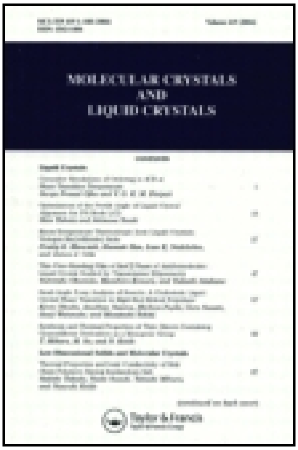

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The Crystal Structure of Bis[4-(5′-carboxylpentyloxy)acetophenon]azine

R. Centore [a] , M. R. Ciajolo [a] , A. Roviello [a] , A. Sirigu [a] & A. Tuzi [a]

[a] Dipartimento di Chimica, Università di Napoli, Via Mezzocannone 4, 80134, Napoli, Italy

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The Crystal Structure of Bis[4-(5′-carboxylpentyloxy)acetophenon]azine

R. CENTORE, M. R. CIAJOLO, A. ROVIELLO, A. SIRIGU and A. TUZI

Dipartimento di Chimica, Università di Napoli, Via Mezzocannone 4, 80134, Napoli, Italy

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Bis[4-(5′-carboxylpentyloxy)acetophenon]azine, $C_{28}H_{36}N_2O_6$, is a dicarboxylic acid containing a mesogenic core. It has been taken into account as a model compound for main chain liquid crystalline polymers containing mesogenic groups and flexible spacers in alternating sequence. The crystal and molecular structure has been determined, at room temperature, by direct methods. Crystals belong to the triclinic system, space group $P\bar{1}$, $a = 6.519(2)$Å, $b = 7.665(2)$Å, $c = 26.215(7)$Å, $\alpha = 91.97(2)°$, $\beta = 93.70(2)°$, $\gamma = 84.77(2)°$, with two molecules per unit cell. The structure was refined, by full matrix least squares method, to $R = 0.064$ for 2679 observed independent reflections, having $I > 3\sigma(I)$. Molecules are bonded along extended rows through hydrogen bonds between carboxylic groups. The conformation around the central =N—N= bond of the crystallographic independent molecule is not fully transplanar and the phenyl rings belonging to a molecule are twisted with respect to each other, their average planes making a dihedral angle of 63.8(1)°.

Keywords: crystal structure, molecular structure, model for mesogenic compounds, model for crystal packing of mesogenic polymers.

INTRODUCTION

Despite the relevant interest inherent to the solid state structure of linear semiflexible mesogenic polymers, whose detailed investigation could give great insight into the rather central point of the relationship between crystal and mesophase structure, no complete structural analysis has been reported up to now.

The reason for this may be connected with the usually large dimensions of the monomeric units present in these polymers, and with the frequent occurrence of complex solid state polymorphism.

For the purpose of obtaining structural data useful for a more simplified approach to the analysis of the crystal structure of such polymers, some attention has been devoted to low molar mass compounds which stoichiometrically correspond, or are very close, to the monomeric units present in the polymers.[1,2]

However, a strict chemical homology of model compounds with polymers (same rigid group and same linkage between rigid and flexible sections) may not be sufficient, in itself, to ensure also structural homology, especially if the packing is different.

For this reason it seems therefore more convenient to turn the attention toward model compounds in which a packing of the molecules along extended rows, simulating long polymeric chains, will presumably occur.

For this purpose we have recently undertaken the investigation of α,ω-dicarboxylic acids containing a mesogenic core.[3] Our assumption was that these compounds, in the crystal phase, form extended polymer-like rows, with molecules held together by hydrogen bonds, in a way similar to what found by Corradini *et al.* for a series of aliphatic dicarboxylic acids taken as model compounds of stereoregular polyolefins.[4]

The dicarboxylic acid, whose crystal structure is discussed in the present paper, having formula $HOOC—(CH_2)_5—O—R—O—(CH_2)_5—COOH$, contains, as a central core, the rigid group $—R— = —\phi—C(CH_3)=N—N=C(CH_3)—\phi—$, ($—\phi—$ = p-phenylene), which is also present in several classes of mesogenic polymers,[5,6] as well as in low molar mass compounds[7,8]; the crystal structure of a model compound containing the —R— group has been already reported by us,[2] which will be referred to as compound F_3.

