Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl17

Crystal Structure and Conformational Behavior of Smectogenic 2-Phenyl-5-(4-n-pentoxyphenyl) Pyrimidine

G. Winter [a] , H. Hartung [a] , W. Brandt [a] & M. Jaskólski [b]

[a] Sektion Chemie, Martin-Luther-Universitat Halle-Wittenberg, Mühlpforte 1, DDR-4020, Halle, GDP

[b] Faculty of Chemistry, A. Mickiewicz University, ul. Grunwaldzka 6, 60-780, Poznań, Poland

Version of record first published: 13 Dec 2006.

To cite this article: G. Winter , H. Hartung , W. Brandt & M. Jaskólski (1987): Crystal Structure and Conformational Behavior of Smectogenic 2-Phenyl-5-(4-n-pentoxyphenyl) Pyrimidine, Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics, 150:1, 289-300

To link to this article: http://dx.doi.org/10.1080/00268948708074804

Mol. Cryst. Liq. Cryst., 1987, Vol. 150b, pp. 289–300
Photocopying permitted by license only

Printed in the United States of America

Crystal Structure and Conformational Behavior of Smectogenic 2-Phenyl-5-(4-*n*-pentoxyphenyl) Pyrimidine

G. WINTER, H. HARTUNG and W. BRANDT

Sektion Chemie, Martin-Luther-Universität Halle-Wittenberg, Mühlpforte 1, DDR-4020 Halle, GDR

and

M. JASKÓLSKI

Faculty of Chemistry, A. Mickiewicz University, ul. Grunwaldzka 6, 60-780 Poznań, Poland

(*Received February 23, 1987; in final form May 12, 1987*)

The crystal structure of the title compound with the formula C_6H_5—$C_4N_2H_2$—C_6H_4—OC_5H_{11} (2-PPePP) has been determined as part of a study of the conformational behavior of mesogenic phenyl pyrimidines. The compound crystallizes in space group $P\bar{1}$ with four molecules in a unit cell of dimensions a = 10.055(1), b = 9.621(2), c = 19.223(3) Å, α = 91.30(1), β = 103.90(2), γ = 97.60(2)°. The structure has been solved by direct methods and refined to R = 0.074.

The 2-PPePP molecules adopt a stretched form. The pentyloxy groups are partly disordered (molecule 1) and contain gauche bonds. The central pyrimidine ring is approximately coplanar with the phenyl rings attached to it in the 2- and 5-positions with interplanar angles of about 8° in both cases. This somewhat unexpected conformational behavior is compared with the results of stereochemical and theoretical considerations.

The 2-PPePP molecules are arranged in the crystalline state exactly parallel to each other forming sheets as well as a tilted layer structure.

Keywords: mesogenic phenyl pyrimidines, X-ray analysis, crystal structure, molecular structure, conformation

INTRODUCTION

This paper continues a series of structural investigations on the conformational behavior of phenyl pyrimidines carried out in our labo-

ratories. Recently, we have reported in this journal[1] on the structures of 5-phenyl-2-(4-*n*-propoxyphenyl)-pyrimidine (5-PPrPP) and 5-phenyl-2-(4-*n*-butoxyphenyl)-pyrimidine (5-PBuPP). In those homologous compounds with R_1 = alkoxy and R_2 = H (Fig. 1), the torsion angles representing the twists of the aromatic rings around the interring C—C bonds amount to about 5° for τ_1 and to almost 40° for τ_2. These values can be interpreted in terms of two competing effects: the conjugation of the aromatic ring systems favoring coplanar conformation, which is the dominating factor in τ_1, and the repulsion between the *ortho* H atoms supporting a markedly twisted conformation and determining τ_2 (Figure 1).

In this paper, we present the results of an X-ray analysis of the closely related compound 2-phenyl-5-(4-*n*-pentoxyphenyl)-pyrimidine (2-PPePP) with the alternative arrangement of substituents (R_1 = H, R_2 = alkoxy). 2-PPePP was first described by Zaschke and Schubert.[2] In contrast to the above-mentioned 5-phenyl-2-(4-*n*-alkoxyphenyl)-pyrimidines, it forms a smectic A phase in a considerably wide range of temperature (371 to 434 K).

