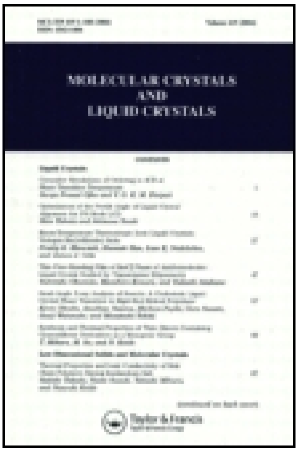

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Crystal and Molecular Structures of Two Isostructural 2,5-Diphenyl Pyrimidines

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Crystal and Molecular Structures of Two Isostructural 2,5-Diphenyl Pyrimidines

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The crystal structures of 5-phenyl-2-(4-*n*-propoxyphenyl)-pyrimidine (5-PPrPP) and 5-phenyl-2-(4-*n*-butoxyphenyl)-pyrimidine (5-PBuPP) have been determined as part of a study of the conformational behavior of mesogenic phenyl pyrimidines. The two compounds are isostructural and crystallize in space group $P2_1/c$ with four molecules per unit cell. The lattice dimensions are (5-PPrPP): a = 12.961(2), b = 15.186(1), c = 7.882(1) Å, β = 98.47(1)° and (5-PBuPP): a = 12.116(1), b = 15.172(1), c = 9.098(1) Å, β = 96.581(9)°. The structures have been solved by direct methods and refined to R values of 0.037 and 0.066, respectively.

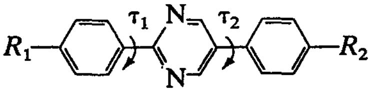
The molecules of both compounds adopt a stretched form. Their phenyl rings are twisted with respect to the central pyrimidine ring in the following extent: the interplanar angles amount to 4.8° (5-PPrPP) and 7.6° (5-PBuPP) for the 2-phenyl pyrimidine and to 39.1° and 37.7°, respectively, for the 5-phenyl pyrimidine fragment. The molecules are arranged in the crystalline state nearly parallel to each other forming sheets parallel to the crystallographic *ac* plane.

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

INTRODUCTION

In analogy to the well-known mesogenic biphenyl derivatives, substituted phenyl pyrimidines found practical applications as components in liquid crystalline materials. The twist of the two aromatic rings influences their thermal phase behavior and is accordingly the subject of special interest.[1,2] It depends markedly on the position of the phenyl group at the pyrimidine group: previous X-ray studies[1,2] suggested that the corresponding torsion angle amounts to less than 10° for 2-phenyl pyrimidines and to more than 20° for 5-phenyl pyrimidines.

In 2,5-diphenyl pyrimidines[3]



the torsion angles τ_1 and τ_2 can be expected to have distinctly different values. In order to verify this hypothesis solid state conformational analysis has been performed for two compounds with R_1 = OC_3H_7 (5-phenyl-2-(4-*n*-propoxyphenyl)-pyrimidine, 5-PPrPP) and R_1 = OC_4H_9 (5-phenyl-2-(4-*n*-butoxyphenyl)-pyrimidine, 5-PBuPP) and R_2 = H in both cases. Only 5-PBuPP forms a monotropic nematic mesophase with the melting point of 402 K and the clearing point at 390 K whereas 5-PPrPP melts isotropically at 424 K and does not form a mesophase also on cooling of the isotropic melt. The results of an X-ray analysis of the smectogenic 2-phenyl-5-(4-*n*-pentoxyphenyl)-pyrimidine (2-PPePP) with R_1 = H and R_2 = OC_5H_{11}, as a representative of the alternative ring sequence, will be discussed in a separate paper.[4]

EXPERIMENTAL

Colorless single crystals of 5-PPrPP and 5-PBuPP suitable for X-ray investigations were obtained by recrystallization from an acetone/water mixture. Space group and preliminary unit-cell dimensions were determined from oscillation and Weissenberg photographs. The crystals belong to the monoclinic system and the systematic absences unequivocally indicated the space group $P2_1/c$. All other X-ray measurements were performed on a Syntex $P2_1$ diffractometer using graphite monochromated CuKα radiation. Accurate cell parameters were determined by a least squares fit of the setting angles of 15 counter reflections. The intensity data were collected in a θ-2θ scan mode and corrected for Lorentz and polarization effects, but no absorption correction was made. The relevant crystal data and experimental details are given in Table I.

