Synthesis and Mesomorphic Properties of 4-((4-*n*-Alkoxy-2,3,5,6-tetrafluorophenyl)ethynyl)phenyl Methoxy-substituted Benzoates

LI, Heng-Feng(李衡峰) **YU, Hong-Bin**(余洪斌) **LIU, Ke-Gang**(刘克刚)
WEN, Jian-Xun*(闻建勋)
Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

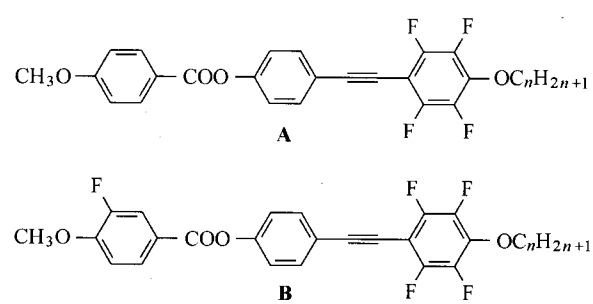
Two series of 4-methoxybenzoate liquid crystals were synthesized. Their phase transition temperatures were also measured by texture observation in a polarizing microscope and confirmed by DSC. Their mesomorphic properities and fluoro-substitute effect were studied in detail.

Keywords Liquid crystal, synthesis, mesomorphic properties

Introduction

The rapid development of flat panel technology in the recent years has attracted much attention throughout the electronic industry. Particularly, liquid crystal displays (LCDs) have been greatly expanded in size and complexity. Now among all flat panel displays, supertwist nematic liquid crystal displays (STN-LCDs) and thin-film-transistor liquid crystal displays (TFT-LCDs) have the best performance. STN-LCDs and TFT-LCDs require liquid crystals with low viscosity to reduce the response time; and with the suitable elastic constants to decrease threshold voltage of the twisted-effect and consequently the driving voltage. Fluorination, which is also well known, has a dramatic effect on mesomorphic behavior and alters certain physical properties of organic compounds, such as increasing chemical and thermal stability, and reducing viscosity.[1-6] Liquid crystals containing a perfluorinated phenyl group have been studied for several years.[7-9] Our group recently reported some fluorinated tolanes.[10-17] The perfluorophenyl-containing liquid crystals show low melting points, and tend to suppress the more ordered smectic phases, *etc*. The work reported here presents two methoxy-substituted benzoate-tolanes containing perfluorophenyl (series **A** and **B**), as shown in Scheme 1.

Scheme 1



In addition to the mesomorphic behaviors of these compounds, the effect of the fluoro-substituents in the mesogenic core is reported in this paper.

Experimental

The structures of all intermediates and final mesogens were determined by spectroscopic methods. IR spectra were recorded on a PE-983G spectrometer using a KBr disc. ^{1}H NMR spectra (with TMS as the internal standard) and ^{19}F NMR spectra (with trifluoroacetic acid (TFA) as external standard) in $CDCl_3$ as the solvent