EXPERIMENTAL

The synthesis and the phase behaviour of the title compound have been already reported.[3] Single crystals (thin yellow plates), suitable for X ray diffraction studies, were obtained, after several unsuccessful trials, by slow evaporation at ambient temperature of a saturated solution of the compound in a pyridine/ethanol mixture.

Weissenberg and oscillation photographs suggested triclinic symmetry. Accurate cell parameters were obtained through a least squares fit of the setting angles of 25, accurately centred, strong reflections, in the range $20° < 2\theta < 60°$, on an Enraf Nonius CAD-4 automated single crystal diffractometer.

Data collection was performed in the range $2° < \theta < 60°$; max. $\sin\theta/\lambda = 0.5614$ Å^{-1}, $-7 \leq h \leq +7$, $-8 \leq k \leq 8$, $0 \leq l \leq 29$, ω scan mode as suggested by peak shape analysis, Ni filterd CuKα radiation. Two standard reflections periodically measured showed only random fluctuations. Of the total of 3863 independent reflections collected, only 2679 having $I > 3\sigma(I)$ were considered to be observed and used in structure analysis. Lorentz and polarization corrections were applied, but no correction for absorption and extinction.

A set of crystallographically relevant parameters is given in Table I.

DETERMINATION AND REFINEMENT OF THE STRUCTURE

The structure was solved by means of program MULTAN82[9] in the space group $P\bar{1}$, as suggested by the E-value statistics. The E map, evaluated with the set of phases corresponding to the highest combined figure of merit, showed all non hydrogen atoms. Refinement (on F), by full matrix least squares method, of coordinates and individual isotropic thermal parameters converged to R = 0.169, and, after introduction of individual anisotropic thermal parameters, to R = 0.103.

TABLE I

Crystal Data
Bis[4-(5′-carboxypentyloxy)acetophenon]azine

$C_{28}H_{36}N_2O_6$
Formula Weight = 496.61
Triclinic
Space Group $P\bar{1}$
a = 6.519(2)Å
b = 7.665(2)Å
c = 26.215(7)Å
α = 91.97(2)°
β = 93.70(2)°
γ = 84.77(2)°

V = 1301(3)$Å^3$
Z = 2
D_x = 1.267g/cm^3
λ(CuKα) = 1.54178Å
μ(CuKα) = 6.9cm^{-1}
F(000) = 532

A difference Fourier map evaluated at this stage showed almost all hydrogen atoms in chemically reasonable positions. However, only the positions of the H atoms of methyl and carboxyl groups were defined on the basis of the Fourier map, the remainder were placed in theoretical positions. For all H atoms isotropic thermal parameters equal to those of the carrier atoms were used; they were included in structure factor calculations but not refined. Further refinement of coordinates and anisotropic thermal parameters of non H atoms gave R = 0.064 (wR = 0.064), S = 0.999; largest parameter shift to error ratio in the last refinement cycle was less than 0.03. The difference Fourier map evaluated in the final stage of the refinement showed no maxima higher than 0.2e$Å^{-3}$. Unit weights used throughout the refinement. Atomic scattering factors were taken from International Tables for X Ray Crystallography,[10] programs from Enraf Nonius Structure Determination Package.[11] All calculations were performed on a Digital MicroVMS V4.7 computer. Additional material to this paper (list of observed and calculated structure factors, coordinates and thermal parameters of hydrogen atoms) is available from the authors on request.

RESULTS AND DISCUSSION

Final refined fractional coordinates and anisotropic thermal parameters of the crystallographic independent molecule are given in Table II and III respectively; selected bond lengths, bond angles and torsion angles are reported in Table IV. Atomic numbering and molecular geometry are shown in Figure 1.

