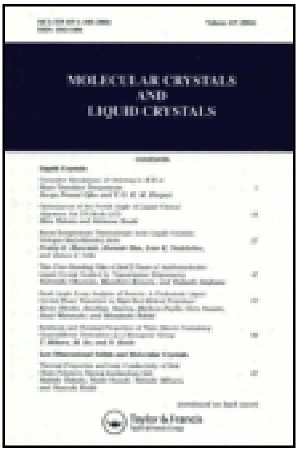

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# X-ray Structural Analysis of a Mesogenic Compound N,N′-Bis-(p-Butoxybenzylidene)-α,α′-bi-p-Toluidine

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# X-ray Structural Analysis of a Mesogenic Compound N,N′-Bis-(p-Butoxybenzylidene)-α,α′-bi-p-Toluidine

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Crystal and molecular structure of a mesogenic compound N,N′-Bis-p-(Butoxybenzylidene)-α,α′-bi-p-Toluidine has been determined by direct methods. The compound crystallizes in triclinic system with space group $P\bar{1}$. Cell parameters are $a = 6.116(3)$, $b = 7.916(3)$, $c = 31.421$ (8)Å, $\alpha = 92.39(2)$, $\beta = 92.35(3)$, $\gamma = 96.59(2)°$ and $z = 2$. The two symmetrical fractions of the molecule are almost planar but the angle between these planes is 63.5°. The molecules are arranged in tilted layers which is precursor to the tilted smectic phase. Crystal-mesophase transition is discussed.

*Keywords: crystal structure of a smectogen, X-ray study of a mesogen, structure-property relationship of liquid crystal.*

## INTRODUCTION

The molecular arrangement in the crystalline state is one of the factors which predetermines the occurrence of thermal mesomorphism in organic compounds. As a part of structural investigations of a series of mesogenic compounds[1] we have determined the crystal structure of N,N′-Bis-(p-butoxybenzylidene)-α,α′-bi-p-toluidine (BBBT) which exhibit both smectic and nematic phases over a wide range of temperature.

Under a crossed polarizing microscope (150*X*) an unspecified variety of mosaic texture in smectic region and marbled texture in nematic region were observed. Nematic droplets were observed at N-I transition both during heating and cooling. Pretransition effect was observed both before Cr-Sm and N-I transitions and super cooling was noticed as indicated below. X-ray diffraction photographs were taken in mesomorphic range with and without magnetic field using a high temperature camera designed by us.[2] From the textures and X-ray photographs following phases and transition temperatures (in°C) are observed:

$$\mathrm{Cr} \underset{145}{\overset{159}{\rightleftharpoons}} \mathrm{Sm} \underset{172}{\overset{188}{\rightleftharpoons}} \mathrm{N} \overset{293}{\longleftrightarrow} \mathrm{I}$$

the smectic phase is tilted, have ordering within layers and most probably it is SmG phase.[3]

## DETERMINATION OF CRYSTAL STRUCTURE

Plate shaped single crystals were grown by slow evaporation from a solution in benzene. Intensity data were measured in a CAD-4 diffractometer using $CuK_\alpha$ radiation and a graphite monochromator. Accurate cell parameters were determined from least squares fit of 21 strong reflections. Space group was found to be $P\bar{1}$ by statistical test. A total of 5108 reflections were measured of which 2129 were treated as observed with $I > 2.5\ Sig(I)$. $L_p$ correction was applied to the intensity data but no absorption correction was made. Important crystallographic data are given in Table I.

The structure was solved by the symbolic phase extension program SIMPEL.[4] 600 strong reflections were phased using four symbols, however the phase set with lowest CFOM revealed the complete molecule. The trial structure was refined by block diagonal least squares method to a $R$-value of 0.19 with individual isotropic temperature factors. Introduction of anisotropic temperature factors reduced the $R$ value to 0.16. Few hydrogen atoms could be located from the $\Delta F$ map at this stage, remaining hydrogen atoms were generated from known geometry. The hydrogen atoms were given isotropic temperature factors of the non-hydrogen atoms to which they were attached and included in the refinement process but kept fixed. Few cycles of refinement reduced the $R$ value to 0.13 and $Rw$ to 0.08. At this stage bond lengths and angles were found to be normal however temperature factors of three end chain atoms (C1, C35 and C36) were very high. We tried to refine the structure by varying the occupancy of those atoms but no improvement was observed. 9 strong reflections were found to have secondary extinction effect. Exclusion of these reflections from the refinement process resulted to a $R$ value of 0.12 and $Rw$ of 0.08 which was accepted as final. At this stage $\Delta F$ map showed the highest peak of 0.41 e/Å$^3$ but not at any chemically sensible position. We used count statistics in weighting scheme. The scattering factors were taken from Cromer and Mann.[5] All the calculations were done using XTAL[6] and PC version of the NRCVAX[7] package utilities.

