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Synthesis and properties of new three-ring phenylacetylene liquid crystals containing the cyclohexyl unit

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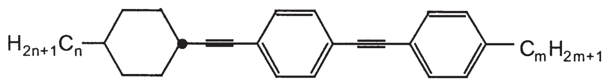
Two series of new three-ring phenylacetylene liquid crystals were synthesized and their physical properties were evaluated. These liquid crystal materials show only a nematic phase, high clearing points and good solubilities. The compounds containing two cyclohexyl rings exhibit moderate birefringence (Δn) values of around 0.14, while those containing one cyclohexyl ring show a broad nematic phase range and high Δn of around 0.33.

1. Introduction

Birefringence is a very important property of liquid crystals, and must be carefully controlled in liquid crystal display applications. Moderate birefringence (about 0.15) liquid crystals are employed in conventional TN and STN displays. High birefringence $(\Delta n > 0.3)$ liquid crystals are useful in flat panel liquid crystal displays that operate by a reflecting or scattering mechanism, such as PDLCDS, cholesterics, holographic switching devices and directional reflectors [1–6]. In addition, these materials are thought to be applicable to laser beam steering, infrared spatial modulators and optical communication [7–9]. From the single-band model [10], the Δn of a liquid crystalline compound is determined mainly by its electron conjugation, differential oscillator strength and order parameter. Molecules that contain highly polarizable groups with high electron density, such as benzene rings or acetylene linking groups, will therefore have large optical anisotropies. Bistolane liquid crystals show birefringences greater than 0.3 in the visible spectral region and as a result have attracted particular attention [11–21]. However, these materials exhibit poor solubility and high viscosities, and the melting points of bistolane liquid crystals without lateral groups are extraordinarily high (>150°C). It is known that replacing a phenyl group with a *trans*-1,4-disubstituted cyclohexane ring in liquid crystal molecules may reduce viscosity, increase the clearing point [22] and enhance solubility. In this paper, we report the synthesis and physical properties of new phenylacetylene derivatives containing one and two cyclohexyl groups; their general structures are shown in scheme 1.

Series A



Series B

Scheme 1. Structures of the target compounds.

2. Experimental

2.1. *Characterization techniques*

The structures of the final products and intermediates were confirmed by a variety of spectral methods. IR spectra were recorded on a PE-983G spectrophotometer. ^1H NMR spectra with TMS as internal standard were recorded on a Varian EM 360L spectrometer (60 MHz). Mass spectra were measured with a Finnigan-4021 mass spectrometer. Elemental analyses were performed on a Heraeus (Germany) Rapid CHN-O instrument. The purity of each compound was verified by HPLC analysis (ODSA-212 column) and all compounds had purifies greater than 99%. The phase transition temperatures of the target compounds were measured visually by polarizing optical microscopy using an Olympus PM-6 microscope fitted with a Mettler FP-80 heating stage and a FP-82 control unit; these were confirmed using differential scanning calorimetry (DSC, TA instrument DSC-2010 calorimeter with a data system) with a heating rate of

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5°C min^{-1}. Refractive indices were evaluated as extrapolated values from mixtures containing a 10 wt% solution of each test compound in MJ931381 (Merck Japan). An Abbe refractometer (2T, Atago) with a sodium lamp (589 nm) was used to measure the refractive indices of the mixtures at 20°C.