Some bond lengths in the aliphatic chain C1 . . . C6, namely C2—C3 and C4—C5, appear smaller than standard values, whereas bond angles appear larger than normal aliphatic values; bond lengths and angles of the other aliphatic chain, C23 . . . C28, all take standard values. A possible account of this feature may be given, following the Busing and Levy's model,[12] if we consider that the thermal motion of the atoms in the chains is highly anisotropic. In particular B_{22} is systematically higher than other components, indicating a large amplitude of vibration along b. The atoms of pairs C2,C3 and C4,C5, in particular, are almost equally stacked along b: therefore the larger anisotropic thermal vibration takes place, for

TABLE II

Refined positional parameters and equivalent temperature factors with e.s.d.'s in parentheses. $B_{eq} = (4/3) \cdot [a^2B_{11} + b^2B_{22} + c^2B_{33} + ab\cos\gamma B_{12} + ac\cos\beta B_{13} + bc\cos\alpha B_{23}]$

atom	x	y	z	B_{eq}(Å^2)
O1	−0.4916(7)	−0.1807(7)	0.9534(2)	10.0 (1)
O2	−0.8206(6)	−0.1073(7)	0.9303(2)	9.4 (1)
O3	0.1003(5)	0.0482(4)	0.7289(1)	4.81(7)
O4	0.4734(5)	0.5131(4)	0.2334(1)	4.80(7)
O5	1.0917(6)	0.7293(6)	0.0120(2)	8.9 (1)
O6	1.4194(6)	0.6657(8)	0.0381(2)	11.2 (1)
N1	0.2353(5)	0.1916(5)	0.4972(1)	4.04(8)
N2	0.3103(5)	0.2317(5)	0.4503(1)	4.08(8)
C1	−0.6264(8)	−0.1209(8)	0.9230(2)	5.9 (1)
C2	−0.5780(9)	−0.0546(9)	0.8730(2)	7.8 (2)
C3	−0.3635(9)	−0.0647(8)	0.8600(2)	6.7 (1)
C4	−0.3347(8)	−0.0309(8)	0.8045(2)	6.1 (1)
C5	−0.1236(8)	−0.0200(9)	0.7911(2)	6.8 (1)
C6	−0.0959(7)	−0.0149(7)	0.7342(2)	4.8 (1)
C7	0.1546(7)	0.0790(6)	0.6803(2)	3.83(9)
C8	0.0445(7)	0.0336(6)	0.6359(2)	3.9 (1)
C9	0.1144(7)	0.0711(6)	0.5890(2)	3.8 (1)
C10	0.2950(6)	0.1546(5)	0.5859(2)	3.26(9)
C11	0.4019(7)	0.2003(6)	0.6313(2)	3.9 (1)
C12	0.3331(7)	0.1636(6)	0.6785(2)	4.1 (1)
C13	0.3658(6)	0.1996(5)	0.5354(2)	3.46(9)
C14	0.5781(7)	0.2572(7)	0.5328(2)	5.1 (1)
C15	0.1835(6)	0.3254(5)	0.4209(2)	3.52(9)
C16	−0.0286(7)	0.3980(7)	0.4345(2)	4.8 (1)
C17	0.2595(6)	0.3683(5)	0.3709(2)	3.47(9)
C18	0.1454(7)	0.4874(6)	0.3386(2)	3.9 (1)
C19	0.2200(7)	0.5314(6)	0.2928(2)	4.1 (1)
C20	0.4121(7)	0.4573(6)	0.2790(2)	3.9 (1)
C21	0.5267(7)	0.3375(6)	0.3102(2)	4.1 (1)
C22	0.4514(7)	0.2935(6)	0.3558(2)	4.0 (1)
C23	0.6824(7)	0.4625(7)	0.2208(2)	5.1 (1)
C24	0.7152(8)	0.5591(8)	0.1729(2)	6.0 (1)
C25	0.9261(8)	0.5086(8)	0.1528(2)	5.9 (1)
C26	0.9582(8)	0.6079(8)	0.1045(2)	5.9 (1)
C27	1.1731(9)	0.5572(9)	0.0863(2)	7.4 (2)
C28	1.2250(9)	0.6623(9)	0.0422(2)	6.8 (2)

these atoms, in a direction which is quite perpendicular to that of the bond, so determining a striking effect of apparent contraction of the bond length.

The conformations taken by the terminal aliphatic chains of the molecule are not equivalent to each other and show remarkable deviations from the trans-planar, most extended, conformation around several CH_2—CH_2 bonds, mainly in the chain C1 . . . C6.