## RESULTS AND DISCUSSIONS

A perspective view of the molecule, along with numbering scheme, is shown in Figure 1. Positions of the non-hydrogen atoms, their anisotropic temperature factors, bond lengths and bond angles are given in Tables II–V. Bond lengths and angles, as mentioned earlier, are normal. We calculated the equations of different planes and the

TABLE I

Important crystallographic data

| Mol. formula C36 H40 N2 O2 | Mol. weight 532.73 gms |
|---|---|
| $a = 6.1162(29)$ Å | $V = 1508.238$ Å$^3$ |
| $b = 7.9157(25)$ Å | $D_{obs} = 1.65$ gms/cc |
| $c = 31.4213(81)$Å | $D_{cal} = 1.172$ gms/cc |
| $\alpha = 92.3927(216)^\circ$ | $z = 2$ |
| $\beta = 92.3545(313)^\circ$ | Space group $P\bar{1}$ |
| $\gamma = 96.5935(207)^\circ$ | $\lambda = 1.5418$ Å |

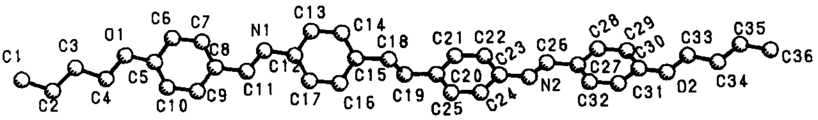

FIGURE 1 Perspective view of BBBT molecule along with numbering scheme.

TABLE II

Atomic parameters x,y,z and Biso of non-hydrogen atoms. E. S. Ds refer to the last digit printed

| | x | y | z | Biso |
|---|---|---|---|---|
| C1 | .484(3) | .6746(23) | .4435(5) | 12.3(13) |
| C2 | .5280(24) | .6843(20) | .3963(5) | 8.7(11) |
| C3 | .7312(22) | .5961(17) | .3859(4) | 6.7(10) |
| C4 | .7753(19) | .6063(16) | .3386(4) | 5.3(8) |
| O1 | .9651(12) | .5163(9) | .3329(3) | 4.9(5) |
| C5 | 1.0194(18) | .4908(14) | .2912(4) | 4.1(7) |
| C6 | 1.2088(19) | .4052(14) | .2873(4) | 4.6(7) |
| C7 | 1.2817(17) | .3738(13) | .2477(4) | 3.7(6) |
| C8 | 1.1773(18) | .4180(13) | .2118(4) | 3.5(6) |
| C9 | .9826(20) | .4977(15) | .2161(4) | 5.1(7) |
| C10 | .9104(18) | .5322(14) | .2564(4) | 3.9(6) |
| C11 | 1.2542(19) | .3912(14) | .1693(4) | 4.1(7) |
| N1 | 1.4350(14) | .3309(11) | .1628(3) | 4.7(6) |
| C12 | 1.5039(17) | .3025(13) | .1209(4) | 3.5(6) |
| C13 | 1.6940(18) | .2217(13) | .1195(4) | 4.2(7) |
| C14 | 1.7759(19) | .1789(13) | .0800(4) | 4.3(7) |
| C15 | 1.6701(19) | .2149(13) | .0432(4) | 4.0(7) |
| C16 | 1.4811(19) | .2985(14) | .0440(4) | 4.4(7) |
| C17 | 1.4012(18) | .3374(14) | .0838(4) | 4.4(7) |
| C18 | 1.7671(23) | .1649(15) | .0010(4) | 6.4(9) |
| C19 | 1.7789(22) | .3002(15) | −.0313(4) | 5.8(8) |
| C20 | 1.8780(20) | .2462(14) | −.0715(4) | 5.2(8) |
| C21 | 2.0977(19) | .3137(15) | −.0823(4) | 5.0(7) |
| C22 | 2.1854(19) | .2648(15) | −.1194(4) | 4.9(7) |
| C23 | 2.0641(17) | .1496(13) | −.1502(4) | 3.7(7) |
| C24 | 1.8476(18) | .0829(14) | −.1388(4) | 4.2(7) |
| C25 | 1.7625(19) | .1334(14) | −.1021(4) | 4.9(7) |
| N2 | 2.1351(15) | .0873(11) | −.1887(3) | 4.8(6) |
| C26 | 2.3178(19) | .1366(15) | −.2011(4) | 5.1(7) |
| C27 | 2,3899(18) | .0731(14) | −.2414(4) | 4.3(7) |
| C28 | 2.5992(19) | .1364(15) | −.2560(4) | 5.2(7) |
| C29 | 2.6718(19) | .0770(16) | −.2941(4) | 6.1(8) |
| C30 | 2.5416(19) | −.0462(15) | −.3201(4) | 4.9(8) |
| C31 | 2.3361(18) | −.1117(15) | −.3046(4) | 4.7(7) |
| C32 | 2.2639(18) | −.0540(15) | −.2676(4) | 4.7(7) |
| O2 | 2.5953(12) | −.1082(10) | −.3581(3) | 5.5(5) |
| C33 | 2.8063(21) | −.0573(18) | −.3731(5) | 7.4(10) |
| C34 | 2.8300(24) | −.1643(19) | −.4147(5) | 9.4(11) |
| C35 | 3.048(3) | −.122(3) | −.4309(6) | 14.2(15) |
| C36 | 3.078(3) | −.233(3) | −.4717(6) | 16.2(17) |