2.2. *Synthesis*

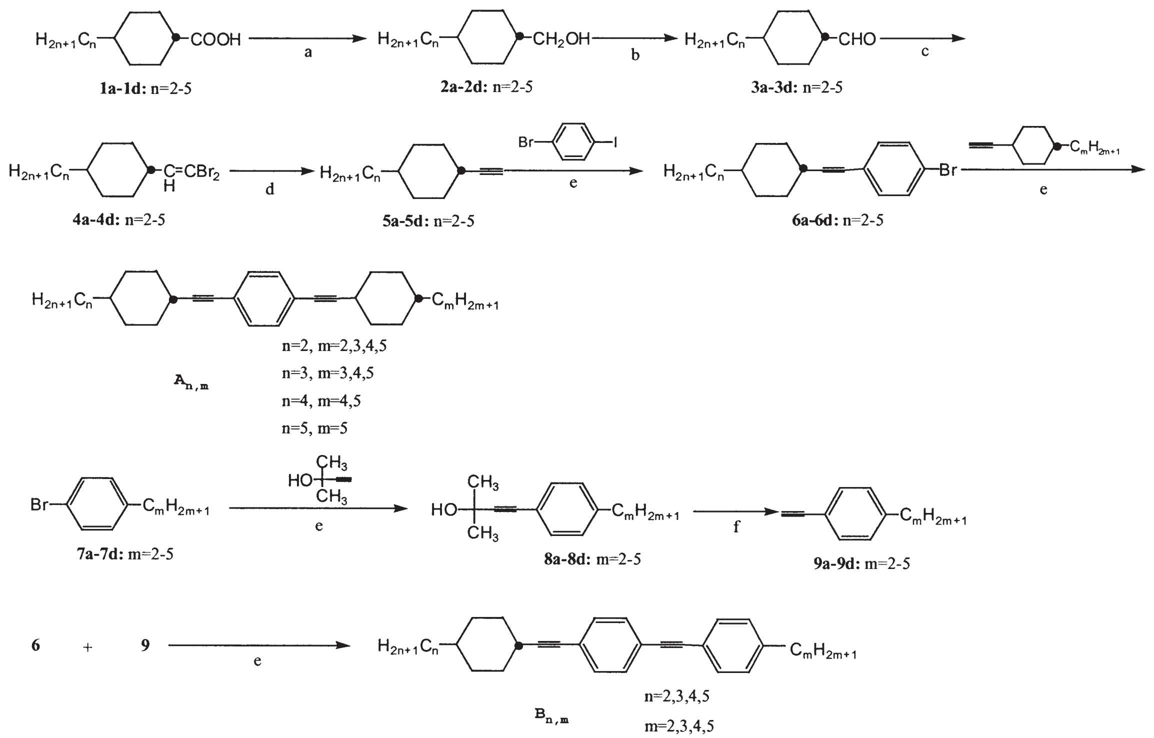
The synthesis of the target compounds is outlined in scheme 2. All homologues were prepared using the same methods and therefore the general experimental procedures are illustrated using compounds $\mathbf{A}_{2,2}$ and $\mathbf{B}_{2,2}$ as examples.

2.2.1. *trans-4-Ethylcyclohexylmethanol,* ***2a***

A solution of compound **1a** (78.0 g, 0.5 mol) in dry THF (400 ml) was added dropwise to a stirred suspension of $LiAlH_4$ (38.0 g, 1.0 mol) in dry THF (400 ml) below 0°C. The reaction mixture was stirred for 2 h at room temperature. A solution of potassium sodium tartrate (84.0 g, 0.4 mol) in H_2O (300 ml) was added dropwise to the reaction mixture, which was then filtered, and the insoluble fraction washed with THF. The combined organic layers were dried (Na_2SO_4), the solvent removed *in vacuo*, and the residue purified by distillation under reduced pressure. Yield 63.1 g (88.9%), b.p. 78–80°C/0.5 mmHg. ν_{max}(film)/cm^{-1}: 3266, 2908, 2844, 1452, 1376, 1222, 1072, 977, 894, 757. ^1H NMR(δ): 0.85–1.00 (m, 7H), 1.13–1.19 (m, 3H), 1.23–1.33 (m, 2H), 1.67–1.79 (m, 4H), 3.44–3.46 (d, 2H, $J = 6.3$ Hz).

2.2.2. *trans-4-Ethylcyclohexylmethanal,* ***3a***

A solution of compound **2a** (61.2 g, 0.43 mol) in dry CH_2Cl_2 (150 ml) was added to a stirred suspension of pyridinium chlorochromate (PCC, 139.2 g, 0.64 mol) in dry CH_2Cl_2 (400 ml) at room temperature. The reaction mixture was stirred for 2 h, and then dry ether (500 ml) was added and stirred for 5 min. The mixture was stood for 1 h, and the solution was poured off. The insoluble residue was washed with dry ether (3 × 100 ml), and the combined organic solution was concentrated, and the residue purified by distillation under reduced pressure. Yield 50.4 g (82.5%), b.p. 61–63°C/5 mmHg. ν_{max} (film)/cm^{-1}: 2921, 2850, 1697, 1448, 1419, 1317, 1263, 1000, 958, 896, 748. ^1H NMR(δ): 0.83–0.95 (m, 5H), 1.07–1.22 (m, 3H), 1.25–1.46 (m, 2H), 1.79–1.83 (m, 2H), 1.97–2.01 (m, 2H), 2.18–2.26 (m, 1H), 9.68 (d, 1H, $J = 6.8$ Hz).



