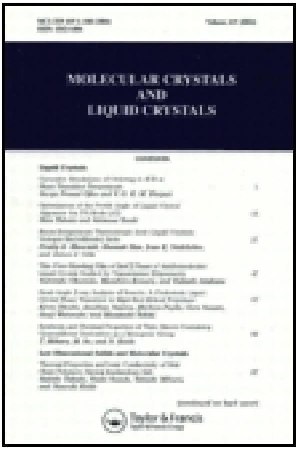

Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

Crystal and Molecular Structure of a Mesogenic Compound with a Large Lateral Benzene Ring-containing Substituent

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To cite this article: F. Hoffmann , H. Hartung , W. Weissflog , P. G. Jones & A. Chrapkowski (1996): Crystal and Molecular Structure of a Mesogenic Compound with a Large Lateral Benzene Ring-containing Substituent, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 281:1, 205-214

To link to this article: http://dx.doi.org/10.1080/10587259608042245

Mol. Cryst. Liq. Cryst., 1996, Vol. 281, pp. 205–214
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Crystal and Molecular Structure of a Mesogenic Compound with a Large Lateral Benzene Ring-containing Substituent

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(Received June 12, 1994; in final form October 13, 1994)

The crystal and molecular structure of the nematic compound 4-nitrobenzyl 2,5-bis(4-ethyloxybenzoyloxy)-benzoate (NEBB) has been determined at − 100°C and at room temperature by X-ray analysis. NEBB crystallizes in space group $P\bar{1}$ with two molecules in a unit cell of dimensions (values of the room temperature investigation are given in brackets) $a = 9.547(3)$ [9.643 (2)], $b = 12.294(4)$ [12.452 (3)], $c = 13.536(5)$ [13.660 (3)] Å, $\alpha = 68.67(3)$ [68.34 (1)], $\beta = 75.15(3)$ [74.40 (1)], $\gamma = 70.98(3)$ [69.65 (1)]°. The structure was solved by direct methods and refined on F^2 to R values of $wR2 = 0.107$ [0.109] and $R1 = 0.043$ [0.049], respectively, for 3347 [2422] observed unique reflections.

The phenylene bis(benzoate) basic core of the NEBB molecule has a non-planar but optimally stretched shape and the lateral ring-containing substituent is oriented parallel to it. The molecules in the crystalline state are arranged perfectly parallel to each other with an anti-parallel orientation of the neighbours. They form lamellar sheets and a tilted layer structure.

Keywords: Crystalline mesogens, X-ray analysis, molecular structure, crystal packing, laterally branched mesogens, phenylene bis(benzoates).

1. INTRODUCTION

The molecular shape of most liquid crystals is rod-like or disc-like. However, systematic investigations concerning the relation between molecular structure and liquid