Biso is the Mean of the Principal Axes of the Thermal Ellipsoid

TABLE III

Thermal parameters Uij × 100 of non-hydrogen atoms. E. S. Ds refer to the last digit printed

| | u11 | u22 | u33 | u12 | u13 | u23 |
|---|---|---|---|---|---|---|
| C1 | 17.2(19) | 20.0(20) | 9.4(16) | −.5(15) | 7.3(14) | −3.3(15) |
| C2 | 9.9(14) | 14.7(16) | 8.6(14) | 2.6(11) | 2.1(11) | −2.5(12) |
| C3 | 7.7(12) | 10.9(14) | 6.5(12) | .7(10) | .3(10) | −2.1(10) |
| C4 | 4.6(10) | 9.3(12) | 6.1(11) | 1.0(8) | .5(8) | −3.1(9) |
| O1 | 4.5(6) | 6.2(6) | 7.5(7) | −1.4(5) | .9(5) | −2.1(6) |
| C5 | 4.6(9) | 5.0(9) | 5.1(10) | −2.7(7) | .5(8) | −1.3(8) |
| C6 | 4.9(9) | 5.2(10) | 6.9(11) | −1.9(7) | .8(8) | −.8(8) |
| C7 | 4.0(9) | 3.5(8) | 6.0(10) | −1.0(6) | −.4(8) | −.1(8) |
| C8 | 4.3(8) | 3.3(8) | 5.2(9) | −1.6(6) | −.3(7) | −.8(7) |
| C9 | 6.0(10) | 5.3(10) | 7.4(11) | −1.7(7) | .9(8) | −1.3(8) |
| C10 | 4.5(9) | 4.2(9) | 5.8(10) | −1.0(7) | 1.0(7) | −.2(8) |
| C11 | 5.6(9) | 3.5(8) | 5.9(10) | −1.6(7) | .2(8) | −1.5(7) |
| N1 | 3.8(7) | 5.2(8) | 8.3(9) | −1.7(6) | .7(7) | −2.7(7) |
| C12 | 4.0(8) | 2.8(8) | 5.8(10) | −2.0(6) | .5(7) | −.4(7) |
| C13 | 5.0(9) | 2.6(8) | 8.3(11) | −.5(6) | 1.4(8) | −.4(8) |
| C14 | 5.3(9) | 3.5(8) | 6.9(11) | −1.4(7) | 1.0(8) | −.5(8) |
| C15 | 6.4(10) | 2.5(8) | 6.1(10) | −1.0(7) | 1.6(8) | −.9(7) |
| C16 | 5.7(10) | 3.9(9) | 7.0(11) | −.5(7) | .5(8) | −.8(8) |
| C17 | 4.9(9) | 4.5(9) | 6.9(11) | −1.4(7) | .9(8) | −.8(8) |
| C18 | 11.2(13) | 5.3(10) | 8.3(13) | 2.2(9) | 2.3(11) | −.8(9) |
| C19 | 10.3(12) | 4.7(10) | 6.9(12) | −.4(8) | 3.1(10) | −1.3(9) |
| C20 | 6.8(10) | 4.1(9) | 8.9(12) | .2(7) | 3.8(9) | −.3(9) |
| C21 | 5.7(10) | 6.5(10) | 6.0(10) | −2.1(8) | 1.3(8) | −2.1(8) |
