Influence of spacer chain lengths and polar terminal groups on the mesomorphic properties of tethered 5-phenylpyrimidines

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Abstract

Based on 5-(4-hydroxyphenyl)-2-octylpyrimidine 8, 5-phenylpyrimidine derivatives 3–7, 9 with different spacer chain lengths (C₂ up to C₆) and different terminal polar groups (Br, Cl, N₃, OH, CN) were synthesized by etherification and nucleophilic substitution. The mesomorphic behaviour of these compounds was investigated by differential scanning calorimetry (DSC), polarizing optical microscopy (POM) and X-ray diffraction (WAXS and SAXS) and revealed smectic A mesophases for bromides, chlorides and azides 3, 4 and 6. For these compounds a maximum phase width was observed for the C₅ spacer regardless of the terminal group, whereas the hydroxy- and cyano-substituted derivatives 5 and 7, respectively, were non mesomorphic and showed only melting transitions.

Introduction

A tremendous amount of work has been done on calamitic liquid crystals, which has led to applications in the field of LC displays [1]. Among the large family of various calamitic mesogens 2-alkoxy-5-phenylpyrimidines 1 are prominent members due to the fact that the two nitrogen atoms increase the polarity of the rigid rod core structure (Scheme 1) as exemplified by the derivative 1a which displays a SmA phase between 45 °C and

Scheme 1: Comparison of mesomorphic properties of 1a and 2a.
Scheme 2: Variation of spacer lengths and terminal group at 5-phenylpyrimidine.
Scheme 3: Synthesis of compounds 3–5, 9 and 11.

POM observation of 3a–e revealed fan-shaped textures typical of SmA phases. An illustrative example is depicted in Figure 2. The assignment of the SmA mesophases was further confirmed by XRD experiments (see the Supporting Information).

It should be noted that the allyloxy-substituted byproduct 9 showed a smectic mesophase between 50 °C and 67 °C as well. In contrast, the corresponding 4-allyloxy-4’-octylbiphenyl 11 showed only isotropic melting at 92 °C. The DSC results of chlorides 4 are summarized in Table 2.

All members 4a–e showed enantiotropic SmA phases. For compounds 4a,c,e with even numbered spacer lengths smaller mesophase widths were observed as compared to compounds 4b,d with odd numbered spacer lengths. Furthermore an odd–even effect of both melting and clearing points was found. A typical DSC curve which is shown in Figure 3 for chloride 4e with hexyloxy spacer, revealed a melting transition at 59 °C to the smectic A phase and a clearing transition at 66 °C upon a second heating. Upon the second cooling run an isotropic to SmA transition at 64 °C and a crystallization peak at 42 °C were observed.

POM investigation displayed fan-shaped and focal conic textures, as exemplified in Figure 4. XRD experiments proved the smectic phase.

In contrast to the bromides 3 and chlorides 4, the hydroxy and azide derivatives 5a,b and 7b–e were non mesomorphic and showed only melting transitions at 76 °C and 75 °C for compounds 5a,b and at 77 °C, 86 °C, 67 °C and 68 °C for the azides 7b–e, respectively (upon heating or cooling) in the DSC curve. Presumably, the higher polarity of the terminal hydroxy group with respect to the azido group, together with hydrogen bonding, inhibits mesophase formation. Next the azides 6 were investigated by DSC (Table 3).
Scheme 4:

Table 1: Phase transition temperatures [°C] and enthalpies [kJ/mol] of compounds 3.