EXPERIMENTAL

Data collection

Oscillation and Weissenberg photographs taken on colorless single crystals grown from an acetone/water solution indicated triclinic symmetry and gave preliminary lattice parameters. The crystal for diffractometer measurements (Syntex $P2_1$ diffractometer, MoKα radiation, graphite monochromator) was of approximate size 0.34 × 0.34 × 0.45 mm. Precise values of the lattice constants were obtained by a least-squares fit of the setting angles of 15 counter reflections. 4805

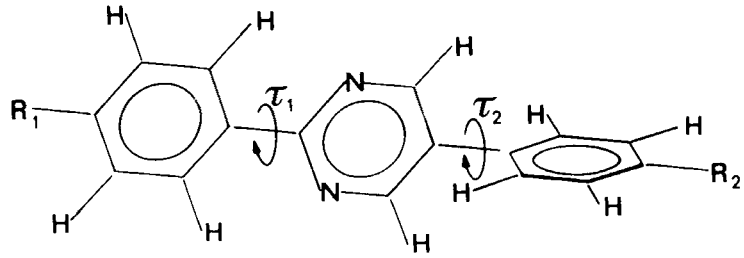

FIGURE 1 Definition of torsion angles describing the conformation in 2,5-diphenyl pyrimidines.

TABLE I

Crystal data

2-Phenyl-5-(4-*n*-pentoxyphenyl)-pyrimidine (2-PPePP)	
$C_{21}H_{22}N_2O$	M.W. = 318.4 g mol^{-1}
triclinic	Space group $P\bar{1}$
a = 10.055(1) Å	Z = 4
b = 9.621(2) Å	D_m = 1.22(1) g cm^{-3}
c = 19.223(3) Å	D_c = 1.18 g cm^{-3}
α = 91.30(1)°	μ(MoKα) = 0.4 cm^{-1}
β = 103.90(2)°	F(000) = 680
γ = 97.60(2)°	

intensity data ($2\theta \leq 60°$) were collected as previously described[3] but after data reduction, using the program PRARA,[4] only 1837 unique reflections with $|F| \geq 3.92\,\sigma(F)$ were used during structure refinement. No absorption or extinction corrections were applied. The E-value statistics strongly indicated the centrosymmetric space group $P\bar{1}$. It was confirmed by the successful structure determination. The crystal data of 2-PPePP are given in Table I.

Structure determination and refinement

The structure was solved by direct methods using the program MULTAN-80.[5] An E-map showed chemically reasonable fragments of the two symmetry-independent molecules (the rings and the O atoms). The remaining C atoms of the alkyl chains were located in two subsequent Fourier maps.

Full-matrix least-squares refinement of the non-hydrogen atoms with isotropic temperature factors (4 cycles) resulted in an R-value of 0.22. After anisotropic refinement (6 cycles, R = 0.13) the ring H atoms could be located in a difference Fourier map. Chain H atoms were generated geometrically (C—H = 1.08 Å, tetrahedral coordination of the C atoms). Subsequent four cycles of refinement with anisotropic non-H atoms and isotropic ring H atoms (the calculated H atoms were kept fixed) converged at R = 0.074. However, the values of bond distances and angles in the alkyl chain of molecule I (C(17) . . . C(21)) were unsatisfactory and the corresponding C atoms had anomalously high temperature factors. Therefore, a difference Fourier map with exclusion of those five C atoms was calculated. The peak heights and profiles in the region of the pentyl group of molecule I illustrated an increasing smearing and deformation of electron density with increasing number of the C atom. This phenomenon is typical of a kind of disorder analysed in detail for terephthalylidene-bis-(4-*n*-butylaniline) (TBBA) by Doucet et al.[6] and generally interpreted

as a 'partial melting' of the alkyl chain. We attempted to improve the results by including in the refinement several borderpositions for atoms C(20) and C(21) based on the profiles of their electron density distribution. However, all those attempts were without success. For this reason, the R of 0.074 was considered as final. Eight reflections with low indices and high intensity exhibiting large $\Delta F/\sigma(F)$ (with $|F_0| < |F_c|$) were apparently affected by extinction and therefore removed from the final calculations. Unit weights were used throughout the refinement.