| C22 | 4.3(9) | 7.0(11) | 6.6(11) | −2.3(7) | 1.0(8) | −1.8(9) |
| C23 | 4.0(9) | 5.0(9) | 5.1(9) | .4(7) | .8(7) | −.7(8) |
| C24 | 3.6(8) | 5.8(10) | 6.0(10) | −1.6(7) | 1.1(8) | −1.1(8) |
| C25 | 4.9(9) | 4.9(9) | 8.5(12) | −.7(7) | 1.5(9) | −2.1(9) |
| N2 | 5.1(8) | 4.5(7) | 7.8(9) | −2.6(6) | 1.5(7) | −1.7(6) |
| C26 | 5.8(10) | 4.6(9) | 8.4(12) | −1.4(7) | 2.4(9) | −1.5(8) |
| C27 | 4.4(9) | 5.8(10) | 5.3(10) | −1.0(7) | .5(8) | −2.2(8) |
| C28 | 4.3(9) | 6.9(10) | 7.9(12) | −2.3(7) | 2.3(8) | −1.7(9) |
| C29 | 4.9(10) | 8.8(12) | 8.4(12) | −3.0(8) | 2.2(9) | −3.6(10) |
| C30 | 5.9(10) | 7.2(11) | 5.1(10) | −.6(8) | −.1(8) | −2.2(8) |
| C31 | 4.4(9) | 5.6(9) | 7.3(11) | −1.9(7) | 1.0(8) | −1.0(8) |
| C32 | 3.8(9) | 6.8(11) | 6.7(11) | −2.4(7) | .9(8) | −1.3(9) |
| O2 | 5.8(7) | 8.1(7) | 6.5(7) | −1.5(5) | 2.2(5) | −2.4(6) |
| C33 | 5.9(11) | 14.0(15) | 8.0(13) | .5(10) | 2.0(10) | −1.9(12) |
| C34 | 9.1(13) | 14.0(16) | 12.1(16) | −1.4(11) | 5.7(12) | −3.3(13) |
| C35 | 16.5(20) | 23.6(24) | 13.4(19) | .7(17) | 7.6(16) | −5.0(17) |
| C36 | 21.2(23) | 27.9(26) | 13.7(21) | 7.1(19) | 8.7(18) | −4.8(19) |

Anisotropic Temperature Factors are of the form − −
Temp = −2 × Pi × Pi × (h × h × u11 × astar × astar + − − − + 2 × h × k × u12 × astar × bstar + − − −)

dihedral angles between them which are given in Tables VI and VII. It is observed that all the phenyl rings are planar. Moreover the symmetrical portions of the molecule C1–C18 and C19–C36 also show a high degree of planarity, but the angle between them is 63.5°. However minimum energy (bond, angle, torsion and van der waals') configuration of a free molecule shows an angle of about 15° between those planes.