TABLE I

Summary of crystal data and experimental details

	5-PPrPP	5-PBuPP
Molecular formula	$C_{19}H_{18}N_2O$	$C_{20}H_{20}N_2O$
Molecular weight (g mol^{-1})	290.4	304.4
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$P2_1/c$
a (Å)	12.961(2)	12.116(1)
b (Å)	15.186(1)	15.172(1)
c (Å)	7.882(1)	9.098(1)
β(°)	98.47(1)	96.581(9)
V (Å^3)	1535.7(3)	1661.4(3)
D_c (g cm^{-3})	1.26	1.22
D_m (g cm^{-3})	1.23(1)	1.25(1)
Z	4	4
F(000)	616	648
μ(CuKα) (cm^{-1})	5.4	5.2
Crystal size (mm)	0.17 × 0.24 × 0.38	0.12 × 0.32 × 0.38
Number of indep. reflections	2092	2216
Number of observed reflections ($\|F\| \geq 3.92\ \sigma(F)$)	1600	1684

STRUCTURE DETERMINATION AND REFINEMENT

A. 5-Phenyl-2-(4-*n*-propoxyphenyl)-pyrimidine

The structure resisted solution by the program MULTAN-80.[5] Also all attempts to solve the structure by means of the centrosymmetric direct methods program EEES included in the SHELX-76 system[6] failed. SHELX, however, yielded some structural fragments suitable as sufficient input model that was expanded by the program DIRDIF.[7] The first run of DIRDIF revealed the positions of all non-hydrogen atoms ($R = 0.29$). Full-matrix least-squares refinement of the structure proceeded smoothly. After 4 cycles with isotropic thermal parameters ($R = 0.23$) and 6 cycles of anisotropic refinement ($R = 0.095$) all hydrogen atoms were located in a difference electron density map, and their positional and isotropic thermal parameters were included in the refinement as variables. Seven strong reflections with large $||F_o| - |F_c||/\sigma(F)$ were excluded from the final refinement. The final R is 0.037. In the final cycle the shifts in the parameters were less than 0.2σ and the difference Fourier map showed max. and min. heights of 0.114 and -0.106 e.Å^{-3}, respectively. Unit weights were used throughout the refinement.

All calculations were performed on an ESER 1040 computer of the Halle University using the above-mentioned and other programs of the CRYPOZ library.[8]

B. 5-Phenyl-2-(4-*n*-butoxyphenyl)-pyrimidine

The structure was solved by application of DIRDIF and refined in the same way as described above for 5-PPrPP, except for some complications caused by structural disorder of atoms C(18) and C(19). The disorder was manifested by unrealistically high temperature factors of those atoms and an extremely short distance between them. In addition, the positional parameters of the corresponding H atoms located on a difference Fourier map did not meet the expectation. Therefore, the coordinates of the H atoms at C(18) and C(19) were placed at calculated positions (C—H = 1.08 Å, tetrahedral coordination of the C atoms) and were kept fixed in the final refinement. Moreover, nine strong reflections with large $\Delta F/\sigma(F)$ (= reflections with low indices and high intensities affected by extinction) were excluded from the refinement. This reduced R to 0.066 which was accepted as final. The final difference Fourier map contains two maxima of 0.344 and 0.276 e.Å^{-3} located between C(18) and C(19) which can be attributed to the above-mentioned disorder.

RESULTS AND DISCUSSION

Molecular structure

Atomic parameters, together with their standard deviations, are given in Table II (5-PPrPP) and Table III (5-PBuPP).

The molecular structures and atomic numbering schemes are shown in Figures 1 and 2. The bond lengths and angles are summarized in Table IV.