All calculations were performed on the ESER 1040 computer of the Halle University using programs of the CRYPOZ library.[7]

Final positional and isotropic thermal parameters are presented in Table II.

RESULTS AND DISCUSSION

Molecular structure

An ORTEP drawing of the two independent molecules is given in Figure 2. As can be seen, both molecules adopt a widely stretched and nearly linear form.

The bond distances and angles are tabulated in Tables III and IV. With the exception of the alkyl chain, the corresponding values in both molecules are equal within 3σ limit and in good agreement with those reported in literature. The unusual distances and angles in the C(18) . . . C(21) group have to be attributed to the disorder of the terminal atoms of the alkyl chain discussed in the preceding section. A striking feature in both molecules is the presence of gauche conformations within the alkyl chains at the C(17)—C(18) and C(19)—C(20) bonds (molecule I) and the C(38)—C(39) bond (molecule II). A similar situation was observed in 5-PPrPP and 5-PBuPP[1] but the majority of crystalline mesogens have the alkyl chains in the all-trans conformation.

As pointed out in the introduction, of particular interest in the structure of 2-PPePP is the mutual orientation of the aromatic rings. It can be described either by the corresponding torsion angles (Table V) or by the dihedral angles between the pyrimidine and the phenyl rings:

	molecule I	molecule II
pyrimidine ring/benzene ring 1	7.8°	8.8°
pyrimidine ring/benzene ring 2	8.3°	6.7°
benzene ring 1/benzene ring 2	2.9°	3.1°

TABLE II

Final fractional coordinates ($\times 10^4$) and isotropic equivalents ($\times 10^3$) of the anisotropic thermal parameters for the non-hydrogen atoms (esd's in parentheses).
$U_{eq} = (U_{11} \cdot U_{22} \cdot U_{33})^{1/3}$

Molecule I					Molecule II				
Atom	*x/a*	*y/b*	*z/c*	U_{eq}	Atom	*x/a*	*y/b*	*z/c*	U_{eq}
C(1)	2772(10)	−79(8)	6659(5)	69(4)	C(22)	7145(11)	5131(12)	13314(6)	99(5)
C(2)	4118(10)	484(10)	6674(5)	74(4)	C(23)	6908(11)	6005(10)	12776(6)	82(5)
C(3)	5029(9)	916(8)	7312(5)	65(4)	C(24)	6021(8)	5491(9)	12115(5)	61(4)
C(4)	4629(9)	861(8)	7957(5)	57(4)	C(25)	5376(9)	4092(9)	12022(4)	63(4)
C(5)	3279(10)	328(9)	7936(5)	68(7)	C(26)	5666(9)	3220(9)	12584(5)	70(4)
C(6)	2358(9)	−149(9)	7297(6)	70(4)	C(27)	6571(10)	3751(11)	13228(5)	81(5)
C(7)	5654(10)	1377(10)	8637(5)	63(4)	C(28)	4448(8)	3558(8)	11331(5)	56(4)
C(8)	7733(10)	2546(10)	9244(5)	74(4)	C(29)	3484(9)	3838(9)	10162(5)	71(4)
C(9)	7419(7)	2373(7)	9890(5)	45(3)	C(30)	2632(8)	2540(8)	10044(4)	49(3)
C(10)	6105(10)	1632(9)	9840(5)	74(4)	C(31)	2919(8)	1825(8)	10671(5)	60(4)
C(11)	8356(8)	2968(7)	10583(4)	51(3)	C(32)	1646(8)	2012(8)	9366(4)	56(3)
C(12)	9719(9)	3520(8)	10621(4)	57(3)	C(33)	1357(9)	2861(8)	8793(5)	63(4)
C(13)	10631(8)	4031(8)	11268(5)	60(3)	C(34)	424(8)	2374(9)	8151(5)	66(4)
C(14)	10208(9)	4052(8)	11903(5)	59(3)	C(35)	−100(8)	993(10)	8102(5)	63(4)
C(15)	8860(8)	3478(8)	11877(4)	61(3)	C(36)	16(8)	99(9)	8652(5)	60(3)
C(16)	7948(9)	2918(8)	11238(4)	61(3)	C(37)	951(9)	641(8)	9275(5)	65(4)
C(17)	12415(9)	5184(10)	12599(5)	74(4)	C(38)	−1848(9)	−845(9)	7350(5)	79(4)
C(18)	13088(11)	5543(13)	13394(6)	111(6)	C(39)	−2516(9)	−1141(9)	6574(5)	72(4)
C(19)	12438(15)	6493(14)	13731(7)	131(7)	C(40)	−1499(12)	−1328(12)	6119(6)	105(5)
C(20)	13038(35)	6673(20)	14555(12)	273(16)	C(41)	−2062(16)	−1755(18)	5371(9)	184(12)
C(21)	13623(33)	7623(20)	14789(14)	334(32)	C(42)	−1014(17)	−2071(15)	4970(7)	179(9)
N(1)	6876(8)	2038(8)	8610(4)	72(3)	N(3)	4351(7)	4374(7)	10769(4)	69(3)
N(2)	5213(7)	1152(8)	9233(4)	68(3)	N(4)	3747(8)	2267(7)	11307(4)	68(3)
O(1)	11033(6)	4511(6)	12562(3)	68(2)	O(2)	−1093(6)	552(5)	7435(5)	65(2)