Scheme 2. Synthetic route for series **A** and **B** compounds. Reagents and conditions: a. (1) $LiAlH_4$/THF (2) H_2O; b. PCC/CH_2Cl_2; c. Zn/CBr_4/PPh_3/CH_2Cl_2; d. (1) *n*-BuLi/*n*-hexane (2) H_2O; e. Pd(PPh_3)Cl_2/PPh_3/CuI/Et_3N/ethyl acetate; f. NaOH/toluene.

2.2.3. *trans-4-Ethylcyclohexyl-β,β′-dibromoethene,* ***4a***

Triphenylphosphine (190.1 g, 0.73 mol) was added in portions to a stirred mixture of carbon tetrabromide (240.7 g, 0.73 mol) and zinc (47.4 g, 0.73 mol) in dry CH_2Cl_2 (600 ml). The suspension was stirred for 24 h, then compound **3a** (50.4 g, 0.36 mol) was added and the mixture stirred for 3 h. The reaction mixture was filtered, the solvent removed *in vacuo*, and the residue purified by distillation under reduced pressure. Yield 81.5 g (76.5%), b.p. 98–100°C/1 mmHg. ν_{max} (film)/cm^{-1}: 2992, 2923, 2850, 1618, 1448, 1376, 1079, 898, 835, 767. ^{1}H NMR(δ): 0.86–0.99 (m, 5H), 1.04–1.18 (m, 3H), 1.21–1.28 (m, 2H), 1.77–1.81 (m, 4H), 2.17–2.28 (m, 1H), 6.20–6.23 (d, 1H, $J=9.0$ Hz).

2.2.4. *trans-4-Ethylcyclohexylethyne,* ***5a***

A solution of *n*-butyllithium in *n*-hexane (308.6 ml, 0.56 mol) was added dropwise to a stirred solution of compound **4a** (81.5 g, 0.28 mol) in dry THF (600 ml) at −78°C under nitrogen. The reaction mixture was stirred for 2 h at −78°C, and was then warmed to room temperature. Water (400 ml) was added to the reaction mixture. The product was extracted into *n*-hexane (2×200 ml) and the combined organic layers were washed with water (2×200 ml) and then dried (Na_2SO_4). The solvent was removed *in vacuo*, and the residue purified by distillation under reduced pressure. Yield 27.2 g (71.5%), b.p. 36–38°C/1.1 mmHg. ν_{max}/cm^{-1} (film): 3297, 2929, 2871, 2109, 1508, 1488, 1378, 1072, 838, 642. ^{1}H NMR (δ): 0.85–1.05 (m, 5H), 1.08–1.20 (m, 3H), 1.25–1.40 (m, 2H), 1.65–1.76 (m, 3H), 1.97–2.02 (m, 2H), 2.09–2.18 (m, 1H).

2.2.5. *1-Bromo-4-(trans-4-ethylcyclohexylethynyl)-benzene,* ***6a***

Dichlorobis(triphenylphosphine)palladium (0.5 g, 0.7 mmol), CuI (0.5 g, 2.6 mmol) and triphenylphosphine (1.0 g, 3.8 mmol) were added to a solution of 1-bromo-4-iodobenzene (42.5 g, 0.15 mol), **5a** (20.4 g, 0.15 mol) and triethylamine (100 ml) in ethyl acetate (200 ml). The reaction mixture was stirred at 65°C for 8 h. After cooling to room temperature, it was filtered, and the insoluble fraction washed with ethyl acetate. The combined organic solution was dried (Na_2SO_4), and the solvent removed *in vacuo*. The residue was purified by column chromatography (silica gel, *n*-hexane), then recrystallized from aqueous ethanol to give **6a** (white crystals). Yield 34.2 g (78.4%), m.p. 97.3°C. IR ν_{max} (KBr, cm^{-1}): 2917, 2848, 2225, 1583, 1484, 1444, 1392, 1068, 941, 823. ^{1}H NMR(δ): 0.86–0.98 (m, 5H), 1.12–1.32 (m, 3H), 1.34–1.48 (m, 2H), 1.76–1.80 (m, 2H), 2.00–2.05 (m, 2H), 2.32–2.35 (m, 1H), 7.22–7.25 (d, 2H, $J=8.4$ Hz), 7.38–7.41 (d, 2H, $J=8.4$ Hz).