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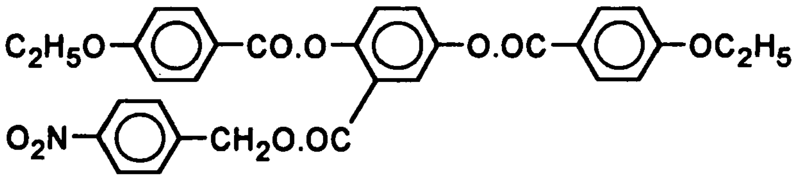
crystalline properties, as performed by several groups in the last few years, have shown that deviations from the "perfect" molecular architecture are possible without loss of mesomorphic behaviour.[1] In this connection lateral substitution of mesogenic cores by small groups was often involved. But a three-ring compound having a long-chain substituent at the middle ring can also show a nematic phase because the laterally attached aliphatic chain is aligned in the direction of the molecular long axis.[2,3] Mesogens bearing aromatic branches at a lateral position are of great interest since their shape is neither rod-like nor disc-like. As we have discovered, unexpected high clearing temperatures can be observed if the lateral cyclic segment is linked by an odd-numbered spacer to the basic mesogen.[4] In contrast to the laterally long-chain substituted nematogens, for mesogens with lateral branches containing suitably (e.g. electron-withdrawing groups) substituted aromatic rings also smectic phases were found. The 4-nitrobenzyl 2,5-bis (4-*n*-alkyloxybenzoyloxy)benzoates are such a class of compounds. Nematic and/or smectic *A* phases can be observed at temperatures up to 165°C. As shown by dielectric and X-ray measurements, the S_A phases are characterized by an extremely strong antiparallel correlation of the molecules and a packing with partial intercalation of the terminal alkyl chains.[5,6] The striking feature of the short range order is also revealed by miscibility experiments. In binary systems of 4-nitrobenzyl 2,5-bis(4-*n*-alkyloxybenzoyloxy)benzoates with selected rod-like components, the S_A phase can be stabilized or destabilized depending on the molecular length of the rod-like molecule. This unusual behaviour is mainly due to steric interaction and can be interpreted by a simple packing model proposed by Diele *et al.*:[6] according to this model, the smectic *A* phase of the 4-nitrobenzyl 2,5-bis(4-*n*-alkyloxy-benzoyloxy)benzoates should have a molecular arrangement showing gaps between adjacent molecules. A reason for this peculiarity is that different parts of a molecule have quite different cross-sections. The breadth of the fragment containing the lateral substituent with a parallel orientation to the mesogenic core is more than twice the breadth of the other terminal part of the molecule.

For the above-mentioned and other related reasons, it seemed of interest to acquire an exact knowledge of both the molecular structure and packing of one member of the homologous series under discussion. Because of the difficulties of preparing suitable single crystals only the 4-nitrobenzyl 2,5-bis(4-ethyloxybenzoyloxy)benzoate (NEBB) was available for X-ray investigations.



Moreover, the X-ray analysis of NEBB was also initiated by the general lack of precise structural data concerning mesogens with an unconventional molecular structure and in particular for such compounds with a large lateral substituent.[7] The following transition temperatures, obtained by calorimetric measurements, characterize the phase behaviour of NEBB:

Cr 183–185°C (*N* 166°C) *Is*

2. EXPERIMENTAL

2.1 Synthesis

NEBB was prepared by acylation of 4-nitrobenzyl 2,5-dihydroxybenzoate with 4-*n*-ethyloxy-benzoylchloride in toluene in the presence of triethylamine. Recrystallization from pentanol-1 yielded the pure compound.

2.2 X-ray analysis

Optically clear, colorless crystals of prismatic habit were obtained by recrystallization from pentanol-1. The results of a structure analysis performed with intensity data measured at room temperature by means of a Stoe STADI 4 diffractometer were unsatisfactory: the *R* value and estimated standard deviations were rather high. Some details of the estimated molecular structure and anisotropic displacement parameters indicate the presence of disorder of some structural fragments (especially of the nitro group). We have observed structural disorder several times for relatively low-melting crystalline mesogens.[7] For this reason, measurements were repeated on a Siemens R3m/V diffractometer at − 100°C.

Crystal data and relevant details of X-ray analysis for both the low (LT) and room temperature (RT) investigations are summarized in Table I. Lattice parameters were derived by a least-squares treatment of the setting angles for 48 and 70 reflections, respectively. The structures were solved by direct methods and refined by full-matrix least-squares refinement on F^2 with anisotropic displacement parameters for the non-H atoms. The positions of the H atoms were geometrically calculated and refined isotropically using the riding model. For the LT investigation an empirical extinction correction was applied to F_c with $F_{c,corr} = F_c \cdot k[1 + 10^{-6} \cdot \chi \cdot F_c^2 \cdot \lambda^3 \cdot \sin 2\Theta]^{1/4}$ where χ and k were refined to 8(1) and 0.671 (1), respectively. For the calculations programs SHELXS-86[8]. SHELXL-93[9], and Siemens XP[10] were used.