* E-mail: lihf72@hotmail.com
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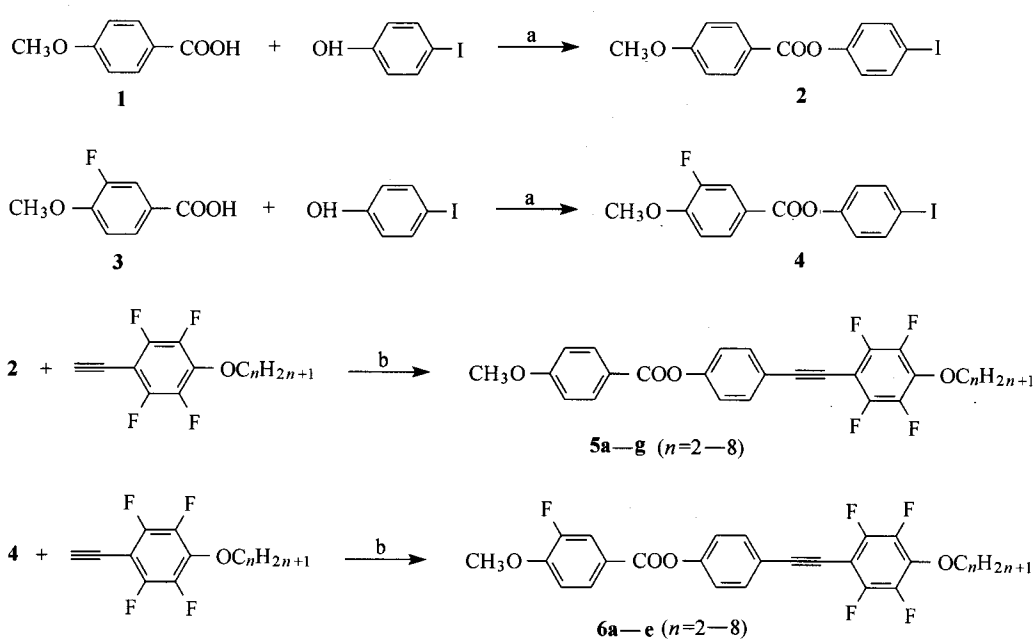
were recorded on an FX-90Q (90 MHz) or a Bruker 300 AM instrument. For ^{19}F NMR spectra, the high field was positive. MS spectra were measured with a Finnign 4021 spectrometer. The phase transition temperatures of all compounds were measured by optical microscopy using a polarizing optical microscope (POM, Olympus PM-6) equipped with a hot stage (Mettler FP-80) and a control unit (FP-82) and also by differential scanning calorimeter (DSC, Shimadzu DSC-50 calorimeter with a data system) with heating and cooling rates of 5℃/min. The transition temperatures shown in this paper are the peak values of the transitions on DSC traces. Phase identification was made by comparing the observed textures with those reported in the literature.[18-19]

The compounds (4-(4-*n*-alkyloxy-2, 3, 5, 6-tetrafluorophenyl) acetylenyl) phenyl 4′-methoxybenzoates (**5a—g**) and (4-(4-*n*-alkyloxy-2,3,5,6-tetrafluorophenyl)acetylenyl)phenyl 3′-fluoro-4′-methoxybenzoates (**6a—e**) were synthesized with the reactions shown in Scheme 2. The compounds **2** and **4** were prepared from esterification between **1** or **3** and 4-iodophenol in the presence of both dicyclohexylcarbodiimide (DCC) and DMAP catalyst in dried CH_2Cl_2. The compound 4-alkyloxy-2, 3, 5, 6-tetrafluorophenylacetylene was prepared according to the published procedure.[15] The coupling reaction between the substituted phenylacetylene and **2** (or **4**) under the catalysis of bis(triphenylphosphine)palladium dichloride and copper(I) iodide in dried triethylamine gave the target compounds.

Scheme 2 Synthetic route of the target compounds



Reagents and conditions: a, DCC, DMAP, CH_2Cl_2; b, $Pd(PPh_3)_2Cl_2$, CuI, Et_3N.

Synthesis of 4-*iodophenyl* 4′-*methoxylbenzoate* (**2**)

Compound **1** (2.80 g, 18.4 mmol), 4-iodophenol (4.0 g, 18.2 mmol), DCC (4.50 g, 21.8 mmol), catalytic DMAP (10 mg) and dry CH_2Cl_2 (60 mL) were stirred under N_2 atmosphere at room temperature for 24 h. The mixture was filtered and the residue was washed with CH_2Cl_2. The collected filtrates were evaporated on rotary evaporator. The residue was purified by flash chromatography and recrystallized from petroleum ether (bp 60—90℃) to give white solid (3.5 g, yield: 54.4%). ^1H NMR ($CDCl_3$/TMS) δ_H: 8.14 (d, J = 9 Hz, 2H), 7.73 (d, J = 9 Hz, 2H), 6.99(d, J = 9 Hz, 4H), 3.92 (s, 3H, OCH_3).

Synthesis of 4-*iodophenyl* 3-*fluoro*-4-*methoxylbenzoate* (**4**)

4-Iodophenyl 3-fluoro-4-methoxylbenzoate (**4**) was prepared with the same procedure as compound **2**.