2.2.6. *1-(trans-4-Ethylcyclohexylethynyl)-4-(trans-4-ethylcyclohexylethynyl)benzene,* $\boldsymbol{A_{2,2}}$

The preparative method was the same as for the preparation of compound **6a**. The following quantities were used: **6a** (0.58 g, 2.0 mmol), **5a** (0.27 g, 2.0 mmol), triethylamine (10 ml) ethyl acetate (20 ml), dichlorobis (triphenylphosphine)palladium (0.007 g, 0.01 mmol), CuI (0.007 g, 0.036 mmol) and triphenylphosphine (0.014 g, 0.053 mmol). Yield 0.52 g (75.8%), m.p. 149.7°C. IR ν_{max}(KBr, cm^{-1}): 3030, 2923, 2842, 2223, 1511, 1446, 1375, 941, 837, 551. ^{1}H NMR (δ): 0.88–1.00 (m, 10H), 1.17–1.32 (m, 6H), 1.35–1.46 (m, 4H), 1.67–1.82 (m, 4H), 2.03–2.08 (m, 4H), 2.35–2.40 (m, 2H), 7.31(s, 4H). MS (*m/z*): 346 (M^+, 61.1), 264 (100.0), 182 (99.6). Elemental analysis (%): calc. (found) for $C_{26}H_{34}$: C 90.17 (90.09); H 9.83 (9.85).

2.2.7. *4-4′-Ethylphenyl-3-butyne-2-ol,* ***8a***

The preparative method was the same as for the preparation of compound **6a**. The following quantities were used: *p*-bromoethylbenzene (37.0 g, 0.2 mol), 2-methyl-3-butyne-2-ol (18.2 g, 0.22 mol), triethylamine (85 ml), ethyl acetate (170 ml), CuI (0.7 g, 3.6 mol), triphenylphosphine (1.4 g, 5.3 mmol) and dichlorobis (triphenylphosphine)palladium (0.7 g, 1.0 mmol). The crude product was purified by distillation under reduced pressure. Yield 29.2 g (78.9%), b.p. 126–128°C/5.0 mmHg. IR ν_{max}(film, cm^{-1}): 3332, 2981, 2937, 2228, 1615, 1509, 1457, 1178, 970, 822.

2.2.8. *4-Ethylphenylacetylene,* ***9a***

NaOH (9.6 g, 0.24 mol) was added to a solution of compound **8a** (29.2 g, 0.16 mol) in toluene (300 ml). The reaction mixture was heated under reflux for 4 h, then cooled to room temperature and filtered. The organic solution was dried (Na_2SO_4); toluene was removed *in vacuo*. The crude product was purified by distillation under reduced pressure to give a colourless liquid. Yield 15.2 g (75.1%), b.p. 69–71°C/5.0 mmHg. IR ν_{max}(film, cm^{-1}): 3297, 2929, 2871, 2109, 1608, 1508, 1465, 1378, 1012, 838.

2.2.9. *1-(trans-4-Ethylcyclohexylethynyl)-4-(4-ethylphenylethynyl)benzene,* $\boldsymbol{B_{2,2}}$