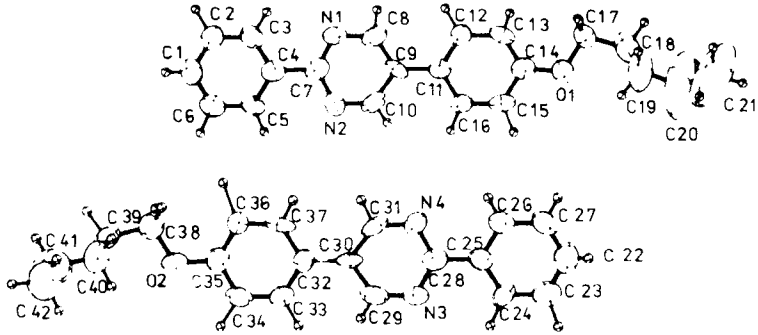

FIGURE 2 Molecular structure with atom numbering for the two independent 2-PPePP molecules.

TABLE III

Bond distances for the non-hydrogen atoms (esd's in parentheses)

Molecule I		Molecule II	
Atoms	Distance (Å)	Atoms	Distance (Å)
Benzene rings			
Benzene ring 1 (C(1) . . . C(6))		Benzene ring 1 (C(22) . . . C(26))	
C—C (average)	1.38(1)	C—C (average)	1.38(1)
Benzene ring 2 (C(11) . . . C(16))		Benzene ring 2 (C(32) . . . C(36))	
C—C (average)	1.39(1)	C—C (average)	1.38(1)
C(4)—C(7)	1.49(1)	C(25)—C(28)	1.46(1)
Pyrimidine ring			
C(7)—N(1)	1.32(1)	C(28)—N(3)	1.34(1)
N(1)—C(8)	1.35(1)	N(3)—C(29)	1.32(1)
C(8)—C(9)	1.36(1)	C(29)—C(30)	1.40(1)
C(9)—C(10)	1.40(1)	C(30)—C(31)	1.39(1)
C(10)—N(2)	1.32(1)	C(31)—N(4)	1.33(1)
N(2)—C(7)	1.34(1)	N(4)—C(28)	1.34(1)
C(9)—C(11)	1.49(1)	C(30)—C(32)	1.47(1)
Pentyloxy group			
C(14)—O(1)	1.36(1)	C(35)—O(2)	1.40(2)
O(1)—C(17)	1.44(1)	O(2)—C(38)	1.44(1)
C(17)—C(18)	1.52(2)	C(38)—C(39)	1.49(1)
C(18)—C(19)	1.42(2)*	C(39)—C(40)	1.52(2)
C(19)—C(20)	1.55(3)*	C(40)—C(41)	1.44(2)
C(20)—C(21)	1.05(4)*	C(41)—C(42)	1.50(2)

*Values influenced by the disorder of the pentyl group in molecule I (see text).

