Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.64$, $T_{\max} = 1.44$
2147 measured reflections
2052 independent reflections
1136 observed reflections $[I > 3\sigma(I)]$
$R_{\rm int} = 0.021$
$\theta_{\max} = 25°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 15$
2 standard reflections frequency: 30 min intensity variation: ±1.1%

Refinement

$R = 0.060$
$wR = 0.067$
$S = 2.06$
1136 reflections
192 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(|F_o|) + 0005|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Atomic scattering factors from *SHELX*76 (Sheldrick, 1976)

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

$B_{\rm eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$B_{\rm eq}$
N	0.9897 (5)	−0.1691 (3)	0.3651 (4)	5.5 (2)
O(1)	1.0093 (4)	−0.0953 (3)	0.2030 (3)	5.9 (2)
O(2)	0.9191 (5)	0.1227 (3)	0.1327 (3)	6.9 (2)
O(3)	0.7406 (4)	0.0022 (3)	0.1071 (3)	5.6 (1)
O(4)	0.6906 (6)	0.1612 (4)	0.4119 (4)	9.9 (2)
O(5)	0.6467 (6)	0.1944 (3)	0.2454 (3)	10.0 (2)
C(2)	0.9580 (6)	−0.0984 (4)	0.2909 (5)	4.7 (2)
C(3)	0.8522 (6)	−0.0224 (3)	0.3388 (4)	4.3 (2)
C(4)	0.7464 (7)	−0.0247 (4)	0.5306 (4)	5.8 (2)
C(5)	0.7482 (8)	−0.0803 (6)	0.6230 (5)	7.2 (3)
C(6)	0.8374 (8)	−0.1685 (5)	0.6319 (5)	7.0 (3)
C(7)	0.9194 (7)	−0.2026 (4)	0.5495 (5)	6.2 (2)
C(8)	0.9150 (6)	−0.1478 (4)	0.4586 (4)	4.6 (2)
C(9)	0.8317 (6)	−0.0572 (4)	0.4464 (4)	4.5 (2)
C(10)	0.7956 (6)	0.0547 (4)	0.2804 (4)	4.2 (2)
C(11)	0.8291 (7)	0.0637 (4)	0.1665 (4)	4.8 (2)
C(12)	0.7697 (8)	0.0033 (6)	−0.0048 (5)	8.0 (3)
C(13)	0.6702 (9)	−0.0786 (6)	−0.0561 (5)	9.5 (3)
C(14)	0.7046 (8)	0.1390 (4)	0.3210 (5)	6.2 (2)
C(15)	0.568 (1)	0.2870 (7)	0.2750 (8)	13.3 (5)
C(16)	0.515 (1)	0.3331 (8)	0.1908 (9)	15.1 (6)

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

N—C(2)	1.349 (7)	N—C(8)	1.404 (7)
O(1)—C(2)	1.218 (7)	O(2)—C(11)	1.199 (7)
O(3)—C(11)	1.331 (7)	O(3)—C(12)	1.451 (7)
O(4)—C(14)	1.200 (8)	O(5)—C(14)	1.292 (8)
O(5)—C(15)	1.46 (1)	C(2)—C(3)	1.510 (7)
C(3)—C(9)	1.458 (7)	C(3)—C(10)	1.344 (7)
C(4)—C(5)	1.384 (8)	C(4)—C(9)	1.394 (8)
C(5)—C(6)	1.41 (1)	C(6)—C(7)	1.366 (9)
C(7)—C(8)	1.362 (8)	C(8)—C(9)	1.409 (7)
C(10)—C(11)	1.489 (7)	C(10)—C(14)	1.476 (8)
C(12)—C(13)	1.52 (1)	C(15)—C(16)	1.30 (1)
C(2)—N—C(8)	111.1 (4)	C(11)—O(3)—C(12)	115.2 (4)
C(14)—O(5)—C(15)	117.4 (6)	N—C(2)—O(1)	126.1 (5)
N—C(2)—C(3)	107.1 (4)	O(1)—C(2)—C(3)	126.7 (5)
C(2)—C(3)—C(9)	105.4 (4)	C(2)—C(3)—C(10)	120.2 (4)
C(9)—C(3)—C(10)	134.4 (5)	C(5)—C(4)—C(9)	119.7 (5)
C(4)—C(5)—C(6)	119.9 (6)	C(5)—C(6)—C(7)	121.2 (6)
C(6)—C(7)—C(8)	118.2 (6)	N—C(8)—C(7)	127.5 (5)
N—C(8)—C(9)	109.5 (4)	C(7)—C(8)—C(9)	123.0 (5)
C(3)—C(9)—C(4)	135.1 (5)	C(3)—C(9)—C(8)	106.9 (4)
C(4)—C(9)—C(8)	117.9 (5)	C(3)—C(10)—C(11)	120.8 (4)
C(3)—C(10)—C(14)	125.1 (5)	C(11)—C(10)—C(14)	114.0 (4)
O(2)—C(11)—O(3)	124.6 (5)	O(2)—C(11)—C(10)	123.8 (5)
O(3)—C(11)—C(10)	111.5 (4)	O(3)—C(12)—C(13)	106.9 (5)
O(4)—C(14)—O(5)	121.2 (6)	O(4)—C(14)—C(10)	126.8 (6)
O(5)—C(14)—C(10)	111.7 (5)	O(5)—C(15)—C(16)	109.8 (9)

