Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: NA1259). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,13-Tridecanediol

Naotake Nakamura, Yasuhiro Tanihara and Taiki Takayama

Department of Chemistry, Faculty of Science and Engineering, Ritsumeikan University, 1916 Noji, Kusatsu, Shiga 525-77, Japan. E-mail: nakamura@bkc.ritsumei.ac.jp

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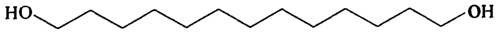
Abstract

The crystal structure of tridecane-1,13-diol, $C_{13}H_{28}O_2$, a model for liquid crystals, has been determined by X-ray diffraction. The molecules form a layer structure along the *a* axis as in a smectic A liquid crystal. The skeleton of the molecule has an all-*trans* conformation. One of the terminal hydroxyl groups is in a *gauche* conformation, while the other is *trans*.

Comment

Long-chain compounds such as *n*-higher primary alcohols and α,ω-disubstituted *n*-alkanes exhibit a high-temperature phase just below their melting points. It has been proposed that the molecules rotate about their long axes in this phase (Asai, Yoda & Yamanaka, 1955), a fact which shows the molecules to have a considerable degree of motional freedom in the phase. The crystal structures of some long-chain compounds similar to the title compound, (I), have been determined previously (for example, Watanabe, 1961; Seto, 1962; Nakamura & Yamamoto, 1994). Almost all the structures reported are similar to that of a smectic liquid crystal, in which long molecules arrange themselves in parallel to form a layer. These long-chain compounds have been of great interest recently as liquid-crystal model materials.



(I)

The molecular structure of 1,13-tridecanediol is shown in Fig. 1. One of the hydroxyl groups of the molecule is in a *gauche* conformation, while the other is *trans*. The molecular skeleton of the hydrocarbon chain is all-*trans*. The *gauche* conformation has been observed in 1-heptadecanol (Seto, 1962), where the *gauche* and *trans* conformations of the hydroxyl groups appear alternately in laterally adjoining molecules in the layers, *i.e.* the hydroxyl group of a 1-heptadecanol molecule is in a *gauche* conformation if that of the longitudinally adjoining molecule in the adjoining layer is *trans*. As a result, interlayer hydrogen-bond formation is in a *trans*/*gauche*

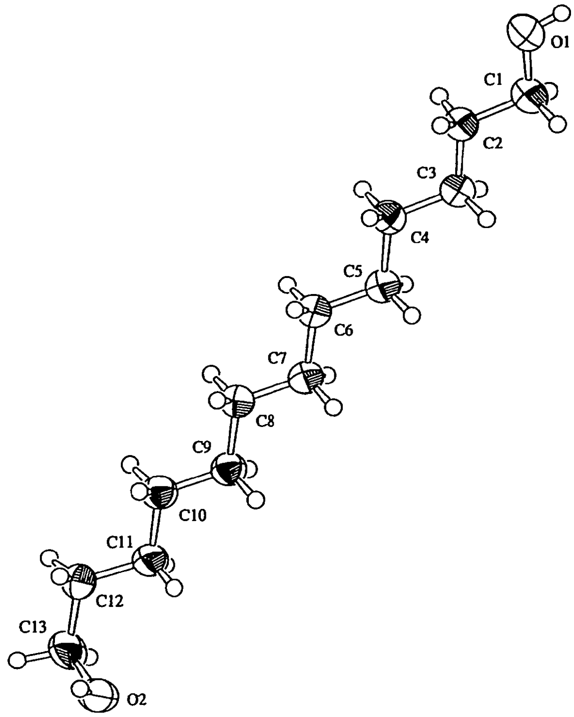


Fig. 1. An *ORTEP*II (Johnson, 1976) view of the title molecule showing the crystallographic numbering scheme.

sequence. The same interlayer hydrogen-bond formation was observed in the crystal of 1,13-tridecanediol as can be seen from the *ab* projection shown in Fig. 2, where the layers can be seen edge on. This packing is very similar to the smectic A structure of a liquid crystal. In contrast, the molecular packing of 1,16-hexadecanediol, which is a homologue with an even number of C atoms (Nakamura & Yamamoto, 1994), is smectic C-like and a herringbone pattern is observed (Gray & Goodby, 1984). The hydroxyl groups located at either end of the molecular skeleton are in *trans* conformations and interlayer hydrogen bonding is *trans* to *trans*.