Final atomic parameters of the low temperature structure are given in Table II. Those of the room temperature structure together with further details of both crystal structure determinations are available on request from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information, D-76344 Eggenstein-Leopoldshafen, on quoting the depository numbers CSD-400897 (LT) and CSD-400898 (RT), respectively, the names of the authors, and the journal citation.

3. RESULTS AND DISCUSSION

As already mentioned above, X-ray analyses of NEBB both at room temperature and − 100°C have been performed giving in principle identical results. Furthermore,

TABLE 1

Crystal data and details of X-ray structure analysis of NEBB

Compound	NEBB (room temperature)	NEBB (low temperature)
Empirical formula	$C_{32}H_{27}NO_{10}$	
Molecular weight ($g\,mol^{-1}$)	585.55	
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Lattice parameters		
a(Å)	9.643(2)	9.547(3)
b(Å)	12.452 (3)	12.294(4)
c(Å)	13.660(3)	13.536(5)
α(°)	68.34(1)	68.67(3)
β(°)	74.40(1)	75.15(3)
γ(°)	69.65(1)	70.98(3)
V(Å^3)	1410.5(8)	1381.9(8)
Z	2	2
F(000)	612	
D_{meas}($g\,cm^{-3}$)	1.38(1)	–
D_{calc}($g\,cm^{-3}$)	1.379	1.407
μ(MoK_α)(cm^{-1})	1.03	1.06
Crystal size (mm)	0.49 × 0.36 × 0.21	0.56 × 0.36 × 0.28
Wavelength (Å)	0.71073	
Check reflections	3, ± 1.6% max. intensity variation	3, ± 3% max, intensity variation
scan method	ω/θ-scan	ω-scan
Range of measurement (°)	$1.63 \leqslant \theta \leqslant 23.03$	$3.01 \leqslant \theta \leqslant 25.05$
Min. h, k, l/max, h, k, l	$\overline{10}, \overline{13}, \overline{15}/10, 13, 15$	$\overline{11}, \overline{14}, \overline{16}/10, 13, 1$
$R_{(int)}$	0.024	0.058
Measured reflections	7842	5105
Observed reflections, $I > 2\sigma(I)$	2422	3347
Reflections used in refinement	3921	4873
Refined parameters	395	391
Weighting scheme		
$w = 1/[\sigma^2(F_0^2) + (a \cdot P)^2 + b \cdot P]$,	$a = 0.0723$	$a = 0.0705$
$P = (F_0^2 + 2F_c^2)/3$	$b = 0.0417$	$b = 0.3905$
Max. shift/σ (last least squares cycle)	0.007	0.000
Min./max. heights in the final $\Delta\rho$ map (e$Å^{-3}$)	− 0.51/0.54	− 0.24/0.25
R1/wR2/S (observed data)*	0.049/0.109/1.053	0.043/0.107/1.011
R1/wR2/S (all data)*	0.088/0.124/1.213	0.070/0.133/1.016

*Values of R1, *wR*2 and S are defined as $R1 = \Sigma \| F_0| - |F_c\| / \Sigma |F_0|$, $wR2 = \{\Sigma[w(F_0^2 - F_c^2)^2] / \Sigma[w(F_0^2)^2]\}^{1/2}$, $S = \{\Sigma[w(F_0^2 - F_c^2)^2]/(n-p)\}^{1/2}$, where n is the number of reflections and p is the total number of parameters refined.

heating to the melting point led to no observable phase transition. Therefore, polymorphism in the solid state can be excluded for NEBB. Because of improved precision, all data and figures in the following discussion refer to the low temperature investigation.