TABLE IV

Bond angles for the non-hydrogen atoms (esd's in parentheses)

Molecule I		Molecule II	
Atoms	Angle (°)	Atoms	Angle (°)
Benzene rings			
Benzene ring 1 (C(1) . . . C(6))		Benzene ring 1 (C(22) . . . C(26))	
C—C—C (average)	120.2(1.3)	C—C—C (average)	120.1(1.2)
C(3)—C(4)—C(7)	119.6(7)	C(24)—C(25)—C(28)	120.3(7)
C(5)—C(4)—C(7)	122.5(7)	C(26)—C(25)—C(28)	120.8(7)
Benzene ring 2 (C(11) . . . C(16))		Benzene ring 2 (C(32) . . . C(36))	
C—C—C (average)	120.0(2.1)	C—C—C (average)	120.0(3.3)
C(9)—C(11)—C(12)	120.6(6)	C(30)—C(32)—C(33)	121.3(7)
C(9)—C(11)—C(16)	122.6(7)	C(30)—C(32)—C(37)	121.4(7)
C(13)—C(14)—O(1)	125.5(7)	C(34)—C(35)—O(2)	114.6(7)
C(15)—C(14)—O(1)	116.9(7)	C(36)—C(35)—O(2)	121.2(7)
Pyrimidine ring			
C(4)—C(7)—N(1)	119.2(7)	C(25)—C(28)—N(3)	118.9(7)
C(4)—C(7)—N(2)	115.0(7)	C(25)—C(28)—N(4)	116.8(7)
C(7)—N(1)—C(8)	116.3(8)	C(28)—N(3)—C(29)	116.1(7)
N(1)—C(8)—C(9)	123.9(8)	N(3)—C(29)—C(30)	127.6(7)
C(8)—C(9)—C(10)	113.7(7)	C(29)—C(30)—C(31)	108.1(7)
C(9)—C(10)—N(2)	124.7(7)	C(30)—C(31)—N(4)	129.0(7)
C(10)—N(2)—C(7)	115.6(8)	C(31)—N(4)—C(28)	114.8(7)
N(2)—C(7)—N(1)	125.8(8)	N(4)—C(28)—N(3)	124.3(7)
C(8)—C(9)—C(11)	123.4(7)	C(29)—C(30)—C(32)	125.6(7)
C(10)—C(9)—C(11)	122.8(7)	C(31)—C(30)—C(32)	126.3(7)
Pentyloxy group			
C(14)—O(1)—C(17)	118.0(6)	C(35)—O(2)—C(38)	118.2(6)
O(1)—C(17)—C(18)	106.2(7)	O(2)—C(38)—C(39)	107.5(7)
C(17)—C(18)—C(19)	114.8(9)*	C(38)—C(39)—C(40)	113.6(8)
C(18)—C(19)—C(20)	112.8(13)*	C(39)—C(40)—C(41)	117.6(10)
C(19)—C(20)—C(21)	120.1(23)*	C(40)—C(41)—C(42)	114.8(12)

*Values influenced by the disorder of the pentyl group in molecule I (see text).

The values for the 2-phenyl pyrimidine fragment agree well with those observed in 5-PPrPP (4.8°) and 5-PBuPP (7.6°)[1] as well as with the expectations based on stereochemical considerations. Quite surprisingly, the rings of the 5-phenyl pyrimidine fragment are also nearly coplanar in contrast to the situation in 5-PPrPP and 5-PBuPP where they are considerably twisted (39.1 and 37.7°, respectively). That twist could be easily explained by the repulsions between the H atoms in *ortho* position to the C—C interring bond. Coplanarity analogous to that in 2-PPePP was found in some biphenyl derivatives, e.g. in the mesogenic 4-*n*-pentylcyclohexyl-4′-cyanobiphenyl (2.4°)[8] and 4-*n*-pentyloxy-4′-cyanobiphenyl (0.8°),[9] and explained by crystal packing forces. In the present case of 2-PPePP, however, an additional ar-

TABLE V

Selected torsion angles (°)