The length of the molecule (C1–C36) in the crystalline state is 34.3 Å which is equal to the length found from a stereomodel of the molecule. This suggests that the molecules are in their most extended conformation. The direction cosines of the molecular long

TABLE IV

Bond distances involving non-hydrogen atoms. E. S Ds refer to the last digit printed

| | | | |
|---|---|---|---|
| C(1)–C(2) | 1.520(22) | C(18)–C(19) | 1.504(18) |
| C(2)–C(3) | 1.534(20) | C(19)–C(20) | 1.490(18) |
| C(3)–C(4) | 1.525(19) | C(20)–C(21) | 1.447(16) |
| C(4)–O(1) | 1.444(14) | C(20)–C(25) | 1.397(17) |
| O(1)–C(5) | 1.377(14) | C(21)–C(22) | 1.364(17) |
| C(5)–C(6) | 1.414(17) | C(22)–C(23) | 1.424(16) |
| C(5)–C(10) | 1.330(17) | C(23)–C(24) | 1.434(15) |
| C(6)–C(7) | 1.361(17) | C(23)–N(2) | 1.392(15) |
| C(7)–C(8) | 1.353(16) | C(24)–C(25) | 1.348(17) |
| C(8)–C(9) | 1.419(17) | N(2)–C(26) | 1.225(14) |
| C(8)–C(11) | 1.450(17) | C(26)–C(27) | 1.447(17) |
| C(9)–C(10) | 1.384(18) | C(27)–C(28) | 1.421(15) |
| C(11)–N(1) | 1.274(15) | C(27)–C(32) | 1.409(15) |
| N(1)–C(12) | 1.418(15) | C(28)–C(29) | 1.376(18) |
| C(12)–C(13) | 1.391(16) | C(29)–C(30) | 1.399(16) |
| C(12)–C(17) | 1.353(17) | C(30)–C(31) | 1.417(16) |
| C(13)–C(14) | 1.401(18) | C(30)–O(2) | 1.340(14) |
| C(14)–C(15) | 1.360(18) | C(31)–C(32) | 1.341(18) |
| C(15)–C(16) | 1.397(17) | O(2)–C(33) | 1.413(15) |
| C(15)–C(18) | 1.530(18) | C(33)–C(34) | 1.549(20) |
| C(16)–C(17) | 1.398(17) | C(34)–C(35) | 1.455(20) |
| | | C(35)–C(36) | 1.550(20) |

TABLE V

Bond angles involving non-hydrogen atoms. E. S. Ds refer to the last digit printed

| | | | |
|---|---|---|---|
| C(1)–C(2)–C(3) | 110.3(13) | C(18)–C(19)–C(20) | 112.6(10) |
| C(2)–C(3)—C(4) | 110.3(12) | C(19)–C(20)–C(21) | 122.0(11) |
| C(3)–C(4)–O(1) | 104.8(10) | C(19)–C(20)–C(25) | 122.5(10) |
| O(1)–C(5)–C(6) | 112.9(11) | C(20)–C(21)–C(22) | 121.4(10) |
| O(1)–C(5)–C(10) | 127.1(11) | C(21)–C(22)–C(23) | 122.1(10) |
| C(6)–C(5)–C(10) | 120.0(11) | C(22)–C(23)–C(24) | 115.7(10) |
| C(5)–C(6)–C(7) | 118.8(11) | C(22)–C(23)–N(2) | 127.8(10) |
| C(6)–C(7)–C(8) | 122.6(11) | C(24)–C(23)–N(2) | 116.4(9) |
| C(7)–C(8)–C(9) | 118.0(11) | C(23)–C(24)–C(25) | 121.5(10) |
| C(7)–C(8)–C(11) | 124.0(11) | C(20)–C(25)–C(24) | 123.8(10) |
| C(9)–C(8)–C(11) | 118.1(11) | C(23)–N(2)–C(26) | 121.2(10) |
| C(8)–C(9)–C(10) | 119.5(12) | N(2)–C(26)–C(27) | 121.0(11) |
| C(8)–C(11)–N(1) | 122.1(11) | C(26)–C(27)–C(32) | 123.3(10) |
| C(11)–N(1)–C(12) | 120.9(10) | C(28)–C(27)–C(32) | 116.5(11) |
| N(1)–C(12)–C(13) | 113.2(10) | C(27)–C(28)–C(29) | 121.2(10) |
| N(1)–C(12)–C(17) | 127.5(10) | C(28)–C(29)–C(30) | 121.4(11) |
| C(13)–C(12)–C(17) | 119.2(11) | C(29)–C(30)–C(31) | 116.9(11) |
| C(12)–C(13)–C(14) | 119.5(12) | C(29)–C(30)–O(2) | 125.7(11) |
| C(13)–C(14)–C(15) | 120.4(11) | C(31)–C(30)–O(2) | 117.5(10) |
| C(14)–C(15)–C(16) | 120.8(11) | C(30)–C(31)–C(32) | 122.1(10) |
| C(14)–C(15)–C(18) | 118.1(11) | C(27)–C(32)–C(31) | 122.0(10) |
| C(16)–C(15)–C(18) | 121.1(11) | C(30)–O(2)–C(33) | 119.3(9) |
| C(15)–C(16)–C(17) | 117.6(11) | O(2)–C(33)–C(34) | 107.5(9) |
| C(12)–C(17)–C(16) | 122.5(11) | C(33)–C(34)–C(35) | 110.1(10) |
| C(15)–C(18)–C(19) | 114.8(10) | C(34)–C(35)–C(36) | 110.7(10) |