Data were corrected for Lorentz, polarization and absorption effects. The structure was solved by direct methods. Refinement was by full-matrix least squares. H atoms were included as fixed contributors; those of the 3*H*-indole-2(1*H*)-one moiety were found in difference synthesis and the remaining H atoms were placed in calculated positions. Two overall isotropic temperature factors were refined, one for each group of H atoms. Programs used were: *SHELXS*86 (Sheldrick, 1985), *SHELX*-76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71756 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1067]

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1,16-Hexadecanediol, $C_{16}H_{34}O_2$

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Abstract

The crystal structure of the title compound was determined by X-ray diffraction. The skeleton of the molecule is all *trans* and the molecules form a layer

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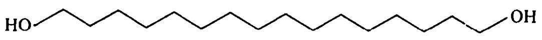
structure along the *b* axis as in a long-chain primary alcohol or a smectic C liquid crystal. The inclination angle of the long axis of the molecule to the layer plane is equal in each layer, but the direction of the long axis is opposite in alternate layers. This arrangement is different from that of the long-chain primary alcohols.

Comment

The crystal structures of long-chain primary alcohols with a similar number of C atoms to the title compound have been reported by several workers (Seto, 1962; Fujimoto, Yamamoto & Hara, 1985). The crystal structures of α,ω-alkanediols, except 1,6-hexanediol, however, have not yet been analyzed (Lindgren, Gustafsson, Westerling & Lund, 1986). The crystal structure analysis performed in this work is the first example in the long-chain homologous series. Fig. 1 shows the molecular structure of (I).



(I)

The molecular skeleton is constructed of a zigzag hydrocarbon chain, with the molecular structure resembling that of other long-chain molecules. The *ab* projection of the crystal structure is shown in Fig. 2. The molecule is centrosymmetric. The molecular arrangement along the *a* axis is zigzag, which may be attributed to the formation of hydrogen bonds at both sides of the molecules. The crystal structure obtained in this study has subtle differences from that of long-chain primary alcohols, while it resembles that of 1,12-dibromododecane (Kulpe, Seidel, Szulzewsky, Steger & Steger, 1981).

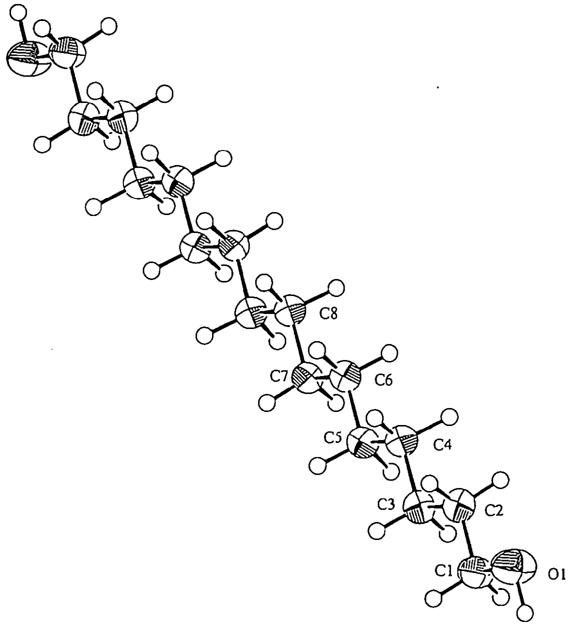


Fig. 1. Molecular structure of 1,16-hexadecanediol.

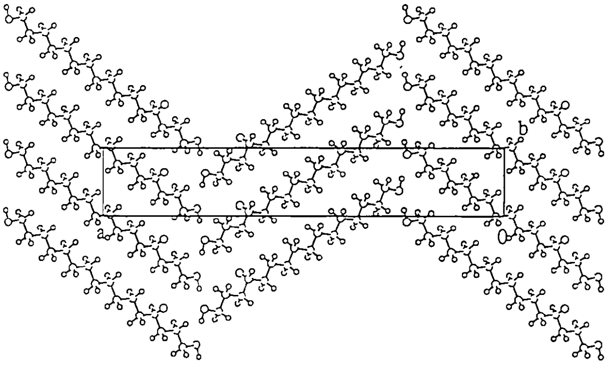


Fig. 2. The *ab* projection of the crystal structure.

Experimental

The commercially obtained sample was purified by recrystallization from ethanol.