3.1 Molecular structure

The molecular structure of NEBB including used atomic numbering is illustrated by a thermal motion plot[10] in Figure 1. A realistic picture of the molecular shape can be

TABLE II

Final fractional coordinates and equivalent isotropic displacement parameters (Å^2) for non-H atoms

$$U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

Atom	x/a	y/b	z/c	U_{eq}
C1	−0.4033(2)	1.0547(2)	0.5712(2)	0.0419(9)
C2	−0.3228(2)	0.9492(2)	0.6536(2)	0.0365(9)
C3	−0.0683(2)	0.8306(2)	0.6682(2)	0.0298(8)
C4	−0.1106(2)	0.7555(2)	0.7693(2)	0.0361(8)
C5	−0.0028(2)	0.6603(2)	0.8220(2)	0.0348(8)
C6	0.1465(2)	0.6388(2)	0.7744(2)	0.0270(7)
C7	0.1874(2)	0.7160(2)	0.6733(2)	0.0307(8)
C8	0.0812(2)	0.8110(2)	0.6204(2)	0.0324(8)
C9	0.2638(2)	0.5373(2)	0.8266(2)	0.0266(8)
C10	0.3212(2)	0.3838(2)	0.9849(1)	0.0269(7)
C11	0.3696(2)	0.2673(2)	0.9754(1)	0.0274(7)
C12	0.4814(2)	0.1824(2)	1.0344(2)	0.0296(8)
C13	0.5394(2)	0.2160(2)	1.0989(2)	0.0299(8)
C14	0.4858(2)	0.3287(2)	1.1122(2)	0.0339(9)
C15	0.3764(2)	0.4134(2)	1.0539(2)	0.0318(8)
C16	0.7967(2)	0.1312(2)	1.1050(2)	0.0332(8)
C17	0.9093(2)	0.0410(2)	1.1709(2)	0.0302(8)
C18	0.8721(2)	−0.0183(2)	1.2795(2)	0.0316(8)
C19	0.9813(2)	−0.1025(2)	1.3377(2)	0.0328(8)
C20	1.1292(2)	−0.1302(2)	1.2873(2)	0.0317(8)
C21	1.1685(2)	−0.0708(2)	1.1798(2)	0.0381(9)
C22	1.0583(2)	0.0150(2)	1.1225(2)	0.0372(9)
C23	1.3780(2)	−0.2610(2)	1.3040(2)	0.043(1)
C24	1.4532(3)	−0.3647(2)	1.3906(2)	0.053(1)
C25	0.3104(2)	0.2227(2)	0.9092(2)	0.0315(8)
C26	0.1422(3)	0.2678(2)	0.7899(2)	0.041(1)
C27	0.0298(2)	0.3766(2)	0.7336(2)	0.0343(9)
C28	−0.1223(2)	0.3873(2)	0.7683(2)	0.0370(9)
C29	−0.2275(2)	0.4861(2)	0.7159(2)	0.0351(9)
C30	−0.1759(2)	0.5735(2)	0.6289(2)	0.0292(8)
C31	−0.0256(2)	0.5662(2)	0.5923(2)	0.0370(9)
C32	0.0770(2)	0.4671(2)	0.6459(2)	0.0384(9)
N1	−0.2866(2)	0.6773(2)	0.5722(1)	0.0368(8)
O1	−0.1660(2)	0.9254(1)	0.6093(1)	0.0363(6)
O2	0.3936(2)	0.5095(1)	0.7870(1)	0.0357(6)
O3	0.2115(1)	0.4756(1)	0.9288(1)	0.0287(5)
O4	0.6529(2)	0.1306(1)	1.1566(1)	0.0346(6)
O5	0.8223(2)	0.1977(2)	1.0162(1)	0.0551(7)
O6	1.2266(2)	−0.2181(1)	1.3516(1)	0.0401(6)
O7	0.3501(2)	0.1197(1)	0.9080(1)	0.0530(8)
O8	0.2071(2)	0.3079(1)	0.8528(1)	0.0384(6)
O9	−0.2439(2)	0.7438(2)	0.4862(1)	0.0663(8)
O10	−0.4179(2)	0.6931(2)	0.6139(2)	0.0653(8)

visualized by the space-filling model[10] using the van der Waals radii given by Bondi[11] for plotting the atom contours in Figure 2. Essential parameters of the molecular geometry are summarized in Tables III and IV.