TABLE VI

Equations of least-squares planes and rms displacements (Å) of the atoms from the planes

Plane 1 $2.691(17)x + 6.645(15)y + 0.06(7)z = 5.99(3)$

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Atoms in plane | C5 | C6 | C7 | C8 | C9 | C10 | C12 | C13 | C14 | C15 | C16 | C17 |
| Disp. | .03 | −.03 | −.04 | −.03 | −.02 | .01 | .08 | .05 | −.02 | −.06 | −.02 | .03 |
| Atoms out of plane | C1 | C2 | C3 | C4 | O1 | C11 | N1 | C18 | | | | |
| Disp. | −.18 | .00 | −.04 | .15 | .06 | .00 | .08 | −.14 | | | | |

Plane 2 $2.810(17)x - 6.365(16)y + 15.12(4)z = 2.61(4)$

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Atoms in plane | C20 | C21 | C22 | C23 | C24 | C25 | C27 | C28 | C29 | C30 | C31 | C32 |
| Disp. | .02 | .05 | .04 | −.03 | −.04 | −.05 | −.01 | −.04 | −.04 | −.01 | .06 | .05 |
| Atoms out of plane | C19 | N2 | C26 | O2 | C33 | C34 | C35 | C36 | | | | |
| Disp. | .01 | −.02 | −.01 | −.04 | .00 | .12 | .22 | .39 | | | | |

Plane 3 $2.80(3)x + 6.565(23)y - 0.41(16)z = 5.94(5)$

| | | | | | | |
|---|---|---|---|---|---|---|
| Atoms in plane | C5 | C6 | C7 | C8 | C9 | C10 |
| Disp. | .02 | −.02 | .00 | −.01 | −.01 | .00 |

Plane 4 $2.80(3)x + 6.577(23)y - 1.33(16)z = 6.03(4)$

| | | | | | | |
|---|---|---|---|---|---|---|
| Atoms in plane | C12 | C13 | C14 | C15 | C16 | C17 |
| Disp. | .00 | .00 | .00 | −.01 | .01 | −.01 |

Plane 5 $2.68(3)x - 6.539(23)y + 14.56(14)z = 2.37(7)$

| | | | | | | |
|---|---|---|---|---|---|---|
| Atoms in plane | C20 | C21 | C22 | C23 | C24 | C25 |
| Disp. | .01 | −.01 | .01 | −.01 | .01 | −.01 |

Plane 6 $3.00(3)x - 6.20(3)y + 15.26(14)z = 3.04(9)$

| | | | | | | |
|---|---|---|---|---|---|---|
| Atoms in plane | C27 | C28 | C29 | C30 | C31 | C32 |
| Disp. | .01 | .00 | .01 | −.01 | .01 | .00 |

axis, defined as best fitted line through all atoms, are .51, −.15 and −.85 indicating that the molecule makes an angle 32° with $z$-axis.

Crystal structure of the molecule viewed along $x$, $y$ and $z$ axes are shown in Figures 2–4. It is observed that molecules are arranged in layers inclined to xy–plane and the layers are stacked along $z$-axis. This tilted layer like structure is precursor to the tilted smectic phase.