|   | n  | Cr1  | T   | ΔH  | Cr2  | T   | ΔH  | SmA  | T   | ΔH  | I    |
|---|----|------|-----|-----|------|-----|-----|------|-----|-----|------|
| a | 2  |      | 52  | 3.9 | -    | 63  | 32.2| -    | -   | -   | •    |
|   |    |      |     | -   | -    |     |     |      |      |      | 2. heating |
| b | 3  |      | 29  | -25.8| -    | -   | -   | •    | 56  | -5.6 | •    |
|   |    |      |     |     | -    |     |     |      |      |      | 2. cooling |
| c | 4  |      | 46  | 13.0| -    | -   | -   | •    | 52  | 1.9  | •    |
|   |    |      |     |     | -    |     |     |      |      |      | 2. heating |
| d | 5  |      | 35  | -13.9| -    | -   | -   | •    | 52  | -3.2 | •    |
|   |    |      |     |     | -    |     |     |      |      |      | 2. cooling |
| e | 6  |      | 49  | -24.1| -    | -   | -   | •    | 63  | -5.0 | •    |
|   |    |      |     |     | -    |     |     |      |      |      | 2. cooling |
|   |    |      | 40  | 17.6| -    | -   | -   | •    | 57  | 5.1  | •    |
|   |    |      |     |     | -    |     |     |      |      |      | 2. heating |
|   |    |      | 24  | -16.7| -    | -   | -   | •    | 60  | -5.3 | •    |
|   |    |      |     |     | -    |     |     |      |      |      | 2. cooling |
|   |    |      | 64  | 36.3| -    | -   | -   | •    | 58  | -5.5 | •    |
|   |    |      |     |     | -    |     |     |      |      |      | 2. cooling |

Whereas compound 6a with an ethoxy spacer was non mesomorphic, enantiotropic SmA phases were detected for all other chain lengths 6b–e. Compound 6d showed an additional crystal to crystal transition. A typical DSC curve of derivative 6c is shown in Figure 5. POM revealed fan-shaped and focal conic texture, see for example Figure 6. Figure 7 and Figure 8 reveal that due to substantial supercooling for all spacer chain lengths and terminal groups the mesophases are smaller during the heating cycle as compared to the cooling cycle. The broadest mesophase was observed for the azide derivative 6d with ΔT = 27 °C upon heating and ΔT = 45 °C upon cooling. In comparison to the compounds with an azide as terminal group the halides (n = 2, 5, 6) have a lower tendency to supercooling. Whereas for azides 6 the broadest mesophase was observed for C5 spacer (6d), for chlorides 4 derivatives 4b and 4d with C3 and C5 spacer displayed similar mesophase width. For bromides 3 again the derivative 3d with C5 spacer showed the broadest mesophase.

From the X-ray data, the following model (Figure 9) of the layer structure is proposed. The d values obtained from the X-ray experiments fit with the molecular lengths derived from simple molecular modelling (Chem3D) [13]. For example, the XRD pattern of the azide derivative 6c results in a layer dis-
Figure 1: DSC curve of compound 3d (heating/cooling rate 10 K/min).

Figure 2: Fan-shaped texture of 3e under crossed polarizers upon cooling from the isotropic liquid (magnification 200×): at 55 °C (cooling rate 5 K/min): smectic A phase.

Figure 2: Fan-shaped texture of 3e under crossed polarizers upon cooling from the isotropic liquid (magnification 200×): at 55 °C (cooling rate 5 K/min): smectic A phase.

Table 2: Phase transition temperatures [°C] and enthalpies [kJ/mol] of compounds 4.a

| 4  | n  | Cr | T   | ∆H  | SmA | T   | ∆H  | I    |
|----|----|----|-----|-----|-----|-----|-----|------|
| a  | 2  | *  | 50  | 21.6| *   | 55  | 1.4 | *    |
|    |    | *  | 25  | −18.7| *  | 53  | −1.5| *    |
| b  | 3  | *  | 37  | 11.9| *   | 53  | 3.9 | *    |
|    |    | *  | 23  | −11.8| *  | 58  | −4.2| *    |
| c  | 4  | *  | 63  | 21.9| *   | 67  | 2.7 | *    |
|    |    | *  | 46  | −23.5| *  | 70  | −4.9| *    |
| d  | 5  | *  | 42  | 15.7| *   | 56  | 5.0 | *    |
|    |    | *  | 27  | −17.0| *  | 62  | −4.8| *    |
| e  | 6  | *  | 55  | 20.8| *   | 59  | 4.7 | *    |
|    |    | *  | 42  | −25.3| *  | 64  | −4.5| *    |

aCr crystalline; SmA smectic A; I isotropic; * phase was observed; - phase was not observed. Heating and cooling rate: 10 K/min for 4a–d, 5 K/min for 4e.