According to our expectation, the molecular structure of the homologous compounds 5-PPrPP and 5-PBuPP are in nearly complete agreement. This can be seen from Table IV: except for the terminal atoms of the alkoxy groups which are disordered in 5-PBuPP there are no significant differences between the corresponding values of bond lengths and angles in both structures. Also the individual values for the bonding parameters are quite normal and agree well with standard values, so that their discussion can be restricted to the following aspects.

Both the N—C—N and C—C—C angles in the pyrimidine rings of 5-PPrPP and 5-PBuPP are significantly smaller than those found in unsubstituted pyrimidine[9] (128.2(5) and 116.3(5)°, respectively). The

TABLE II

Atomic parameters of 5-PPrPP

a) Final fractional coordinates (1 [illegible] sotropic equivalents ($\times 10^3$) of the anisotropic thermal [illegible] s for the no[illegible]n atoms (esd's in parentheses).

$$U_{eq} = (U_{11} \cdot U_{22} \cdot U_{33})^{1/3}$$

Atom	x/a	y/b	z/c	U_{eq}	Atom	x/a	y/b	z/c	U_{eq}
C(1)	545(2)	4093(2)	−3643(3)	73(1)	C(12)	6265(1)	4235(1)	4945(3)	49(1)
C(2)	1553(2)	3974(2)	−3945(3)	73(1)	C(13)	6990(2)	4223(1)	6401(3)	51(1)
C(3)	2367(2)	3909(1)	−2599(3)	62(1)	C(14)	6788(1)	3768(1)	7857(2)	48(1)
C(4)	2180(1)	3961(1)	−909(2)	50(1)	C(15)	5852(2)	3319(1)	7798(3)	53(1)
C(5)	1154(2)	4090(2)	−632(3)	61(1)	C(16)	5139(2)	3326(1)	6316(3)	51(1)
C(6)	348(2)	4154(2)	−1982(3)	71(1)	C(17)	7316(2)	3402(2)	10812(3)	61(1)
C(7)	3022(1)	3885(1)	547(2)	48(1)	C(18)	8254(2)	3509(2)	12159(3)	67(1)
C(8)	4004(2)	4256(1)	575(3)	53(1)	C(19)	9156(2)	2951(3)	11818(4)	87(1)
C(9)	4530(1)	3799(1)	3321(2)	46(1)	N(1)	4761(1)	4219(1)	1925(2)	51(1)
C(10)	2895(2)	3434(1)	2035(3)	54(1)	N(2)	3621(1)	3387(1)	3412(2)	55(1)
C(11)	5321(1)	3785(1)	4864(2)	45(1)	O	7548(1)	3799(1)	9244(1)	57(1)

b) Final fractional coordinates ($\times 10^3$) and isotropic temperature factors ($\times 10^3$) for the hydrogen atoms (esd's in parentheses). Atoms are numbered according to the carbon atoms to which they are attached.

Atom	x/a	y/b	z/c	U_{iso}	Atom	x/a	y/b	z/c	U_{iso}
H(1)	−6(2)	410(1)	−464(1)	84(7)	H(15)	570(1)	302(1)	878(1)	59(6)
H(2)	167(2)	392(2)	−512(2)	103(9)	H(16)	448(1)	301(1)	624(1)	52(5)
H(3)	307(2)	380(1)	−282(1)	73(7)	H(17a)	668(2)	374(2)	1118(2)	87(8)
H(5)	105(2)	413(1)	56(1)	74(7)	H(17b)	716(2)	275(1)	1053(1)	67(7)
H(6)	−39(2)	424(2)	−176(2)	85(8)	H(18a)	811(2)	332(2)	1338(2)	103(9)
H(8)	417(2)	459(1)	−43(1)	66(6)	H(18b)	849(2)	412(2)	1231(2)	125(11)
H(10)	213(1)	310(1)	207(1)	62(6)	H(19a)	900(3)	227(3)	1167(3)	225(21)
H(12)	640(1)	455(1)	395(1)	52(6)	H(19b)	975(2)	309(2)	1272(2)	126(11)
H(13)	765(2)	455(1)	647(1)	66(6)	H(19c)	934(2)	318(2)	1070(2)	137(12)