Synthesis of **5a—g** *and* **6a—e**

All the twelve target compounds were prepared by the coupling reaction of the compound **2** and the substituted phenylacetylene in the presence of palladium catalyst.

Typical procedure In a dry 50 mL three-necked flask equipped with a magnetic stir bar, a gas inlet, a reflux condenser provided with a bubbler, were placed 4-iodophenyl 4′-methoxybenzoate (200 mg, 0.56 mmol), $Pd(PPh_3)_2Cl_2$ (20 mg, 0.03 mmol), CuI (11.5 mg, 0.06 mmol) and 20 mL of anhydrous Et_3N. The mixture was stirred under nitrogen when 123 mg (0.56 mmol) of 4-ethoxy-2, 3, 5, 6-tetrafluorophenylacetylene was added. The resulting mixture was refluxed for 5 h and TLC analysis showed the complete reaction. The precipitate was filtered off and washed with ether. The filtrate was washed with water and dried over anhydrous sodium sulfate, concentrated and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel using petroleum ether (bp 60—90℃)/CH_2Cl_2 (3 : 1) as eluent to give yellow solid which was recrystallized from petroleum ether to give white crystal of compound **5a**. Yield: 205 mg (82%). mp: 105℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1125, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 8.12(d, J = 9 Hz, 2H), 7.59(d, J = 9 Hz, 2H), 7.21(d, J = 9 Hz, 2H), 6.96(d, J = 9 Hz, 2H), 4.33(q, J = 6 Hz, 2H, OCH_2), 3.89(s, 3H, OCH_3), 1.41(t, J = 6 Hz, 3H, CH_3). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 60.5(d, J = 18.8 Hz, 2F), 80.0 (d, J = 18.8 Hz, 2F). MS m/z: 445(M^+ + 1). Anal. $C_{24}H_{16}O_4F_4$. Calcd: C, 64.86; H, 3.60; F, 17.12. Found: C, 64.87; H, 3.54; F, 17.25.

4-(4-*n*-*Propoxy*-2, 3, 5, 6-*tetrafluorophenyl*) *ethynyl* 4′-*methoxybenzoate* (**5b**) mp: 95℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1125, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 8.15(d, J = 9 Hz, 2H), 7.62(d, J = 9 Hz, 2H), 7.22(d, J = 9 Hz, 2H), 6.98(d, J = 9 Hz, 2H), 4.23(t, J = 6 Hz, 2H, OCH_2), 3.90(s, 3H, OCH_3), 1.80—1.85 (m, CH_2), 1.07(t, J = 6 Hz, 3H, CH_3). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 60.4(d, J = 18.8 Hz, 2F), 80.0 (d, J = 18.8 Hz, 2F). MS m/z: 458(M^+). Anal. $C_{25}H_{18}O_4F_4$. Calcd: C, 65.50; H, 3.93; F, 16.59. Found: C, 65.14; H, 3.84; F, 16.11.

4-(4-*n*-*Butoxy*-2,3,5,6-*tetrafluorophenyl*) *ethynyl* 4′-*methoxybenzoate* (**5c**) mp: 93℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 8.13(d, J = 9 Hz, 2H), 7.60(d, J = 9 Hz, 2H), 7.20(d, J = 9 Hz, 2H), 6.96(d, J = 9 Hz, 2H), 4.30(t, J = 6 Hz, 2H, OCH_2), 3.93(s, 3H, OCH_3), 0.81—2.00(m, 7H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 60.7(d, J = 18.8 Hz, 2F), 79.8(d, J = 18.8 Hz, 2F). MS m/z: 472 (M^+). Anal. $C_{26}H_{20}O_4F_4$. Calcd: C, 66.10; H, 4.20; F, 16.10. Found: C, 66.23; H, 4.27; F, 16.19.