Conclusion
It has been shown that 5-phenylpyrimidine derivatives with terminal chloro-, bromo-, azido-, hydroxy- and cyano groups

tance of 25.5 Å, whereas the calculated length of the molecule for the most elongated conformation is 26 Å, which is clear evidence for the presence of monolayers. This leads to the assumption that the molecules might be aligned antiparallel within each smectic layer (Figure 9). Packing the molecules in this array prevents close contacts between the polar regions of the rigid core and the terminal groups. The observed maximum phase width for the C5 spacer regardless of the terminal group suggests that for this chain length space filling is optimal and the terminal group X can be accommodated well between the alkyl chains. This model might also explain why the mesophase is lost with strongly polar or hydrogen bonding terminal groups such as cyanides and hydroxy derivatives.

Conclusion
It has been shown that 5-phenylpyrimidine derivatives with terminal chloro-, bromo-, azido-, hydroxy- and cyano groups
Figure 3: DSC curve of compound 4e (heating/cooling rate 5 K/min).

Figure 4: Fan-shaped texture of compound 4d at 45 °C upon cooling from the isotropic liquid (cooling rate 1 K/min) (magnification 200×).

Table 3: Phase transition temperatures [°C] and enthalpies [kJ/mol] of compounds 6.a

| 6 | n | Cr1 | T | ΔH | Cr2 | T | ΔH | SmA | T | ΔH | I |
|---|---|-----|---|-----|-----|---|-----|-----|---|-----|---|
| a | 2 | •  | 48 | 30.6 | - | - | - | - | - | - | •  | 2. heating |
| b | 3 | •  | 45 | 11.9 | - | - | - | - | 49 | 1.1 | •  | 2. heating |
| c | 4 | •  | 42 | 25.3 | - | - | - | - | 60 | 5.0 | •  | 2. heating |
| d | 5 | •  | 7  | 0.9  | 28 | 17.2 | • | 55 | 3.4 | •  | 2. heating |
| e | 6 | •  | 20 | 17.2 | - | - | - | - | 58 | -5.9 | •  | 2. cooling |

aCr crystalline; SmA smectic A; I isotropic; • phase was observed; - phase was not observed. Heating and cooling rate: 10 K/min.
Figure 5: DSC curve of compound 6c (heating/cooling rate 10 K/min).

Figure 6: Fan-shaped texture of compound 6e at 45 °C upon cooling from the isotropic liquid (cooling rate 10 K/min) (magnification 200×).

Figure 7: Comparison of the mesophase range ΔT for the different spacer lengths of compounds 3, 4 and 6: mesophase range upon heating (heating rate 10 K/min).

Gate Single Reflection Diamant ATR system. Mass spectra were recorded on a Finnigan MAT 95 and a Varian MAT 711 apparatus. X-Ray powder experiments were performed on a Bruker Nanostar; software: SAXS 4.1.26. The samples were kept in Hilgenberg glass capillaries of 0.7 mm outside diameter in a temperature-controlled heating stage (±1 °C). A monochromatic Cu-Kα1 beam (λ = 1.5405 Å) was obtained using a ceramic tube generator (1500 W) with cross-coupled Göbel-mirrors as the monochromator. The diffraction patterns were recorded on a real-time 2D-detector (HI-STAR, Bruker). The calibration of the patterns occurred with the powder pattern of Ag-Behenate. Differential scanning calorimetry (DSC) was performed using a Mettler Toledo DSC822, and polarizing optical microscopy (POM) using an Olympus BX50 polarizing microscope combined with a Linkam LTS350 hot stage and a Linkam TP93 central processor. Flash chromatography was
Figure 8: Comparison of the mesophase range $\Delta T$ for the different spacer lengths of compounds 3, 4 and 6: mesophase range upon cooling (cooling rate 10 K/min).