Molecule I		Molecule II	
C(3)—C(4)—C(7)—N(1)	8.3(10)	C(24)—C(25)—C(28)—N(3)	7.7(9)
C(3)—C(4)—C(7)—N(2)	−173.6(8)	C(24)—C(25)—C(28)—N(4)	−172.4(9)
C(5)—C(4)—C(7)—N(1)	−171.4(9)	C(26)—C(25)—C(28)—N(3)	−170.3(9)
C(5)—C(4)—C(7)—N(2)	6.7(9)	C(26)—C(25)—C(28)—N(4)	9.6(10)
C(8)—C(9)—C(11)—C(12)	−12.0(9)·	C(29)—C(30)—C(32)—C(33)	−8.1(10)
C(8)—C(9)—C(11)—C(16)	172.4(7)	C(29)—C(30)—C(32)—C(37)	172.8(10)
C(10)—C(9)—C(11)—C(12)	170.8(8)	C(31)—C(30)—C(32)—C(33)	174.5(9)
C(10)—C(9)—C(11)—C(16)	−4.7(9)	C(31)—C(30)—C(32)—C(37)	−4.7(10)
C(13)—C(14)—O(1)—C(17)	7.7(9)	C(36)—C(35)—O(2)—C(38)	3.5(9)
C(15)—C(14)—O(1)—C(17)	−176.6(7)	C(34)—C(35)—O(2)—C(38)	−176.4(8)
C(14)—O(1)—C(17)—C(18)	−178.6(8)	C(35)—O(2)—C(38)—C(39)	−170.2(8)
O(1)—C(17)—C(18)—C(19)	−62.0(11)*	O(2)—C(38)—C(39)—C(40)	70.5(9)
C(17)—C(18)—C(19)—C(20)	172.5(14)*	C(38)—C(39)—C(40)—C(41)	174.0(11)
C(18)—C(19)—C(20)—C(21)	110.6(29)*	C(39)—C(40)—C(41)—C(42)	−173.7(13)

*Values influenced by the disorder of the pentyl group in molecule I (see text).

gument in favor of coplanarity can be taken into consideration. It is discussed below.

Crystal structure

The packing of the 2-PPePP molecules in the crystal is illustrated in Figures 3 and 4 in two different projections. It is characterized by a perfectly parallel arrangement of the molecular long axes with an antiparallel alignment of neighboring independent molecules I and II. A further striking feature of the crystal structure is the formation of sheets and layers. One sheet is built up by translation of the molecular pair (I and II) in the directions [100] and [011]. The three-dimensional structure is generated by stacking the sheets one upon another with a sheet-to-sheet distance of 4.6 Å. Neighboring sheets are related by inversion centers. From Figures 3 and 4 also a discrete layered arrangement (in the sense of smectic layers) can be recognized. The layer planes are parallel to (001) and the molecular long axes are inclined to them by a tilt angle of about 63°. Referring to the formation of a smectic A phase by 2-PPePP on heating it can be concluded, that the melting process of this substance is connected with an increase of the tilt up to the perpendicular orientation of the molecular long axes.

All intermolecular contacts between non-H atoms are greater than the sums of the corresponding van der Waals radii.

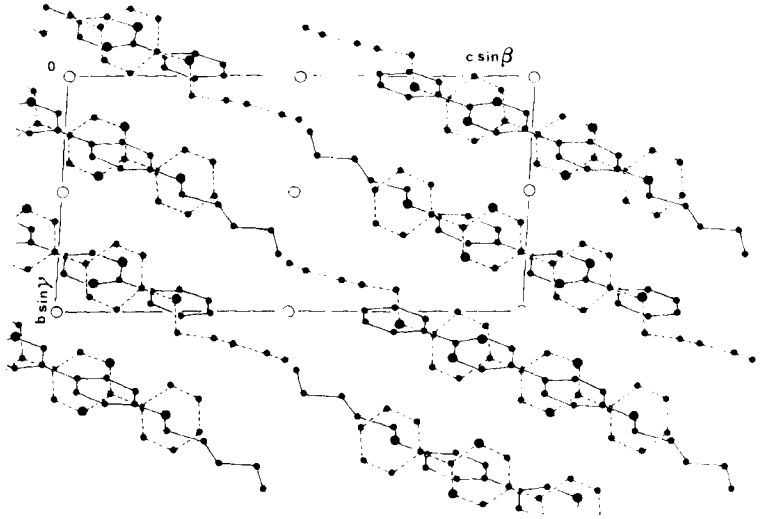

FIGURE 3 Crystal structure of 2-PPePP projected along [100] (—— molecule I, ---- Molecule II).