Packing fraction of the structure, calculated using method of Kitaigorodosky,[8] is found to be 0.73. All intermolecular contact distances less than 3.65 Å are calculated

TABLE VII

Dihedral angles (°) between various planes

| Plane | Plane | Angle | Plane | Plane | Angle |
|---|---|---|---|---|---|
| 1 | 2 | 116.5 | 2 | 5 | 2.2 |
| 1 | 3 | 1.4 | 2 | 6 | 2.3 |
| 1 | 4 | 2.7 | 3 | 4 | 1.7 |
| 1 | 5 | 118.7 | 3 | 5 | 118.2 |
| 1 | 6 | 114.2 | 3 | 6 | 113.7 |
| 2 | 3 | 116.0 | 4 | 5 | 119.1 |
| 2 | 4 | 117.0 | 4 | 6 | 114.7 |
|  |  |  | 5 | 6 | 4.4 |

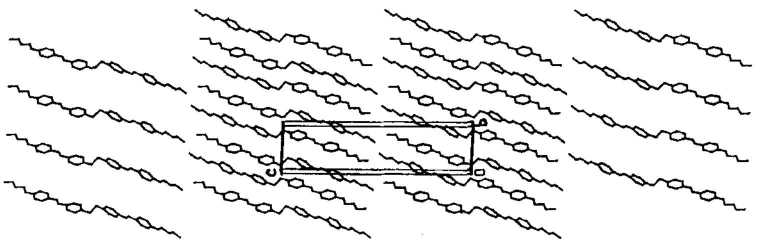

FIGURE 2 Structure of the molecule viewed along $x$-axis.

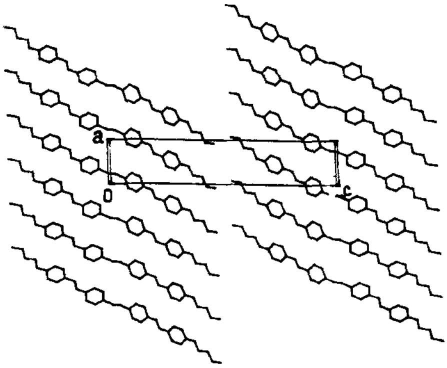

FIGURE 3 Structure of the molecule viewed along $y$-axis.

and listed in Table VIII. We find few C—C distances of centro-symmetrically related molecules are less than the sum of their van der Waals' radii.

We, therefore,like to infer that twisted molecular conformation and dispersion forces resulting from van der Waals' interactions give rise to very efficient packing of the molecules. The high melting point of the compound is a result of this packing. Change of molecular conformation at high temperature might cause the pretransition effect observed in texture study. At the Cr-Sm transition temperature the molecules gain sufficient energy to move within the layers and to undergo rotations about the long

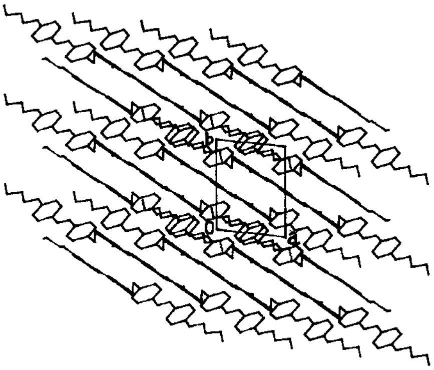

FIGURE 4 Structure of the molecule viewed along z-axis.

TABLE VIII

Intermolecular contact distances less than 3.65 Å

| | | | | | |
|---|---|---|---|---|---|
| O1 | C31$^{a}$ | 3.566 | C10 | C26$^{c}$ | 3.585 |
| C5 | C31$^{a}$ | 3.548 | N1 | C24$^{a}$ | 3.561 |
| C6 | C30$^{b}$ | 3.547 | C12 | C24$^{a}$ | 3.603 |
| C6 | O2$^{b}$ | 3.575 | C13 | C23$^{b}$ | 3.588 |
| C9 | C23$^{c}$ | 3.575 | C13 | N2$^{b}$ | 3.555 |
| C9 | N2$^{c}$ | 3.575 | C17 | C19$^{c}$ | 3.624 |
| C9 | C26$^{c}$ | 3.639 | | | |

Superscripted atoms are at a) 3–x, –y, –z: b) 4–x, –y, –z: c) 3–x, 1–y, –z

molecular axis. The Cr-Sm transition is thus displacive type contrary to reconstitutive type[9] usually observed at this transitions.

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