As far as the structure of the central —R— group is concerned, bond lengths and bond angles are equal, within experimental error, to those observed for the same group in compound F_3.[2]

On the other hand, a significant difference with respect to the structure of the —R— group, as previously reported in F_3, has been found, regarding the confor-

TABLE III

Refined anisotropic temperature factors with e.s.d.'s in parentheses. The temperature factor is of the form: $\exp[-0.25(h^2a^{*2}B_{11} + k^2b^{*2}B_{22} + l^2c^{*2}B_{33} + 2hka^*b^*B_{12} + 2hla^*c^*B_{13} + 2klb^*c^*B_{23})]$, where a^*, b^*, c^* are reciprocal lattice constants.

atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O1	6.6(2)	16.8(3)	6.8(2)	0.3(2)	2.2(2)	5.5(2)
O2	6.4(2)	14.3(3)	8.0(2)	0.1(2)	2.3(2)	4.9(2)
O3	4.7(1)	6.6(2)	3.4(1)	−1.2(1)	0.8(1)	1.1(1)
O4	4.5(1)	6.2(2)	4.0(1)	−0.1(1)	1.4(1)	1.6(1)
O5	6.5(2)	13.5(3)	7.2(2)	−0.3(2)	2.2(2)	4.6(2)
O6	5.8(2)	21.0(4)	7.3(2)	−2.1(2)	1.5(2)	6.0(2)
N1	4.0(2)	4.8(2)	3.5(1)	−0.4(1)	0.9(1)	1.1(1)
N2	4.0(2)	5.0(2)	3.4(1)	−0.5(1)	1.1(1)	0.9(1)
C1	5.7(3)	7.5(3)	4.7(2)	−0.2(2)	1.5(2)	1.4(2)
C2	6.6(3)	11.7(4)	5.2(3)	−0.5(3)	1.7(2)	2.9(3)
C3	6.1(3)	9.9(4)	4.5(2)	−0.4(3)	1.9(2)	2.0(2)
C4	5.6(3)	8.6(3)	4.4(2)	−0.5(2)	1.5(2)	1.4(2)
C5	4.9(2)	11.4(4)	4.4(2)	−1.2(3)	1.0(2)	2.4(2)
C6	4.5(2)	6.0(2)	4.0(2)	−0.6(2)	1.3(2)	1.1(2)
C7	4.0(2)	4.1(2)	3.5(2)	−0.2(2)	0.8(2)	1.0(2)
C8	3.8(2)	4.4(2)	3.6(2)	−0.9(2)	0.6(2)	0.7(2)
C9	3.9(2)	4.0(2)	3.5(2)	−0.6(2)	0.6(2)	0.4(2)
C10	3.4(2)	3.2(2)	3.3(2)	−0.2(1)	0.6(1)	0.7(1)
C11	3.7(2)	4.3(2)	3.9(2)	−0.8(2)	0.3(2)	0.7(2)
C12	4.4(2)	4.5(2)	3.5(2)	−0.7(2)	0.2(2)	0.4(2)
C13	3.4(2)	3.2(2)	3.9(2)	−0.1(1)	0.8(1)	0.6(2)
C14	3.9(2)	7.3(3)	4.4(2)	−1.6(2)	0.8(2)	0.8(2)
C15	3.8(2)	3.6(2)	3.3(2)	−0.8(2)	0.5(1)	0.1(2)
C16	3.9(2)	5.9(2)	4.6(2)	0.6(2)	1.5(2)	1.3(2)
C17	3.7(2)	3.7(2)	3.2(2)	−0.7(2)	0.7(1)	0.3(1)
C18	3.7(2)	4.2(2)	3.8(2)	−0.3(2)	0.6(2)	0.4(2)
C19	4.0(2)	4.4(2)	3.8(2)	−0.3(2)	0.6(2)	1.0(2)
C20	4.0(2)	4.6(2)	3.2(2)	−0.9(2)	0.7(2)	0.6(2)
C21	3.8(2)	4.6(2)	4.0(2)	0.0(2)	0.9(2)	0.8(2)
C22	3.9(2)	4.1(2)	4.0(2)	−0.0(2)	0.7(2)	0.8(2)
C23	4.5(2)	6.3(3)	4.5(2)	−0.2(2)	1.6(2)	1.3(2)
C24	5.4(2)	7.9(3)	4.8(2)	0.2(2)	2.0(2)	2.3(2)
C25	5.2(2)	8.1(3)	4.6(2)	−0.7(2)	1.6(2)	1.9(2)
C26	5.6(2)	8.0(3)	4.4(2)	−0.6(2)	1.8(2)	1.6(2)
C27	5.6(3)	11.4(4)	5.7(2)	−0.3(3)	2.4(2)	3.3(2)
C28	5.5(3)	10.3(4)	4.9(2)	−0.6(3)	1.9(2)	1.5(3)