Figure 9: Proposed model for the layer structure.

performed using Kieselgel 60, 40–63 μm (Fluka). All solvents were dried, and reactions were performed in dried glassware. The used petroleum ether (PE) had a boiling range of 30–75 °C.

**General procedure 1**
To a solution of 5-(4-hydroxyphenyl)-2-octylpyrimidine 8 (852 mg, 3.00 mmol) in 4 mL DMSO was added powdered KOH (504 mg, 9.00 mmol). After stirring for 10 min at room temperature, the $\alpha,\omega$-dihaloalkane (or $\alpha$-bromo-$\omega$-alkanol respectively) (3.00 mmol) was added. Stirring was continued for 4 h followed by quenching with 20 mL H$_2$O and 100 mL CH$_2$Cl$_2$. The organic layer was dried (Na$_2$SO$_4$) and the solvents were evaporated. Finally the crude product was purified by flash chromatography.

**General procedure 2**
A solution of bromide 3 (0.50 mmol) and NaN$_3$ (81.0 mg, 1.25 mmol) in 15 mL DMF was stirred at 100 °C for 24 h. After cooling to room temperature, the reaction mixture was treated with 20 mL H$_2$O and extracted with CH$_2$Cl$_2$ (3 × 30 mL). The combined organic layers were dried (Na$_2$SO$_4$), the solvent was evaporated and the crude product purified by flash chromatography.

**General procedure 3**
A solution of bromide 3 (0.50 mmol) and KCN (35.8 mg, 0.55 mmol) in 4 mL EtOH/H$_2$O (3:1, v/v) was stirred at 110 °C for 12 h. After cooling to room temperature, 10 mL CH$_2$Cl$_2$ were added and the aqueous layer was extracted with CH$_2$Cl$_2$ (3 × 10 mL). The combined organic layers were washed with 1 N NaOH (1 × 10 mL) and dried (Na$_2$SO$_4$). Finally the solvent was evaporated and the crude product purified by flash chromatography.
5-[4-(6-Bromohexyloxy)phenyl]-2-octylpyrimidine (3e)
Prepared according to general procedure (1). Experiment:
284 mg (1.00 mmol) 5-(4-hydroxyphenyl)-2-octylpyrimidine 8, 160 μL (245 mg, 1.00 mmol) 1,6-dibromohexane, 168 mg (3.00 mmol) KOH. Flash chromatography (PE/ EtOAc, 4:1, v/v; Rf = 0.86: PE/EtOAc, 1:1, v/v) gave 268 mg (0.60 mmol, 60%) of 3e as a colourless crystalline solid. DSC: Cr 35 °C [−23.9 kJ/mol] (SmA 58 °C [−5.5 kJ/mol]) 1. 1H NMR (300 MHz, CDCl3): δ = 0.88 (t, 3H, J = 6.9 Hz, CH3), 1.21–1.45 (m, 10H, CH2), 1.49–1.58 (m, 4H, CH2), 1.79–1.98 (m, 6H, CH2), 2.96–3.01 (m, 2H, 2-CH2), 3.44 (t, 2H, J = 6.6 Hz, CH2Br), 4.02 (t, 2H, J = 6.4 Hz, OCH2), 6.99–7.03 (m, 2H, 3′-H, 5′-H), 7.46–7.50 (m, 2H, 2′-H, 6′-H), 8.83 (s, 2H, 4-H, 6-H) ppm. 13C NMR (125 MHz, CDCl3): δ = 14.1 (CH3), 22.7, 25.6, 25.9, 28.9, 29.2, 29.5, 31.9, 32.7 (CH2), 39.2 (2-CH2), 67.9 (OCH2), 115.2 (C-3′, C-5′), 128.0 (C-2′, C-6′), 126.7, 130.8 (C-1′, C-5′), 154.8 (C-4, C-6), 159.9 (C-4′), 169.5 (C-2) ppm. FT-IR (ATR): V = 2916 (m), 2848 (m), 1586 (m), 1536 (m), 1515 (m), 1445 (s), 1247 (s), 1180 (m), 1116 (m), 1011 (m), 994 (m), 838 (s), 651 (m), 608 (m) cm⁻¹. MS (EI, 70eV): m/z (%) = 384.3 (100) [M]+, 361.0 (20) [M−C6H4]+, 348.0 (78) [M + H−C6H5]+, 199.0 (10) 186.0 (28). C24H35BrN2O (447.45): calcd. C 64.42, H 7.88, N 6.26, Br 17.86; found C 64.46, H 7.88, N 6.14, Br 17.61.