TABLE III

Atomic parameters of 5-PBuPP

a) Final fractional coordinates (×10^4) and isotropic equivalents (×10^3) of the anisotropic thermal parameters for the non-hydrogen atoms (esd's in parentheses).

$$U_{eq} = (U_{11} \cdot U_{22} \cdot U_{33})^{1/3}$$

Atom	x/a	y/b	z/c	U_{eq}	Atom	x/a	y/b	z/c	U_{eq}
C(1)	498(6)	4021(4)	−3733(6)	94(2)	C(13)	6657(3)	4240(3)	5560(5)	64(2)
C(2)	1600(6)	3899(4)	−3815(5)	85(2)	C(14)	6358(3)	3760(2)	6770(4)	59(1)
C(3)	2348(4)	3856(3)	−2549(5)	74(2)	C(15)	5380(3)	3288(3)	6618(4)	59(1)
C(4)	1980(3)	3935(2)	−1160(4)	57(1)	C(16)	4703(3)	3306(2)	5304(4)	57(1)
C(5)	851(4)	4063(3)	−1093(5)	74(2)	C(17)	6762(4)	3365(3)	9341(5)	83(2)
C(6)	104(4)	4103(4)	−2365(6)	87(2)	C(18)	7621(8)	3584(4)	10615(9)	137(4)
C(7)	2763(3)	3895(2)	205(4)	53(1)	C(19)	8349(9)	3082(5)	11082(8)	171(5)
C(8)	3813(3)	4256(3)	319(5)	62(2)	C(20)	9188(8)	3315(6)	12482(8)	121(3)
C(9)	4199(3)	3830(2)	2716(4)	52(1)	N(1)	4536(2)	4234(2)	1538(3)	59(1)
C(10)	2509(3)	3486(3)	1487(5)	65(2)	N(2)	3200(3)	3451(2)	2734(3)	64(1)
C(11)	4956(3)	3792(2)	4091(4)	52(1)	O	7090(2)	3793(2)	8037(3)	76(1)
C(12)	5958(3)	4259(2)	4256(4)	59(1)					

b) Final fractional coordinates (×10^3) and isotropic temperature factors (×10^3) for the hydrogen atoms (esd's in parentheses). Atoms are numbered according to the carbon atoms to which they are attached.

Atom	x/a	y/b	z/c	U_{iso}	Atom	x/a	y/b	z/c	U_{iso}
H(1)	−7(4)	404(3)	−456(5)	116(18)	H(16)	399(3)	299(2)	522(4)	58(10)
H(2)	190(4)	382(3)	−468(5)	98(16)	H(17a)	596(4)	360(3)	941(5)	87(10)
H(3)	313(4)	371(3)	−256(5)	100(16)	H(17b)	669(3)	367(3)	906(5)	113(23)
H(5)	61(4)	415(3)	−17(5)	84(19)	H(18a)	715	349	1154	138
H(6)	−71(4)	419(3)	−225(5)	106(17)	H(18b)	787	427	1057	138
H(8)	406(3)	454(2)	−46(4)	67(11)	H(19a)	811	240	1113	171
H(10)	177(3)	320(2)	141(4)	82(12)	H(19b)	923	315	1101	171
H(12)	618(3)	459(2)	342(4)	61(11)	H(20a)	865(5)	341(4)	1331(6)	144(26)
H(13)	737(3)	458(3)	574(4)	79(13)	H(20b)	936(5)	397(4)	1260(7)	148(26)
H(15)	521(3)	294(2)	746(4)	68(11)	H(20c)	977(6)	302(5)	1284(8)	255(37)

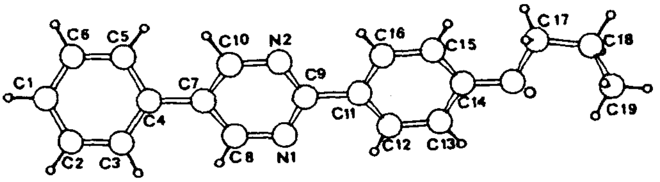


FIGURE 1 Molecular structure of 5-PPrPP with atom-numbering scheme.