mation around the central =N—N= bond, which, in the present case, is well different from the trans-planar one: C13—N1—N2—C15 = −139.3°(4). This result is quite unexpected, on account of the partial double-bond character of the =N—N= bond; by the way we remind that a fixed trans-planar conformation around this bond was also assumed in conformational energy calculations which have been recently performed[13,14] concerning the crystal phase of a series of mesogenic polyesters containing the —R— group.

Reasonable explanations of this structural feature should take into account the different mode of packing of the molecules (see below), as compared to F_3: although

TABLE IV

Selected bond lengths (Å), bond angles(°) and torsion angles (°), with e.s.d.'s in parentheses

O1—C1	1.216(7)	O5—C28	1.226(7)
O2—C1	1.286(7)	O6—C28	1.281(7)
C1—C2	1.486(8)	C27—C28	1.505(9)
C2—C3	1.455(8)	C26—C27	1.521(8)
C3—C4	1.513(7)	C25—C26	1.536(8)
C4—C5	1.452(8)	C24—C25	1.517(7)
C5—C6	1.515(7)	C23—C24	1.518(7)
C6—O3	1.424(6)	C23—O4	1.438(6)
O3—C7	1.377(5)	O4—C20	1.377(5)
C10—C13	1.491(6)	C15—C17	1.487(6)
C13—C14	1.497(6)	C15—C16	1.503(6)
C13—N1	1.276(5)	C15—N2	1.282(5)
N1—N2	1.405(5)		
O1—C1—O2	124.8(5)	O5—C28—O6	124.2(6)
O1—C1—C2	121.7(5)	O5—C28—C27	122.2(5)
O2—C1—C2	113.5(5)	O6—C28—C27	113.5(5)
C1—C2—C3	118.7(5)	C26—C27—C28	113.5(5)
C2—C3—C4	114.2(4)	C25—C26—C27	110.2(4)
C3—C4—C5	116.0(4)	C24—C25—C26	111.8(4)
C4—C5—C6	114.7(4)	C23—C24—C25	112.2(4)
C5—C6—O3	106.7(4)	C24—C23—O4	106.2(4)
C6—O3—C7	118.0(3)	C20—O4—C23	117.5(3)
O3—C7—C8	124.7(4)	C21—C20—O4	124.8(4)
O3—C7—C12	114.7(4)	C19—C20—O4	114.8(4)
C9—C10—C13	120.8(4)	C15—C17—C22	120.5(4)
C11—C10—C13	121.1(4)	C15—C17—C18	121.2(4)
N1—C13—C10	116.5(4)	N2—C15—C17	116.2(4)
N1—C13—C14	124.3(4)	N2—C15—C16	124.5(4)
C10—C13—C14	119.2(4)	C16—C15—C17	119.3(4)
N2—N1—C13	114.5(4)	N1—N2—C15	115.4(3)
O1—C1—C2—C3	1.0(9)	C26—C27—C28—O5	−26.4(8)
O2—C1—C2—C3	−179.3(5)	C26—C27—C28—O6	157.3(5)
C1—C2—C3—C4	167.5(5)	C25—C26—C27—C28	−174.1(5)
C2—C3—C4—C5	173.3(6)	C24—C25—C26—C27	178.6(4)
C3—C4—C5—C6	170.8(5)	C23—C24—C25—C26	−179.7(4)
C4—C5—C6—O3	162.7(5)	O4—C23—C24—C25	−176.0(4)
C7—O3—C6—C5	−174.6(4)	C20—O4—C23—C24	−173.1(4)
C6—O3—C7—C8	−8.2(6)	C23—O4—C20—C21	−9.0(6)
C6—O3—C7—C12	171.1(4)	C23—O4—C20—C19	170.7(4)
C9—C10—C13—N1	13.8(6)	C16—C15—C17—C22	−176.0(4)
C9—C10—C13—C14	−168.5(4)	C16—C15—C17—C18	5.9(6)
C11—C10—C13—N1	−163.6(4)	N2—C15—C17—C22	6.3(6)
C11—C10—C13—C14	14.0(6)	N2—C15—C17—C18	−171.7(4)
N2—N1—C13—C10	−178.8(3)	N1—N2—C15—C17	−179.6(3)
N2—N1—C13—C14	3.7(6)	N1—N2—C15—C16	2.8(6)
C13—N1—N2—C15	−139.3(4)		