The most interesting features of the molecular shape of NEBB are the alignment of the lateral branch with respect to the basis molecule and the mutual orientation of the

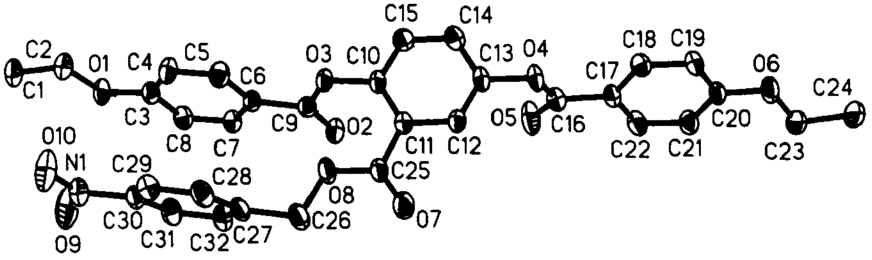


FIGURE 1 Molecular structure of NEBB with atom numbering.

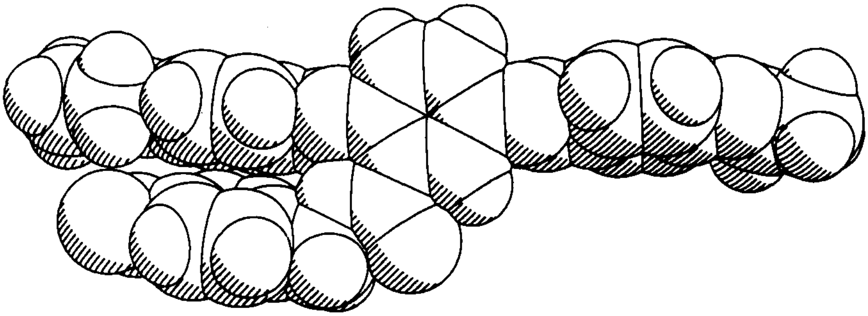


FIGURE 2 Space filling model of the molecular structure of NEBB.

four benzene rings. As expected and known for other related compounds,[7] the phenylene bis(benzoate) three-ring skeleton (= the mesogenic core) has a non-planar but optimally extended shape. The lateral substituent is largely aligned parallel to the long axis of the basic moiety. Therefore, the molecule is characterized by two approximately rod-like parts with different cross-sections. In contrast, the mesogenic compound 4-ethoxy-3'-(4-ethoxy-phenyliminomethyl)-4'-(4-methoxy-benzoyloxy)azobenzene which also contains a lateral branch with a benzene ring, has been found to be distinctly bifurcated with the shape of a slightly deformed Y.[12]

The octyl homologue of NEBB has been studied intensively by Diele *et al.*[6] in order to obtain an insight into the structure of the S_A phase of the pure compound, as well as of mixtures of it with rod-like molecules. The authors used a model for the molecular shape based on the assumption that the benzene rings I (C3...C8), III (C17...C22) and IV (C27...C32) are coplanar and perpendicular to ring II (C10...C15). The correctness of this assumption is confirmed at a good approximation by the molecular structure determined for NEBB. The interplanar angles in question are I/III 11.3, I/IV 13.8, III/IV 24.3, I/II 92.9, II/III 101.7 and II/IV 79.2°. The orientation of the carboxylic groups with respect to the O- and C-bonded adjacent benzene rings are described by the appropriate torsion angles given in Table IV. All these data agree quite well with the results of our previous investigations of mesogenic phenyl benzoates in general[13] and on phenylene bis(benzoates) in particular,[14,15] but two peculiarities seem to be worth mentioning. As already observed for the laterally alkyl branched 2-*n*-nonyl-1,

TABLE III

Selected bond lengths and angles (esd's in parentheses)