bond lengths between the N atoms and C(9) are, compared with their values in pyrimidine, a little bit elongated. These tendencies are also found in other phenyl pyrimidines.[1,2]

The strange values for the bond lengths and angles within the alkyl chain of 5-PBuPP as well as the unrealistically high temperature factors observed for C(18) and C(19) (cf. the ORTEP plot, Figure 2) are caused by a structural disorder of those atoms. This effect has been observed in several other cases and interpreted as a partial melting of the alkyl chain. Surprisingly, the propyloxy and butyloxy chains are not fully staggered but include a gauche conformation (C(17)—C(18); cf. Table V). For the experimentally located H atoms, the C—H distances range from 0.86 to 1.12 Å ($\overline{\text{C—H}}$ = 1.00(4) Å for 5-PPrPP and 0.98(6) Å for 5-PBuPP).

Of particular interest in both diphenyl pyrimidine molecules is the orientation of the central pyrimidine ring with respect to its neighboring benzene rings. The mutual twist of the three planar aromatic rings can be described either by the corresponding torsion angles (vid. Table V) or by the interplanar angles (i.e. the angles between normals to the least-squares planes). The following interplanar angles with respect to the pyrimidine ring have been observed:

	5-PPrPP	5-PBuPP
benzene ring 1 (C(1) . . . C(6))	39.1°	37.7°
benzene ring 2 (C(11) . . . C(16))	4.8°	7.6°

These values agree well with other experimental results as well as with the expectations based on stereochemical considerations and

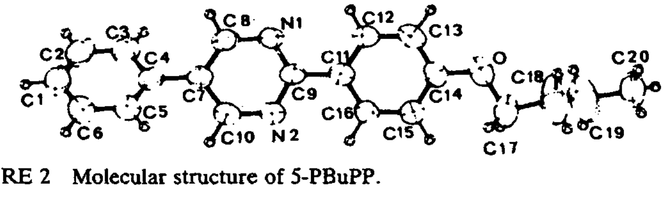


FIGURE 2 Molecular structure of 5-PBuPP.

TABLE IV

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

	Distance (Å)			Angle (°)	
Atoms	5-PPrPP	5-PBuPP	Atoms	5-PPrPP	5-PBuPP
			Benzene ring 1		
C(1)—C(2)	1.374(4)	1.358(10)	C(6)—C(1)—C(2)	119.2(2)	120.1(5)
C(2)—C(3)	1.385(3)	1.383(8)	C(1)—C(2)—C(3)	120.9(2)	121.0(5)
C(3)—C(4)	1.392(3)	1.392(6)	C(2)—C(3)—C(4)	120.7(2)	120.3(4)
C(4)—C(5)	1.394(3)	1.389(6)	C(3)—C(4)—C(5)	117.6(2)	118.1(4)
C(5)—C(6)	1.380(3)	1.386(7)	C(4)—C(5)—C(6)	121.3(2)	121.4(4)
C(6)—C(1)	1.375(4)	1.389(8)	C(5)—C(6)—C(1)	120.4(2)	119.1(5)
C(4)—C(7)	1.468(3)	1.475(5)	C(3)—C(4)—C(7)	122.1(2)	121.2(3)
			C(5)—C(4)—C(7)	120.3(2)	120.7(3)
			Pyrimidine ring		
C(7)—C(8)	1.389(3)	1.383(5)	C(4)—C(7)—C(8)	123.6(2)	123.1(3)
C(8)—N(1)	1.338(2)	1.329(5)	C(4)—C(7)—C(10)	122.2(2)	122.9(3)
N(1)—C(9)	1.344(3)	1.338(5)	C(8)—C(7)—C(10)	114.2(2)	113.9(3)
C(9)—N(2)	1.345(3)	1.342(5)	C(7)—C(8)—N(1)	123.9(2)	124.3(3)
N(2)—C(10)	1.330(2)	1.331(5)	C(8)—N(1)—C(9)	116.5(2)	116.7(3)
C(10)—C(7)	1.389(3)	1.387(6)	N(1)—C(9)—N(2)	124.6(2)	124.3(3)
C(9)—C(11)	1.472(3)	1.465(5)	C(9)—N(2)—C(10)	116.9(2)	116.9(3)
			N(2)—C(10)—C(7)	123.9(2)	123.8(3)
			N(1)—C(9)—C(11)	118.2(2)	118.9(3)
			N(2)—C(9)—C(11)	117.3(2)	116.8(3)
			Benzene ring 2		
C(11)—C(12)	1.395(3)	1.398(5)	C(9)—C(11)—C(12)	122.0(2)	121.5(3)
C(12)—C(13)	1.373(3)	1.377(6)	C(9)—C(11)—C(16)	120.5(2)	121.7(3)
C(13)—C(14)	1.398(3)	1.402(6)	C(16)—C(11)—C(12)	117.5(2)	116.8(3)
C(14)—C(15)	1.386(3)	1.378(5)	C(11)—C(12)—C(13)	121.3(2)	121.5(3)
C(15)—C(16)	1.380(3)	1.371(5)	C(12)—C(13)—C(14)	120.5(2)	119.9(3)
C(16)—C(11)	1.390(3)	1.390(5)	C(13)—C(14)—C(15)	119.9(2)	119.2(3)
C(14)—O	1.361(2)	1.372(5)	C(14)—C(15)—C(16)	119.9(2)	120.0(3)
			C(15)—C(16)—C(11)	121.9(2)	122.6(3)
			C(13)—C(14)—O	116.3(2)	116.1(3)
			C(15)—C(14)—O	124.8(2)	124.7(3)
			Alkyloxy group		
O—C(17)	1.447(3)	1.448(6)	C(14)—O—C(17)	117.1(2)	117.4(3)
C(17)—C(18)	1.500(3)	1.505(10)	O—C(17)—C(18)	108.2(2)	107.5(4)
C(18)—C(19)	1.500(5)	1.205(13)	C(17)—C(18)—C(19)	112.6(2)	123.1(7)
C(19)—C(20)		1.576(13)	C(18)—C(19)—C(20)		121.4(7)