The preparative method was the same as for the preparation of compound **6a**. The following quantities were used: **6a** (0.58 g, 2.0 mmol), **9a** (0.26 g, 2.0 mmol), triethylamine (10 ml) ethyl acetate (20 ml), dichlorobis (triphenylphosphine)palladium (0.007 g, 0.01 mmol), CuI (0.007 g, 0.036 mmol) and triphenylphosphine (PPh_3,0.014 g, 0.053 mmol). Yield 0.59 g (86.8%), m.p. 123.2°C. IR ν_{max}(KBr, cm^{-1}): 2960, 2929, 2850, 2219, 1515, 1448, 1370, 894, 835, 547. ^1H NMR(δ): 0.84–1.06 (m, 8H), 1.17–1.39 (m, 3H), 1.43–1.57 (m, 2H), 1.69–1.78 (m, 2H), 2.05–2.09 (m, 2H), 2.36–2.42 (m, 1H), 2.62–2.67 (quint, 2H, $J = 7.5$ Hz), 7.17–7.19 (d, 2H, $J = 8.1$ Hz), 7.26–7.33 (d, 2H, $J = 8.1$ Hz), 7.41–7.45 (quint, 4H, $J = 3.9$ Hz). MS (*m*/*z*): 340 (M^+, 83.6), 258 (100.0), 229 (34.9). Elemental analysis (%): calc. (found) for $C_{26}H_{28}$: C 91.76 (91.69); H 8.24 (8.21).

3. Results and discussion

The phase transition temperatures, enthalpies and birefringence values (Δn) for series **A** and **B** are listed in table 1. As shown in the table, all the compounds exhibit an enantiotropic nematic phase except $\mathbf{A_{2,2}}$. The symmetry of the molecule ($m = n = 2$) and the poor flexibility of the ethyl result in the disappearance of a mesomorphic phase for $\mathbf{A_{2,2}}$.

For series **A**, when $n \neq 2$, the clearing points (T_c) of the compounds are all about 190°C. On keeping the total carbon number of the two terminal alkyl groups constant, such as $\mathbf{A_{2,4}}$ and $\mathbf{A_{3,3}}$; and $\mathbf{A_{3,5}}$ and $\mathbf{A_{4,4}}$, the asymmetric compound generally shows a lower melting point (T_m) than does the symmetric homologue. For example, T_m of compound $\mathbf{A_{3,3}}$ is 164.9°C, while that of $\mathbf{A_{2,4}}$ is 113.3°C. The larger the difference in carbon number between the two terminal alkyl chains of the compound, the lower T_m is and thus, $\mathbf{A_{2,5}}$ has the lowest T_m. The higher T_m of the symmetric compounds might be caused by more efficient molecular packing arising from the molecular symmetry.

The series **B** compounds exhibit high clearing points ranging from 170 to 212°C. Except for $\mathbf{B_{2,2}}$, the nematic phase temperature range of all these compounds is over 73°C. For pairs of isomers of series **B**, for example, $\mathbf{B_{2,3}}$ and $\mathbf{B_{3,2}}$, and $\mathbf{B_{2,4}}$ and $\mathbf{B_{4,2}}$, when $n < m$ the compound exhibits lower T_m and T_c values (see table 1). This may be because of an enhanced microphase separation arising from the segregation of alkyl and aromatic segments.

For compounds $\mathbf{B_{\mathit{n},3}}$ that contain the same propyl group on the right side and a different alkyl group on the left, phase transition temperatures are plotted against the carbon number (n) of the left alkyl group in figure 1. This reveals that both the melting and clearing temperatures show an odd–even effect.

Figure 2 gives the plot of the phase transition temperature versus carbon number (m) of the right terminal alkyl group of the compounds $\mathbf{B_{3,\mathit{m}}}$ and shows that both the melting and clearing points decrease gradually with increasing carbon number. For the other three groups of compounds, $\mathbf{B_{2,\mathit{m}}}$, $\mathbf{B_{4,\mathit{m}}}$ and $\mathbf{B_{5,\mathit{m}}}$, the phase transition temperatures show essentially the same trend as for compounds $\mathbf{B_{3,\mathit{m}}}$.

The melting points of series **B** compounds with equivalent alkyl/alkyl (*n*/*m*) chains are lower than those of the corresponding series **A** compounds (see table 1). For example, T_m of $\mathbf{B_{3,4}}$ and $\mathbf{A_{3,4}}$ is 101.9 and 147.9°C, respectively. When two compounds have the same

Table 1. Transition temperatures (°C), associated enthalpy changes (kJ mol^{-1}) in square brackets, and birefringence values (Δn) for series **A** and **B** homologues. Cr = crystal, N = nematic phase, I = isotropic phase.