Atoms	Distance (Å)	Atoms	Angle (°)
Carboxylic groups			
C9—O2	1.207(2)	C6—C9—O2	125.5(2)
C9—O3	1.366(2)	C6—C9—O3	112.7(2)
C9—C6	1.471(3)	O2—C9—O3	121.8(2)
O3—C10	1.401(2)	C9—O3—C10	114.9(1)
C16—O5	1.200(3)	C17—C16—O5	126.2(2)
C16—O4	1.372(2)	C17—C16—O4	112.0(2)
C16—C17	1.475(3)	O5—C16—O4	121.9(2)
O4—C13	1.408(2)	C16—O4—C13	115.0(1)
C25—O7	1.202(3)	C11—C25—O7	123.4(2)
C25—O6	1.336(3)	C11—C25—O8	113.5(2)
C25—C11	1.498(3)	O7—C25—O8	123.1(2)
O8—C26	1.458(3)	C25—O8—C26	115.6(2)
Nitro group			
N1—O9	1.214(3)	O9—N1—O10	122.7(2)
N1—O10	1.219(2)	O9—N1—C30	118.8(2)
N1—C30	1.469(3)	O10—N1—C30	118.5(2)
Mean values for benzene rings			
Ring I (C3...C8)			
C—C	1.387 (6)	C—C—C	120(1)
Ring II (C10...C15)			
C—C	1.386(11)	C—C—C	120(2)
Ring III (C17...C22)			
C—C	1.388(5)	C—C—C	120(1)
Ring IV (C27...C32)			
C—C	1.385(6)	C—C—C	120(2)

TABLE IV

Selected torsion angles

Atoms	Angle (°)	Atoms	Angle (°)
Carboxylic groups			
C5—C6—C9—O2	175.3(2)	C5—C6—C9—O3	− 5.7(2)
C6—C9—O3—C10	− 175.4(2)	O2—C9—O3—C10	3.7(2)
C9—O3—C10—C11	− 84.4(2)	C9—O3—C10—C15	97.7(2)
C18—C17—C16—O5	168.7(2)	C18—C17—C16—O4	− 12.2(2)
C17—C16—O4—C13	179.0(2)	O5—C16—O4—C13	− 1.9(2)
C16—O4—C13—C12	92.1(2)	C16—O4—C13—C14	− 90.5(2)
C10—C11—C25—O7	− 175.9(2)	C10—C11—C25—O8	3.0(2)
C11—C25—O8—C26	− 178.0(2)	O7—C25—O8—C26	0.9(2)
C25—O8—C26—C27	179.9(2)	O8—C26—C27—C28	− 106.2(2)
Nitro group			
C29—C30—N1—O9	− 168.1(2)	C29—C30—N1—O10	12.0(2)

4-phenylene bis(4-*n*-octyloxybenzoate)[7] the unsymmetric substitution in NEBB also gives rise to a noticeable difference in the twist of the central benzene ring II relative to the neighbouring carboxylic groups and benzene rings I and III, although to a lesser extent. Moreover, the mutual orientation of the two carboxylic groups attached in 1,4-positions to benzene ring II is a rather surprising one. The planes of the carboxylic groups are, as expected, nearly perpendicular to the ring plane but they are located on the same side of it and not on opposite sides as is more usual. This conformation, which can clearly be seen in Figure 1, contributes considerably to a transversal dipole moment in contrast to the approximately centrosymmetric conformation generally observed in phenylene 1,4-bis(benzoates).

The nitro group at the end of the lateral substituent is slightly twisted about the bond C30-N1 and inclined to the benzene ring by about 12°. All observed bond lengths and angles in NEBB (cf. Table III) are as expected and agree well with the appropriate standard values given by Allen *et al.*[16] Hence they do not warrant further comment.