calculations. In analogy to biphenyl and its derivatives, the conformation of phenyl pyrimidines is determined by two competing effects. Conjugation of the aromatic ring systems favors a coplanar conformation while repulsion between the H atoms (or substituents) in the ortho positions to the C—C interring bond favors a twisted conformation. In consequence, the conformations of 2- and 5-phenyl py-

TABLE V

Selected torsion angles (°)

	5-PPrPP	5-PBuPP
C(3)—C(4)—C(7)—C(8)	39.5(2)	37.3(5)
C(3)—C(4)—C(7)—C(10)	−141.1(2)	−142.3(3)
C(5)—C(4)—C(7)—C(8)	−140.4(2)	−142.1(4)
C(5)—C(4)—C(7)—C(10)	39.1(2)	38.3(5)
N(1)—C(9)—C(11)—C(12)	3.9(2)	8.1(4)
N(1)—C(9)—C(11)—C(16)	−177.2(2)	−173.2(4)
N(2)—C(9)—C(11)—C(12)	−176.1(2)	−171.8(4)
N(2)—C(9)—C(11)—C(16)	2.8(2)	6.9(4)
C(13)—C(14)—O—C(17)	174.4(2)	175.1(4)
C(14)—O—C(17)—C(18)	179.9(2)	−173.1(5)
O—C(17)—C(18)—C(19)	−69.8(3)	−100.8(8)
C(17)—C(18)—C(19)—C(20)		−176.1(9)