4-(4-*n*-*Pentoxy*-2, 3, 5, 6-*tetrafluorophenyl*)-*ethynyl* 4′-*methoxybenzoate* (**5d**) mp: 101℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 8.10(d, J = 9 Hz, 2H), 7.61(d, J = 9 Hz, 2H), 7.19(d, J = 9 Hz, 2H), 6.94(d, J = 9 Hz, 2H), 4.23(t, J = 6 Hz, 2H, OCH_2), 3.83(s, 3H, OCH_3), 0.81—2.00 (m, 9H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 60.8(d, J = 18.8 Hz, 2F), 80.1(d, J = 18.8 Hz, 2F). MS m/z: 486(M^+). Anal. $C_{27}H_{22}O_4F_4$. Calcd: C, 66.67; H, 4.53; F, 15.64. Found: C, 67.04; H, 4.29; F, 15.87.

4-(4-*n*-*Hexoxy*-2,3,5,6-*tetrafluorophenyl*) *ethynyl* 4′-*methoxybenzoate* (**5e**) mp: 92℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 8.10(d, J = 9 Hz, 2H), 7.58(d, J = 9 Hz, 2H), 7.18(d, J = 9 Hz, 2H), 6.93(d, J = 9 Hz, 2H), 4.22(t, J = 6 Hz, 2H, OCH_2), 3.87 (s, 3H, OCH_3), 0.81—2.00 (m, 11H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 60.8(d, J = 18.8 Hz, 2F), 80.0(d, J = 18.8 Hz, 2F). MS m/z: 500 (M^+). Anal. $C_{28}H_{24}O_4F_4$. Calcd: C, 67.20; H, 4.80; F, 15.20. Found: C, 67.40; H, 4.75; F, 15.32.

4-(4-*n*-*Heptoxy*-2, 3, 5, 6-*tetrafluorophenyl*) *ethynyl* 4′-*methoxybenzoate* (**5f**) mp: 86℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 8.10(d, J = 9 Hz, 2H), 7.60(d, J = 9 Hz, 2H), 7.22(d, J = 9 Hz, 2H), 6.93(d, J = 9 Hz, 2H), 4.23(t, J = 6

Hz, 2H, OCH_2), 3.87(s, 3H, OCH_3), 0.81—2.00 (m, 13H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 60.8(d, J = 18.8 Hz, 2F), 79.8(d, J = 18.8 Hz, 2F). MS m/z: 515 (M^+ + 1). Anal. $C_{29}H_{26}O_4F_4$. Calcd: C, 67.70; H, 5.06; F, 14.97. Found: C, 67.76; H, 4.64; F, 14.93.

4-(4-*n*-*Octoxy*-2, 3, 5, 6-*tetrafluorophenyl*)-*ethynyl* 4′-*methoxybenzoate* (**5g**) mp: 79℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 8.12(d, J = 9 Hz, 2H), 7.59(d, J = 9 Hz, 2H), 7.21(d, J = 9 Hz, 2H), 6.95(d, J = 9 Hz, 2H), 4.24(t, J = 6 Hz, 2H, OCH_2), 3.87(s, 3H, OCH_3), 0.81—2.00 (m, 15H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 60.7(d, J = 18.8 Hz, 2F), 79.8(d, J = 18.8 Hz, 2F). MS m/z: 529 (M^+ + 1). Anal. $C_{29}H_{26}O_4F_4$. Calcd: C, 68.18; H, 5.30; F, 14.39. Found: C, 68.20; H, 4.93; F, 14.54.

4-(4-*n*-*Butoxy*-2,3,5,6-*tetrafluorophenyl*) *ethynyl* 3′-*fluoro*-4′-*methoxybenzoate* (**6a**) mp: 106℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 0.99(t, J = 7.25 Hz, 3H, CH_3), 1.48—1.58(m, 2H), 1.74—1.83(m, 2H), 3.99(s, 3H, OCH_3), 4.29(t, J = 6.49 Hz, 2H, OCH_2), 7.03—7.09(m, 1H), 7.24 (d, J = 9 Hz, 2H), 7.64(d, J = 9 Hz, 2H), 7.89 (d, J = 9 Hz, 2H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 57.6 (s, 1F), 62.5(d, J = 18.8 Hz, 2F), 81.5(d, J = 18.8 Hz, 2F). MS m/z: 490(M^+). Anal. $C_{26}H_{19}O_4F_5$. Calcd: C, 63.68; H, 3.90; F, 19.37. Found: C, 63.77; H, 3.93; F, 19.55.