5-[4-(6-Chlorohexyloxy)phenyl]-2-octylpyrimidine (4e)
Prepared according to general procedure (1). Experiment:
85.0 mg (0.30 mmol) 5-(4-hydroxyphenyl)-2-octylpyrimidine 8, 45.0 μL (47.0 mg, 0.30 mmol) 1,6-dichlorohexane, 50.0 mg (0.90 mmol) KOH. Flash chromatography (PE/EtOAc, 5:1, v/v; Rf = 0.40: PE/EtOAc, 3:1, v/v) gave 74.0 mg (0.18 mmol, 60%) of 4e as a colourless crystalline solid. DSC: Cr 42 °C [−23,5 kJ/mol] (SmA 64 °C [−4.5 kJ/mol]) 1. 1H NMR (300 MHz, CDCl3): δ = 0.85–0.90 (m, 3H, CH3), 1.23–1.44 (m, 10H, CH2), 1.50–1.55 (m, 4H, CH2), 1.78–1.90 (m, 6H, CH2), 2.96–3.01 (m, 2H, 2-CH2), 3.56 (t, 2H, J = 6.7 Hz, CH2O), 4.01 (t, 2H, J = 6.4 Hz, OCH2), 6.99–7.04 (m, 2H, 3′-H, 5′-H), 7.46–7.51 (m, 2H, 2′-H, 6′-H), 8.83 (s, 2H, 4-H, 6-H) ppm. 13C NMR (75 MHz, CDCl3): δ = 14.1 (CH3), 22.7, 25.7, 26.5, 28.8, 28.9, 29.1, 29.5, 31.9 (CH2), 39.2 (2-CH2), 45.0 (CH2Cl), 67.9 (OCH2), 115.3 (C-3′, C-5′), 127.9 (C-2′, C-6′), 126.7, 130.8 (C-1′, C-5′), 154.5 (C-4, C-6), 159.6 (C-4′), 169.6 (C-2) ppm. FT-IR (ATR): V = 2917 (m), 2849 (m), 1587 (m), 1536 (m), 1515 (m), 1445 (s), 1247 (s), 1180 (m), 1116 (m), 1011 (m), 994 (m), 838 (s), 651 (m), 608 (m) cm⁻¹. MS (EI, 70eV): m/z (%) = 384.3 (100) [M]+, 299.2 (36), 286.2 (94). HRMS (ESI): m/z [M+H]+ calcd. for C24H35ClN2O2: 385.2850; found: 385.2856. C24H35ClN2O (384.55): calcd. C 74.96, H 9.44, N 7.28; found C 75.05, H 9.27, N 7.26.