in the non-trans-planar conformation the energy of the isolated molecule is higher (owing to the torsional barrier around =N—N= bond), the requirement for optimal intermolecular packing can stabilize the structure.

Mainly because of the torsion around =N—N= bond the planes of the phenyl rings are highly twisted with respect to each other, making a dihedral angle of

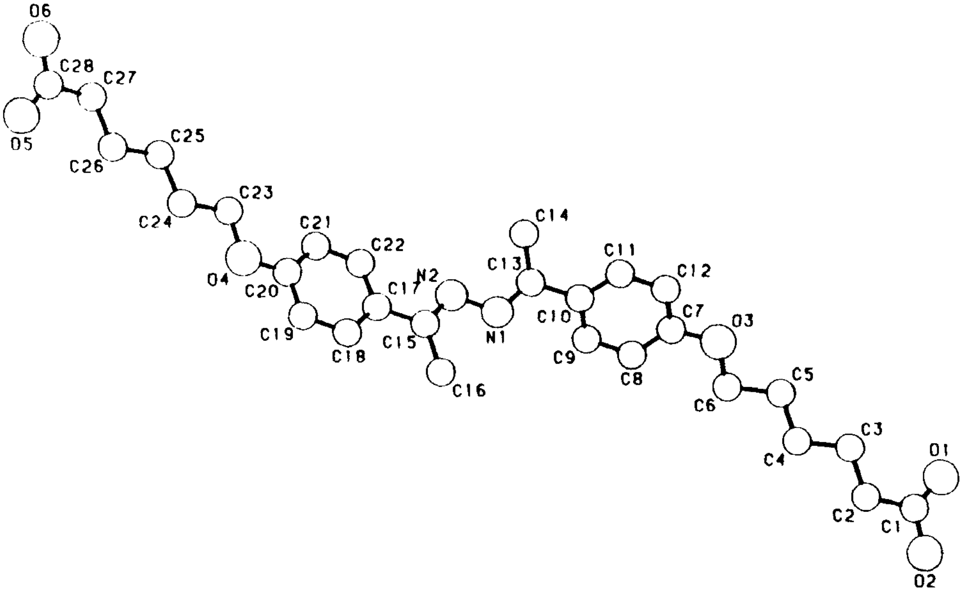

FIGURE 1 Stereoview of the crystallographically independent molecule showing atomic numbering.

63.8°(1). It is worth noticing, however, that the overall length of the —R— group, taken as the distance between O3 and O4 atoms, is almost unchanged by the torsion around N—N bond (O3···O4 = 14.005(4)Å as compared to a distance ≈14.05Å in the trans-planar conformation).

Molecular packing is shown in Figures 2 and 3. Molecules are bonded in extended rows through hydrogen bonds between carboxylic groups; intermolecular distances between oxygen atoms related by hydrogen bond are in good agreement with values reported in literature[15]; the cyclic hydrogen-bonded dimer (C1, O1, O2, $C28^{(i)}$, $O5^{(i)}$, $O6^{(i)}$) is planar within 0.09Å.

The rows are characterized by the lowest possible symmetry, that is *t*1 (molecules along each row are repeated only by translation); it should be noted that also the line repetition group *ti*, which is found in several dicarboxylic acids forming rows,[4] might have been compatible with the space group symmetry of the structure.