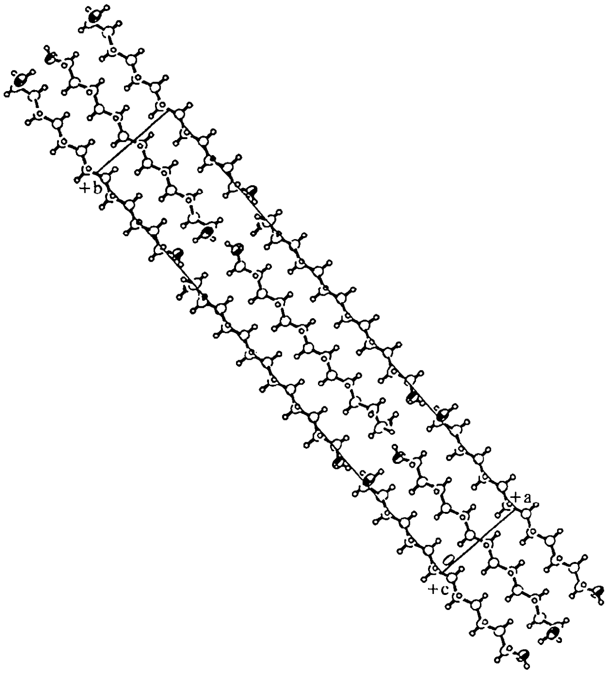


Fig. 2. The *ab* projection of the crystal structure.

Experimental

The crystal of the title compound was obtained from a mixture of heptane and ethyl acetate by slow evaporation.

Crystal data

$C_{13}H_{28}O_2$
$M_r = 216.36$
Orthorhombic
$P2_12_12_1$
$a = 7.163\,(2)$ Å
$b = 37.56\,(1)$ Å
$c = 5.113\,(3)$ Å
$V = 1375.6\,(9)$ Å^3
$Z = 4$
$D_x = 1.045$ Mg m^{-3}
$D_m = 1.05\,(1)$ Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 23.4$–$32.8°$
$\mu = 0.523$ mm^{-1}
$T = 296.2$ K
Plate-like
0.5 × 0.4 × 0.3 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.68$, $T_{max} = 0.85$
1273 measured reflections
1273 independent reflections

969 reflections with $I > 3\sigma(I)$
$\theta_{max} = 60.03°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 42$
$l = 0 \rightarrow 5$
3 standard reflections every 150 reflections intensity decay: 0.71%

Refinement

Refinement on F
$R = 0.0474$
$wR = 0.0758$
$S = 1.102$
969 reflections
136 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.00216|F_o|^2]$

$(\Delta/\sigma)_{max} = 0.0075$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.11$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C1	1.420 (4)	C6—C7	1.525 (4)
O2—C13	1.433 (4)	C7—C8	1.505 (4)
C1—C2	1.501 (4)	C8—C9	1.532 (4)
C2—C3	1.521 (4)	C9—C10	1.507 (4)
C3—C4	1.514 (4)	C10—C11	1.519 (4)
C4—C5	1.532 (4)	C11—C12	1.522 (4)
C5—C6	1.509 (4)	C12—C13	1.503 (4)
O1—C1—C2	108.2 (2)	C7—C8—C9	114.4 (2)
C1—C2—C3	114.0 (2)	C8—C9—C10	113.3 (2)
C2—C3—C4	112.5 (2)	C9—C10—C11	114.3 (2)
C3—C4—C5	114.6 (2)	C10—C11—C12	112.5 (2)
C4—C5—C6	113.1 (2)	C11—C12—C13	114.4 (3)
C5—C6—C7	114.6 (2)	O2—C13—C12	112.4 (2)
C6—C7—C8	113.9 (2)		