Crystal data

$C_{16}H_{34}O_2$
M_r = 258.41
Monoclinic
$P2_1/n$
a = 31.396 (2) Å
b = 5.207 (1) Å
c = 4.980 (2) Å
β = 91.71 (2)°
V = 813.8 (3) Å^3
Z = 2
D_x = 1.055 Mg m^{-3}
D_m = 1.05 Mg m^{-3}
D_m measured by the density gradient-tube method

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 26.625 - 28.47°
μ = 0.48 mm^{-1}
T = 296 (1) K
Thin plates
0.5 × 0.2 × 0.1 mm
Colourless

Data collection

Rigaku *AFC-5R* diffractometer
ω-2θ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\min}$ = 0.89, $T_{\max}$ = 1.13
1543 measured reflections
1369 independent reflections
678 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int}$ = 0.031
$\theta_{\max}$ = 60.1°
$h = -32 \rightarrow 34$
$k = -5 \rightarrow 0$
$l = -5 \rightarrow 0$
3 standard reflections monitored every 150 reflections
intensity variation: none

Refinement

Refinement on F
R = 0.049
wR = 0.079
S = 1.74

$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\max}$ = 0.01
$\Delta\rho_{\max}$ = 0.13 e Å^{-3}
$\Delta\rho_{\min}$ = −0.14 e Å^{-3}

678 reflections
83 parameters
H-atom parameters not refined

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0.73840 (7)	−0.3847 (4)	−0.6651 (5)	6.02 (6)
C(1)	0.70348 (9)	−0.4255 (5)	−0.4967 (6)	4.45 (7)
C(2)	0.68723 (9)	−0.1722 (6)	−0.4012 (6)	4.10 (7)
C(3)	0.64888 (9)	−0.1980 (6)	−0.2249 (5)	3.97 (7)
C(4)	0.63170 (8)	0.0571 (5)	−0.1276 (6)	3.90 (7)
C(5)	0.59305 (9)	0.0307 (5)	0.0450 (6)	3.86 (7)
C(6)	0.57582 (9)	0.2849 (6)	0.1444 (5)	3.92 (7)
C(7)	0.53668 (9)	0.2586 (5)	0.3151 (5)	3.71 (7)
C(8)	0.51936 (8)	0.5142 (5)	0.4132 (6)	3.88 (6)

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

O(1)—C(1)	1.416 (3)	C(5)—C(6)	1.519 (4)
C(1)—C(2)	1.497 (4)	C(6)—C(7)	1.521 (4)
C(2)—C(3)	1.517 (4)	C(7)—C(8)	1.524 (4)
C(3)—C(4)	1.519 (4)	C(8)—C(8^i)	1.520 (5)
C(4)—C(5)	1.514 (4)		
O(1)—C(1)—C(2)	109.4 (2)	C(4)—C(5)—C(6)	113.9 (2)
C(1)—C(2)—C(3)	112.9 (2)	C(5)—C(6)—C(7)	113.9 (2)
C(2)—C(3)—C(4)	113.8 (3)	C(6)—C(7)—C(8)	113.8 (2)
C(3)—C(4)—C(5)	113.6 (2)	C(7)—C(8)—C(8^i)	113.4 (3)

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

The structure was determined by direct methods using *SHELXS*-86 (Sheldrick, 1985) and successive Fourier syntheses (Beurskens *et al.*, 1992). Non-H atoms were refined anisotropically and H atoms were fixed in idealized positions. All calculations were performed using the *TEXSAN* software package (Molecular Structure Corporation, 1992).

The authors wish to express their gratitude to Professor Y. Yoshimura of Ritsumeikan University for useful discussions and suggestions.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71786 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1052]

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5-Nitro-2-furaldehyde Semicarbazone

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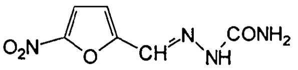
Abstract

The molecules of the title compound, $C_6H_6N_4O_4$, are linked through bifurcated hydrogen bonds. A three-dimensional network of molecules is observed.

Comment

A perspective view showing the atomic numbering scheme and hydrogen bonding is given in Fig. 1. The crystal contains well ordered molecules of 5-nitro-2-furaldehyde semicarbazone, (I), in the form also observed in crystals of 5-nitrofuran-2-aldoxime (Matsuoka, Furukawa, Takao, Kitao, Hamada & Nakatsu, 1991). The bond lengths are similar to within 0.03 Å. The title compound has a *trans* conformation, while 5-nitrofuran-2-aldoxime (Matsuoka *et al.*, 1991) is *cis* with respect to the double bond of the side chain (C21=N22).



(I)

The C3=C2—C21=N22 planar group of atoms does not display any conjugation effect between double bonds. A comparison of the bond lengths with those given by Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987) and Burke-Laing & Laing (1976) shows bond orders of two for C3=C2 and C21=N22 and one for C2—C21.

The furan ring is planar to within experimental error. The plane of the nitro substituent crosses the five-membered ring plane at an angle of 177.2 (3)°. The side chain is also planar and its least-squares plane makes a dihedral angle of 7.8 (1)° with the furan ring. The whole