The lateral packing of the rows, which are placed parallel to each other, shows side by side correspondence of chemically homologous sections (aromatic and aliphatic); this, particularly for the optimization of the interactions among aromatic moieties of adjacent chains, might furnish the driving force for the torsion around =N—N= bond.

The whole crystal structure may be described as an assembly of molecular layers, held together by hydrogen bonds, and piled up along c; within each layer, the long molecular axes are tilted with respect to the normal to the layers, simulating a smectic C type arrangement. The title compound, however, does not exhibit mesogenic properties at all, probably because of the relatively high melting temperature (482 K).

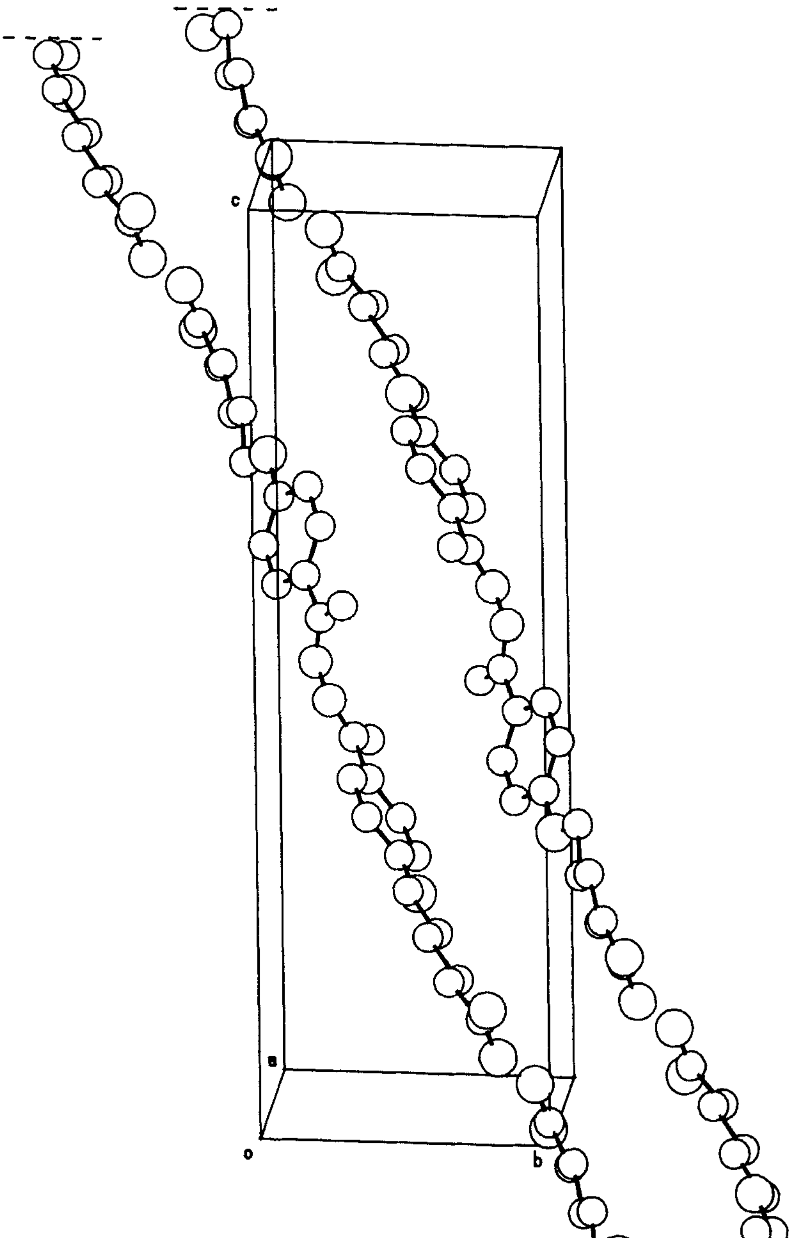

FIGURE 2 Crystal packing viewed down *a* and slightly rotated around *c*.

