)	0.7150 (3)	0.40273 (6)	0.0433 (4)
C5	0.1863 (3)	0.7417 (3)	0.45223 (6)	0.0439 (4)
C6	0.0898 (3)	0.4864 (3)	0.47527 (5)	0.0433 (4)

Table 2. *Selected geometric parameters* (Å, °)

O1—C1	1.413 (2)	C4—C5	1.521 (2)
C1—C2	1.499 (2)	C5—C6	1.520 (2)
C2—C3	1.515 (2)	C6—C6$^{\rm i}$	1.520 (2)
C3—C4	1.521 (2)		
O1—C1—C2	110.1 (1)	C3—C4—C5	113.7 (1)
C1—C2—C3	113.3 (1)	C4—C5—C6	114.0 (1)
C2—C3—C4	113.8 (1)	C5—C6—C6$^{\rm i}$	113.9 (2)

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

All non-H atoms were refined anisotropically by full-matrix least-squares methods. 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: *SHELXS*86 (Sheldrick, 1990). 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: TA1172). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1885–1887

[3-(4-Bromophenyl)-1-(4-methoxyphenyl)-pyrazol-5-yl]acetonitrile†

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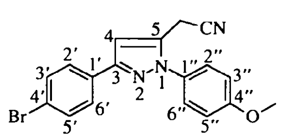
Abstract

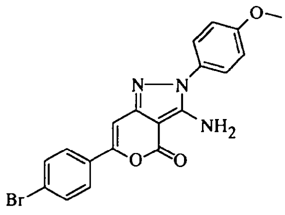
The title compound, $C_{18}H_{14}BrN_3O$, is one of the products obtained from the reaction of 6-(4-bromophenyl)-4-methylthio-2-oxo-2*H*-pyran-3-carbonitrile with 4-methoxyphenylhydrazine hydrochloride. The bromophenyl and methoxyphenyl groups are oriented with dihedral angles of 14.4 (2) and 53.8 (1)°, respectively, with respect to the pyrazole ring.

Comment

Pyrazoles have found applications in the areas of medicine and agriculture, and also in synthetic chemistry (Weily & Wiley, 1964; Taki *et al.*, 1992). Because of their widespread uses, intensive research efforts have been directed towards the development of new and improved synthetic routes for the preparation of pyrazole derivatives. We have synthesized several diphenylpyrazoleacetonitriles in order to extend the series for structure–activity studies.



(I)



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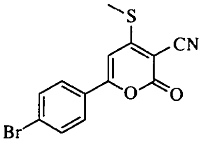

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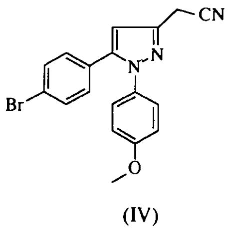

(IV)

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† Alternative systematic name: 5-(4-bromophenyl)-2-(4-methoxyphenyl)-3-pyrazoleacetonitrile.

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