Theoretical considerations concerning the conformational behavior of 2-PPePP

The unexpectedly low value of the torsion angle τ_2 (Figure 1) in 2-PPePP initiated some theoretical consideration of this problem.

Comparison of the π-electron systems of the two series of isomeric phenyl-alkoxyphenyl pyrimidines shows a significant difference with respect to the C—C interring bonds. For 5-phenyl-2-alkoxyphenyl-pyrimidines (*1*) only three essential resonance structures with a double bond corresponding to the torsion angle τ_2 can be drawn whereas for 2-phenyl-5-alkoxyphenyl-pyrimidines (*2*) there are nine such structures (Figure 5). It should be noted that the three upper resonance structures of b) represent in each case two equivalent but different structures. Therefore, they have to be counted doubly). On the assumption that the twist of the rings is influenced by the degree of double-bond character of the interring bond, the above-mentioned difference may contribute to the different values of the torsion angles τ_2 in *1* and *2*. Also simple HMO calculations[10,11]($\beta = -2{,}5$ eV) yielded bond orders of 0.3741 (for *1*) and 0.3760 (for *2*) for the discussed interring bond. The difference is very small but consistent with the schemes in Figure 5. For a twist of 90° the π-electron energy

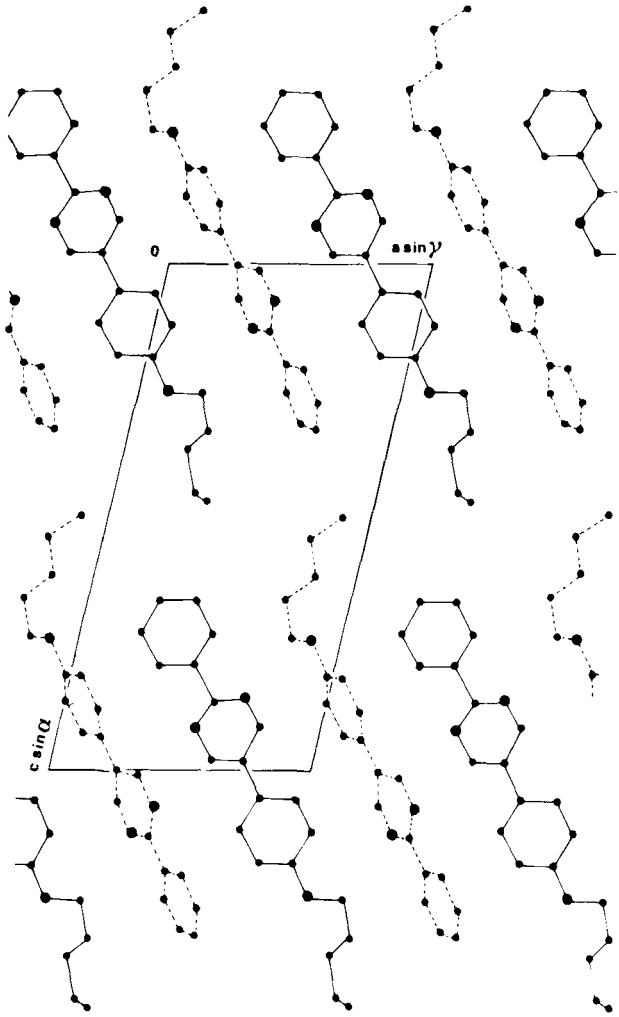

FIGURE 4 One sheet of molecules in the crystal structure of 2-PPePP projected along [010] (—— molecule I, ---- molecule II).

loss for *2* has been calculated to be 0.86 kJ/mol larger than for *1*, i.e. the rotation barrier should be higher by this value for *2*. The question is whether these small differences can be used as an argument (besides crystal packing forces) for the different conformations. A positive answer is supported by the analogous example of biphenyl. Taking into account its very low torsional energy barrier (about 10 kJ/mol