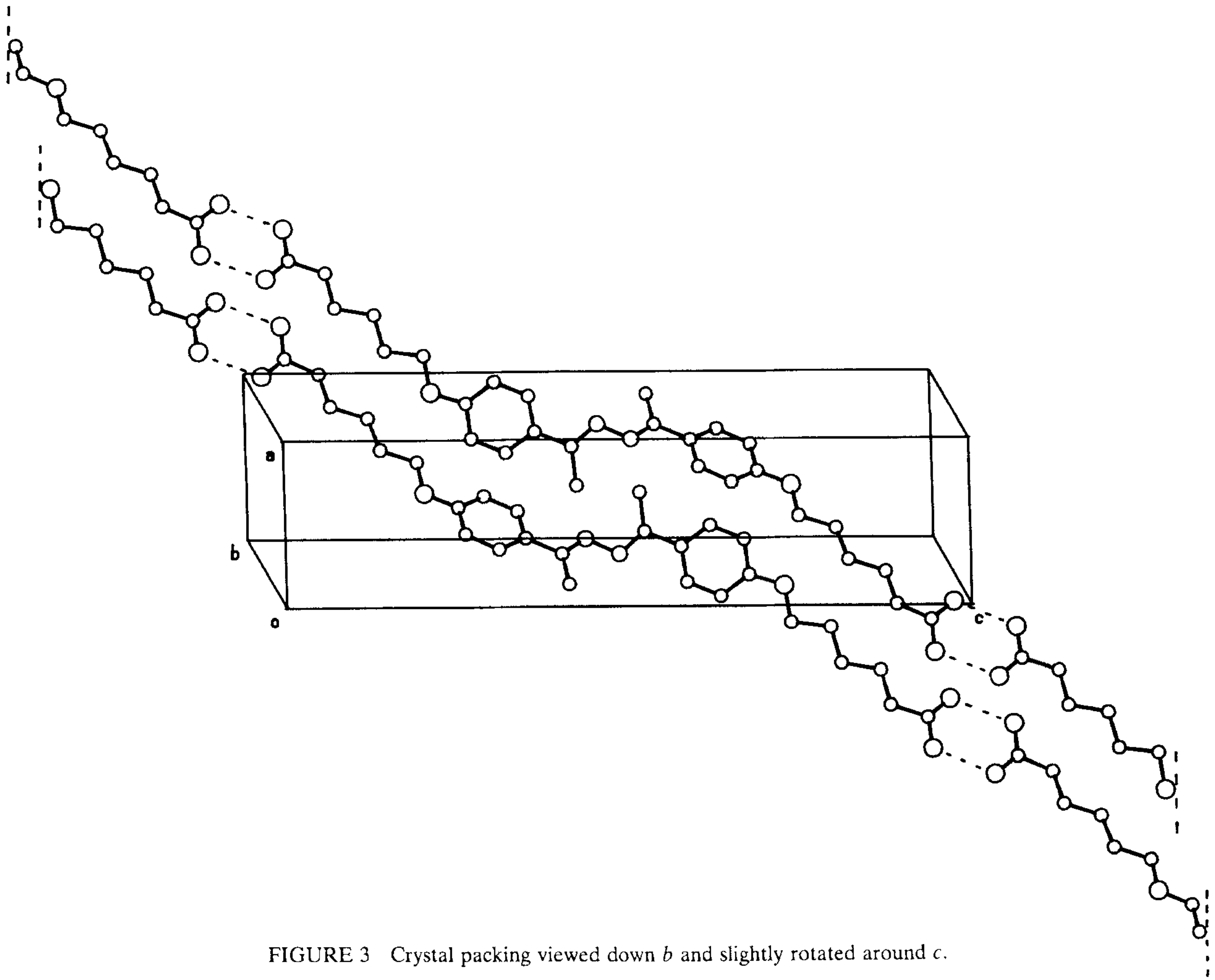

FIGURE 3 Crystal packing viewed down *b* and slightly rotated around *c*.

TABLE V

Hydrogen Bond Distances

O1 . . . O6$^{(i)}$	2.671(7)Å
O2 . . . O6$^{(i)}$	2.638(6)Å

Symmetry code
(i) x − 2, y − 1, z + 1

The possibility, outlined in this paper, that, due to intermolecular packing effects, relevant deviations from the trans-planarity may occur around the =N—N= bond and (by consequence) the —R— group may take conformations which are different from the lowest energy conformation of the isolated molecule, has to be taken into account in the structural investigations of semiflexible mesogenic polymers containing the —R— group.

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