Table 2. *Contact distances (Å)*

O1···O2^{i}	2.713 (2)	O2···C1^{iii}	3.413 (3)
O1···O2^{ii}	2.776 (4)	O2···C12^{iv}	3.448 (4)
O1···C13^{i}	3.509 (4)		

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

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, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: TA1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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L-Histidylglycinium Dichloride

THOMAS STEINER

Institut für Kristallographie, Freie Universität Berlin, Takustraße 6, D-14195 Berlin, Germany. E-mail: steiner@chemie.fu-berlin.de

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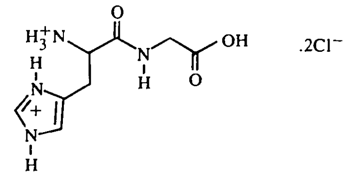
Abstract

In the title compound, $C_8H_{14}N_4O_3^{2+}.2Cl^-$, the dipeptide His–Gly crystallizes as a dication. The positively charged imidazole moiety of the histidine side chain is tightly coordinated by chloride anions and O-atom hydrogen-bond acceptors in an almost planar configuration.

Comment

The crystal structure of the dipeptide L-$^+H_2$His–Gly.2Cl$^-$, (I), was determined in order to characterize the hydrogen-bond interactions and is the fourth in a series of small peptide determinations (previous contributions: Steiner 1996*a*,*b*,*c*). Since dipeptide (I) was crystallized from 6% HCl, it was obtained as a dication, with two chloride anions balancing the charge. No water molecules are co-crystallized.



(I)

The overall conformation of (I) (Fig. 1) is conventional and need not be discussed here in detail. The same is true for most of the N—H···*A* and O—H···*A* hydrogen bonds formed by the peptide main-chain functional groups (Table 2). The O—H···Cl$^-$ hydrogen bond donated by the C-terminal C(=O)—OH group is one of the shortest of its kind [O3···Cl1$^-$ 2.932 (3) Å].

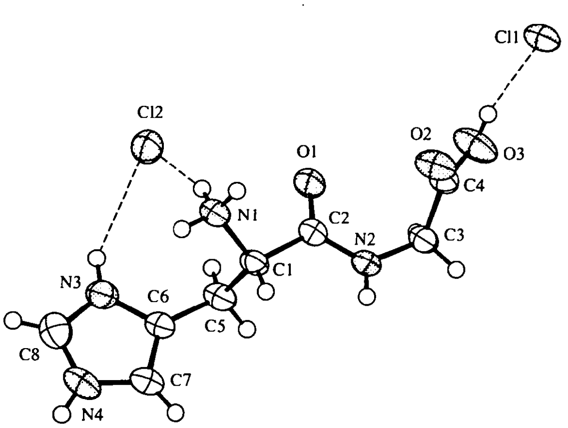


Fig. 1. The molecular structure and atom labeling of the title compound. Displacement ellipsoids are drawn at the 50% probability level. O and N atoms and Cl$^-$ anions are shaded.

The side chain of the histidine residue is oriented with the Cβ—Cγ bond (C5—C6) *trans* with respect to the Cα—C(=O) bond [torsion angle C2—C1—C5—C6 −176.2 (3)°] and the imidazole plane perpendicular to Cα—Cβ—Cγ [torsion angle C1—C5—C6—C7 −87.7 (5)°]; this is one of the common histidine conformations (Steiner, 1996*b*).

The positively charged imidazole moiety of the histidine side chain is tightly coordinated by chloride ions and O-atom hydrogen-bond acceptors in an almost planar configuration (Fig. 2). The mutual approach of the Cl1 and Cl2($x+1$, $y-1$, z) anions is quite short [3.814 (1) Å]; this is facilitated by two three-center N—H···Cl$^-$ hydrogen bonds [donated by N3—H and N1—H3N1 (Table 2)]. It is notable that O1 and Cl1 each accept an N—H···*A* and a C—H···*A* interaction of similar geometries. In particular for the O1 atom, the