5-[4-(6-Hydroxyhexyloxy)phenyl]-2-octylpyrimidine (5e)
Prepared according to general procedure (1). Experiment:
568 mg (2.00 mmol) 5-(4-hydroxyphenyl)-2-octylpyrimidine 8, 270 μL (362 mg, 2.00 mmol) 6-bromohexane-1-ol, 336 mg (6.00 mmol) KOH. Flash chromatography (PE/EtOAc, 1:1, v/v; Rf = 0.29) gave 615 mg (1.60 mmol, 80%) of 5e as a colourless crystalline solid. Mp: 75 °C. 1H NMR (300 MHz, CDCl3): δ = 0.85–0.90 (m, 3H, CH3), 1.24–1.68 (m, 16H, CH2), 1.79–1.90 (m, 4H, CH2), 2.96–3.01 (m, 2H, 2′-CH2), 3.68 (t, 2H, J = 6.5 Hz, CH2O), 4.01 (t, 2H, J = 6.5 Hz, OCH2), 6.99–7.04 (m, 2H, 3′-H, 5′-H), 7.46–7.51 (m, 2H, 2′-H, 6′-H), 8.83 (s, 2H, 4-H, 6-H) ppm. 13C NMR (75 MHz, CDCl3): δ = 14.1 (CH3), 22.7, 25.7, 26.5, 28.8, 28.9, 29.1, 29.5, 31.9 (CH2), 39.2 (2-CH2), 62.8 (CH2OH), 68.0 (OCH2), 115.3 (C-3′, C-5′), 127.9 (C-2′, C-6′), 126.6, 130.8 (C-1′, C-5), 154.5 (C-4, C-6), 159.7 (C-4′), 169.6 (C-2) ppm. FT-IR (ATR): V = 3030 (cm, br), 2917 (s), 2848 (m), 1586 (m), 1536 (m), 1516 (m), 1466 (m), 1445 (s), 1377 (m), 1291 (m), 1288 (m), 1248 (s), 1181 (m), 1118 (m), 1060 (m), 1007 (m), 994 (m), 818 (m), 838 (s), 707 (m), 652 (m) cm⁻¹. MS (EI, 70eV): m/z (%) = 384.3 (100) [M]+, 299.2 (36), 286.2 (94). HRMS (ESI): m/z [M+H]+ calcd. for C24H35N2O2: 385.2850; found: 385.2856. C24H35N2O (384.55): calcd. C 74.96, H 9.44, N 7.28; found C 75.05, H 9.27, N 7.26.
5-[4-(6-Cyanohexyloxy)phenyl]-2-octylpyrimidine (7e)
Prepared according to general procedure (3). Experiment: 224 mg (0.50 mmol) bromide 3e, 36.0 mg (0.55 mmol) KCN. Flash chromatography (PE/EtOAc, 4:1, v/v; Rf = 0.23) gave 142 mg (0.36 mmol, 72%) of 7e as a colourless crystalline solid. Mp: 68 °C. 

1H NMR (500 MHz, CDCl3): δ = 0.88 (t, 3H, J = 6.9 Hz, CH3), 1.20–1.45 (m, 10H, CH2), 1.51–1.60 (m, 4H, CH2), 1.68–1.90 (m, 6H, CH2), 2.37 (t, 2H, J = 7.3 Hz, CH2CN), 2.96–3.01 (m, 2H, 2′-H, 6′-H), 7.46–7.51 (m, 2H, 2′-H, 6′-H). 13C NMR (125 MHz, CDCl3): δ = 14.1 (CH3), 17.1, 22.7, 25.3, 25.4, 28.4, 28.9, 29.2, 29.5, 31.9 (CH2), 39.2 (2-CH2), 67.9 (OCH2), 115.4 (C-3′, C-5′), 119.7 (CN), 128.0 (C-2′, C-6′), 126.9, 130.8 (C-1′, C-5), 154.5 (C-4, C-6), 159.6 (C-4′), 169.7 (C-2) ppm. FT-IR (ATR): ν = 3035 (w), 2950 (m), 2920 (m), 2852 (m), 1608 (m), 1582, 1541, 1540, 1503, 1474, 1479, 1476 ppm.

Analytical data of compounds 4a–d, 5d, 6a–d, 7a–d, 9, 11 and X-ray diffraction data.

Supporting Information File 1
Analytical data of compounds 4a–d, 5d, 6a–d, 7a–d, 9, 11. [http://www.beilstein-journals.org/bjoc/content/supportive/1860-5397-5-63-S1.pdf]

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13. Chem3D Pro 2008 software was used for molecular modelling.

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