4-(4-*n*-*Pentoxy*-2,3,5,6-*tetrafluorophenyl*) *ethynyl* 3′-*fluoro*-4′-*methoxybenzoate* (**6b**) mp: 94℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 0.95(t, J = 7.11 Hz, 3H, CH_3), 1.35—1.49(m, 4H), 1.76—1.85(m, 2H), 3.99(s, 3H, OCH_3), 4.28(t, J = 6.55 Hz, 2H, OCH_2), 7.03—7.09(m, 1H), 7.24 (d, J = 9 Hz, 2H), 7.63(d, J = 9 Hz, 2H), 8.00 (d, J = 9 Hz, 2H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 57.6 (s, 1F), 62.5(d, J = 18.8 Hz, 2F), 81.5(d, J = 18.8 Hz, 2F). MS m/z: 504(M^+). Anal. $C_{27}H_{21}O_4F_5$. Calcd: C, 64.29; H, 4.20; F, 18.83. Found: C, 64.45; H, 4.31; F, 19.26.

4-(4-*n*-*Hexoxy*-2,3,5,6-*tetrafluorophenyl*) *ethynyl* 3′-*fluoro*-4′-*methoxybenzoate* (**6c**) mp: 79℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 0.92(t, J = 6.45 Hz, 3H, CH_3), 1.34—1.53(m, 6H), 1.75—1.84(m, 2H), 3.99(s, 3H, OCH_3), 4.28(t, J = 6.48 Hz, 2H, OCH_2), 7.03—7.09(m, 1H), 7.24 (d, J = 9 Hz, 2H), 7.65(d, J = 9 Hz, 2H), 8.00 (d, J = 9 Hz, 2H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 57.6 (s, 1F), 62.5(d, J = 18.8 Hz, 2F), 81.5(d, J = 18.8 Hz, 2F). MS m/z: 518(M^+). Anal. $C_{28}H_{23}O_4F_5$. Calcd: C, 64.86; H, 4.47; F, 18.32. Found: C, 64.93; H, 4.53; F, 17.94.

4-(4-*n*-*Heptoxy*-2,3,5,6-*tetrafluorophenyl*) *ethynyl* 3′-*fluoro*-4′-*methoxybenzoate* (**6d**) mp: 91℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 0.90(t, J = 6.83 Hz, 3H, CH_3), 1.31—1.49(m, 8H), 1.74—1.83(m, 2H), 3.99(s, 3H, OCH_3), 4.27(t, J = 6.51 Hz, 2H, OCH_2), 7.03—7.09(m, 1H), 7.25 (d, J = 9 Hz, 2H), 7.64(d, J = 9 Hz, 2H), 7.89 (d, J = 9 Hz, 2H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 57.6 (s, 1F), 62.5(d, J = 18.8 Hz, 2F), 81.5(d, J = 18.8 Hz, 2F). MS m/z: 532(M^+). Anal. $C_{29}H_{25}O_4F_5$. Calcd: C, 65.41; H, 4.73; F, 17.84. Found: C, 65.69; H, 4.81; F, 17.78.

4-(4-*n*-*Octoxy*-2,3,5,6-*tetrafluorophenyl*) *ethynyl* 3′-*fluoro*-4′-*methoxybenzoate* (**6e**) mp: 90℃, IR (cm^{-1}): 2900, 2850, 2200, 1740, 1510, 1120, 1070, 875. ^1H NMR ($CDCl_3$/TMS) δ_H: 0.89(t, J = 6.93 Hz, 3H, CH_3), 1.29—1.46(m, 10H), 1.74—1.83(m, 2H), 3.99(s, 3H, OCH_3), 4.27(t, J = 6.53 Hz, 2H, OCH_2), 7.03—7.09(m, 1H), 7.25 (d, J = 9 Hz, 2H), 7.63(d, J = 9 Hz, 2H), 8.00 (d, J = 9 Hz, 2H). ^{19}F NMR ($CDCl_3$/TFA) δ_F: 57.6 (s, 1F), 62.5(d, J = 18.8 Hz, 2F), 81.5(d, J = 18.8 Hz, 2F). MS m/z: 546(M^+). Anal. $C_{30}H_{27}O_4F_5$. Calcd: C, 65.93; H, 4.98; F, 17.38. Found: C, 66.03; H, 5.07; F, 17.46.