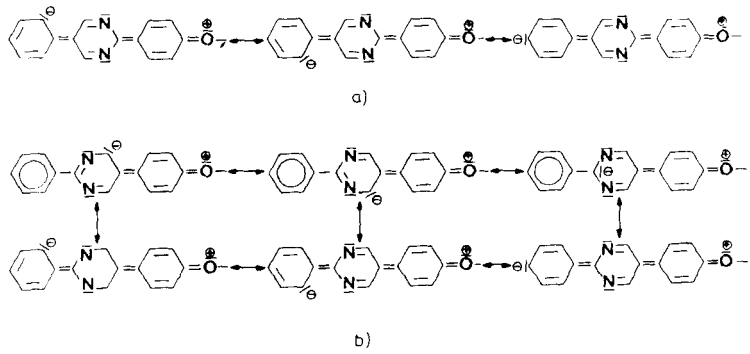

FIGURE 5 Possible resonance structures with a double bond between the pyrimidine ring and the 5-substituted phenyl ring: a) for 5-phenyl-2-(alkoxyphenyl)-pyrimidines; b) for 2-phenyl-5-(alkoxyphenyl)-pyrimidines.

or less, the values given in the literature range considerably[12]) it seems possible that a small increase of the barrier height can shift the torsion angle of minimum energy to lower values. For cyanobiphenyl we calculated a higher bond order for the interring bond (0.3716) than for unsubstituted biphenyl (0.3696) and the rotational barrier should be higher by 0.48 kJ/mol. Therefore, the above-mentioned nearly coplanar conformation observed in some cyanobiphenyls[8,9]can be due not only to crystal packing forces but also to the higher degree of the double-bond character of the interring bond. However, it must be noted that the strongly twisted conformation (torsion angles about 40°) observed in 4-bromo-4′-cyanobiphenyl,[13] 4-*n*-butyl-4′-cyanobiphenyl,[14] and 4-*n*-propyl-4′-cyanobiphenyl[15] is in contradiction to this hypothesis.

References

1. G. Winter, H. Hartung, M. Jaskólski, *Mol. Cryst. Liq. Cryst.*, (1987).
2. H. Zaschke, H. Schubert, *J. prakt. Chem.*, **315**, 1113 (1973).
3. U. Baumeister, H. Hartung, M. Gdaniec, M. Jaskólski, *Mol. Cryst. Liq. Cryst.*, **69**, 119 (1981).
4. M. Jaskólski, PRARA. Syntex-tape reading program, Univ. of Poznań, Poland (1980).
5. P. Main, S. J. Fiske, S. E. Hull, L. Lessinger, G. Germain, J. P. Declercq, M. M. Woolfson, MULTAN-80. A system of computer programs for the automatic solution of crystal structures from X-ray diffraction data. Univ. of York, England, and Louvain, Belgium (1980).

6. J. Doucet, J.-P. Mornon, R. Chevalier, A. Lifchitz, *Acta Cryst.*, **B33**, 1701 (1977).
7. M. Jaskólski, 4th Symposium on Organic Crystal Chemistry, Poznań, Poland (1982). Collected Abstracts, p. 70.
8. W. Haase, H. Paulus, H. T. Müller, *Mol. Cryst. Liq. Cryst.*, **97**, 131 (1983).
9. P. Mandal, S. Paul, *Mol. Cryst. Liq. Cryst.*, **131**, 223 (1985).
10. B. and A. Pullman, Results of Quantum Mechanical Calculations of the Electronic Structure of Biochemicals, Vol. I, p. VI, Paris (1960).
11. O. E. Polansky, M. Grassberger, Mh. Chem. **94**, 647 (1963).
12. G. Häfelinger, C. Regelmann, *J. Comput. Chem.*, **6**, 368 (1985).
13. P. Kronebusch, W. B. Gleason, D. Britton, *Cryst. Struct. Comm.*, **5**, 17 (1976).
14. G. V. Vani, *Mol. Cryst. Liq. Cryst.*, **99**, 21 (1983).
15. W. Haase, H. Paulus, R. Pendzialek, *Mol. Cryst. Liq. Cryst.*, **100**, 211 (1983).