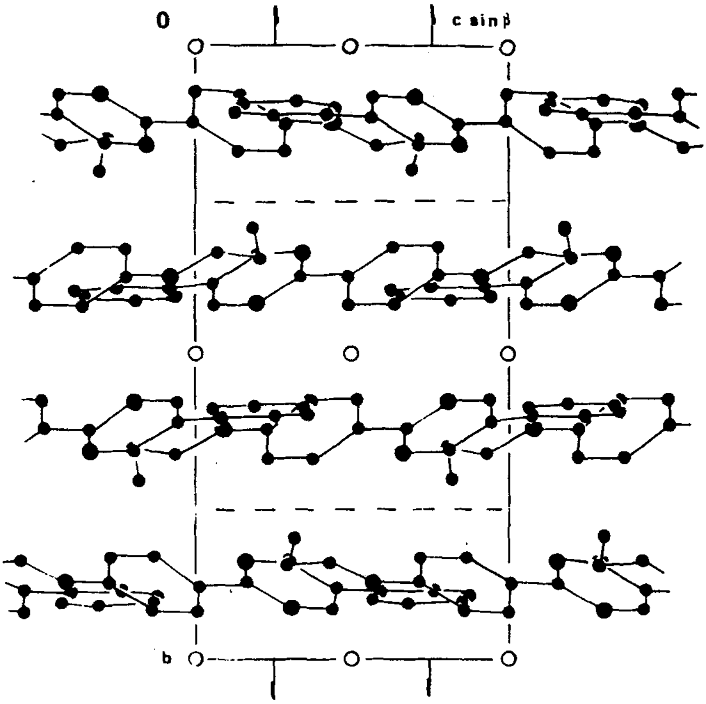


FIGURE 3 Crystal structure of 5-PPrPP projected along. [100].

rimidines should be quite different. For 5-phenyl pyrimidines a markedly twisted conformation and for 2-phenyl pyrimidines a nearly coplanar conformation are expected. Indeed, in 5-chloro-2-(4-*n*-hexoxyphenyl)-pyrimidine[1] interplanar angles of 4.3 and 6.8° (two independent molecules) were observed while for 2-methylthio-5-(4-*n*-butoxyphenyl)-pyrimidine[2] a value of 22.7° was reported. In 2,5-diphenylpyrimidines both kinds of phenyl pyrimidine isomers are present in one molecule. The observed torsion or interplanar angles in the two investigated representatives are excellent illustrations of the above conclusion that the 5-phenyl pyrimidine fragment is much more twisted than the nearly coplanar 2-phenyl pyrimidine fragment.

Crystal structure

The packing of the 5-PPrPP molecules in the crystal lattice is illustrated in Figures 3 and 4. The crystal structures of the two compounds 5-PPrPP and 5-PBuPP agree to such an extent that the following statements are true for both.

Molecules related by translations along *a* and *c* form sheets parallel to the *ac*-plane (Figure 3). The sheets on their part are stacked one upon the other in the *b* direction by the alternating action of inversion centers and *c*-glide planes with the sheet-to-sheet distance of *b*/4 = 3.80 Å. Molecules belonging to sheets related by an inversion center show parallel arrangement in a head-to-tail fashion whereas the long axes (long axis is defined as the straight line through C(1) and the

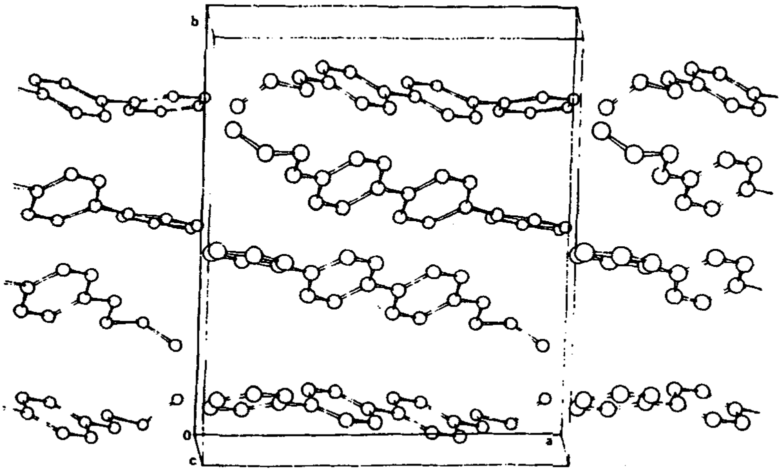


FIGURE 4 Crystal structure of 5-PPrPP.

terminal C atom of the alkyl chain) of the molecules related by the *c*-glide (or the screw axis) make an angle of about 5 (5-PPrPP) and 7° (PBuPP).

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

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