Results and discussion

It is now well established that fluoro-substituents can improve certain characteristics of a material or may

completely change its nature. The effect on the transition temperatures of compounds by lateral fluoro-substitution in aromatic ring is well documented and often confers remarkable changes in mesotypes and transition temperatures. As explained by Gray,[20] lateral substituents usually have two opposing effects. That is, while the change in the molecular polarisability may increase the mesophase thermal stability, the decrease in the length/breadth ratio causes a decrease. The latter effect usually dominates.

The phase transition temperatures of all the compounds were determined by DSC at heating and cooling rates of 5℃/min, the mesomorphic textures were observed on an optical polarizing microscope for determining the type of mesophases. The transition temperatures shown in Table 1 are the peak values on each DSC trace.

Table 1 Phase transition temperatures (℃) of the synthesized compounds

Compounds	n	$T_{Cr\text{-}N}$	$T_{N\text{-}I}$
5a	2	105.1	242.3
5b	3	94.9	233.3
5c	4	93.3	230.7
5d	5	100.9	217.1
5e	6	92.4	211.3
5f	7	85.8	200.7
5g	8	79.2	193.8
6a	4	106.1	205.4
6b	5	93.6	193.5
6c	6	78.7	189.3
6d	7	91.4	180.3
6e	8	89.8	176.5

The transition temperatures of the two synthesized families **5a—g** and **6a—e** are listed in Table 1 and illustrated in Fig. 1.

All of the compounds in series **A** exhibit only a nematic mesophase. The clearing points of both series tend to decrease with increasing alkoxy chain length n, and the nematic phase ranges tend to narrow. To study the influence of the fluoro-substituents in the benzoates, series **B** has been synthesized. All of the compounds in series **B** exhibit only a nematic phase too, and by increasing the alkoxy chain length, the clearing points and the nematic phase ranges decrease similarly to those of compounds in series **A**. However, compared with series **A**, the clearing points of compounds in series **B** show a drop of 25℃ averagely, but the melting points show little change, so the phase ranges of compounds in series **B** narrowed. The fluoro-substituent in the 3-position of the benzoate increases the molecular polarisability, and broadens the molecule. As above-mentioned, the latter effect dominates the thermal stability of the mesophase in series **B**. So the compounds in series **B** show lower thermal stability of nematic phase.

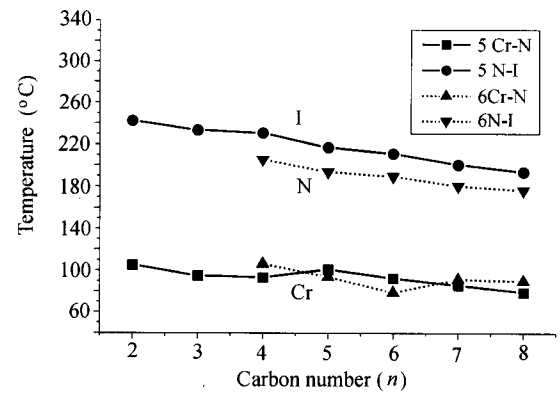


Fig. 1 Transition temperatures as a function of the number of carbon atoms in the alkoxy chain of series **A** and **B**.

The melting points of compounds in both series show an irregular tendency. That is, along with increasing of the alkoxy chain, the melting points tend to decrease firstly, then increase, decrease at last. It is plausibly attributed to the nature of this kind of molecules.

In summary, this investigation, together with our previous study on mesogens containing a perfluorinated tolane group, reveals that when a nonpolar group (such as alkoxy, alkyl, *etc.*) substituted in one end of this three ring system, compounds usually exhibit a broader nematic mesophase, and the fluoro-substituent also plays an important role in the formation of liquid crystalline states.

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