3.2 Crystal packing

The crystal structure of NEBB is illustrated in Figures 3 and 4. It is characterized by a perfectly parallel alignment of the molecular long axes and an antiparallel orientation of neighbouring molecules generated by the three lattice translations and the inversion centres of the space group, respectively. A further striking feature of the crystal

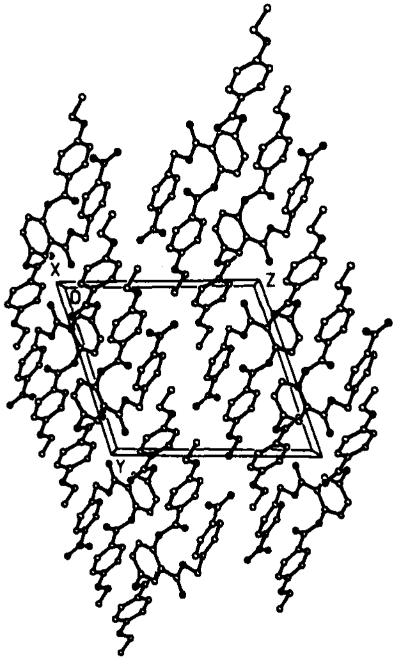


FIGURE 3 Crystal packing of NEBB viewed along the *a* axis.

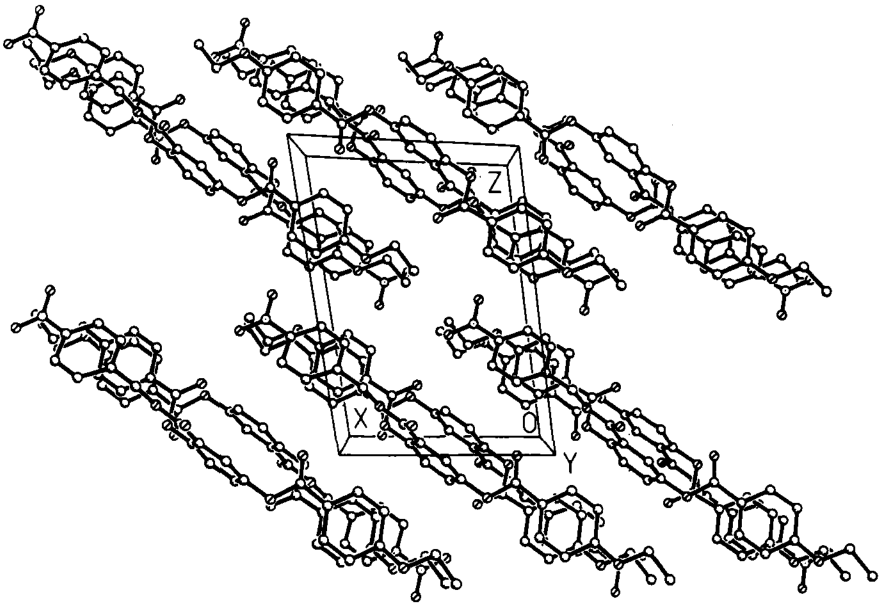


FIGURE 4 Crystal packing of NEBB viewed along the *b* axis.

structure is the formation of sheets and layers. The lamellar sheets oriented parallel to $(\bar{2}01)$ are built up by an antiparallel arrangement of molecules side by side. Two such sheets are shown from above in Figure 3 and from the side in Figure 4. Considering the molecular packing within the sheets, it should be mentioned that for the hexyl homologue of NEBB an extremely strong antiparallel correlation, i.e. the existence of pairs of molecules with an antiparallel orientation, was observed by dielectric measurements.[5] By stacking of the sheets in *a*-direction a layered arrangement (in the sense of smectic layers) is generated clearly recognizable from Figure 4. The molecular long axes are strongly inclined to the layer normal. The distance between neighbouring layers is decisively influenced by the oxygen atoms of the nitro group which protrude into the gaps between the layers.

The intermolecular contact distances in the crystal structure of NEBB indicate only normal van der Waals forces.

Acknowledgments

The authors thank the "Deutsche Forschungsgemeinschaft" and the "Fonds der chemischen Industrie" for support of this work.

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