Compound	*n*	*m*	Cr		N		I	Δn[a]
$\mathbf{A_{2,2}}$	2	2	●	149.7[11.9]			●	
$\mathbf{A_{2,3}}$	2	3	●	135.6[15.8]	●	168.6[1.4]	●	0.148
$\mathbf{A_{2,4}}$	2	4	●	113.3[18.0]	●	178.1[1.1]	●	0.152
$\mathbf{A_{2,5}}$	2	5	●	97.9[13.7]	●	169.6[0.8]	●	0.149
$\mathbf{A_{3,3}}$	3	3	●	164.9[14.0]	●	198.5[1.3]	●	0.146
$\mathbf{A_{3,4}}$	3	4	●	147.9[16.0]	●	194.9[1.3]	●	0.141
$\mathbf{A_{3,5}}$	3	5	●	116.0[19.8]	●	195.7[1.5]	●	0.148
$\mathbf{A_{4,4}}$	4	4	●	138.7[9.3]	●	190.4[0.9]	●	0.132
$\mathbf{A_{4,5}}$	4	5	●	121.6[17.0]	●	192.2[1.2]	●	0.150
$\mathbf{A_{5,5}}$	5	5	●	132.6[15.6]	●	191.8[1.4]	●	0.128
$\mathbf{B_{2,2}}$	2	2	●	123.1[16.9]	●	181.1[1.2]	●	0.323
$\mathbf{B_{2,3}}$	2	3	●	114.8[15.3]	●	188.6[1.3]	●	0.328
$\mathbf{B_{2,4}}$	2	4	●	102.6[15.4]	●	175.3[1.3]	●	0.322
$\mathbf{B_{2,5}}$	2	5	●	87.4[16.9]	●	170.8[0.7]	●	0.321
$\mathbf{B_{3,2}}$	3	2	●	123.2[14.1]	●	208.0[0.9]	●	0.338
$\mathbf{B_{3,3}}$	3	3	●	123.9[16.0]	●	212.7[1.1]	●	0.341
$\mathbf{B_{3,4}}$	3	4	●	101.9[17.7]	●	199.3[1.6]	●	0.339
$\mathbf{B_{3,5}}$	3	5	●	90.4[13.6]	●	198.2[1.3]	●	0.328
$\mathbf{B_{4,2}}$	4	2	●	113.0[18.5]	●	194.8[1.3]	●	0.331
$\mathbf{B_{4,3}}$	4	3	●	113.6[17.3]	●	204.5[1.4]	●	0.337
$\mathbf{B_{4,4}}$	4	4	●	93.9[17.5]	●	192.5[1.5]	●	0.340
$\mathbf{B_{4,5}}$	4	5	●	85.1[15.7]	●	193.7[1.4]	●	0.322
$\mathbf{B_{5,2}}$	5	2	●	115.6[9.0]	●	207.0[0.6]	●	0.348
$\mathbf{B_{5,3}}$	5	3	●	116.1[13.1]	●	206.7[1.1]	●	0.339
$\mathbf{B_{5,4}}$	5	4	●	95.1[12.4]	●	195.3[0.9]	●	0.346
$\mathbf{B_{5,5}}$	5	5	●	94.8[11.8]	●	188.4[0.8]	●	0.336

[a] Extrapolated from 10 wt % solution in MJ931381 at 20°C and $\lambda = 589$ nm.

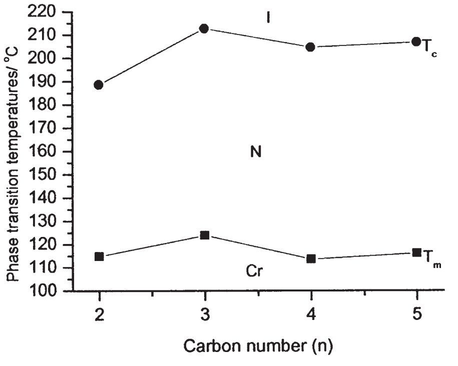


Figure 1. T_m and T_c of $\mathbf{B_{\mathit{n},3}}$ compounds. Cr = Crystal; N = Nematic; I = Isotropic phase.

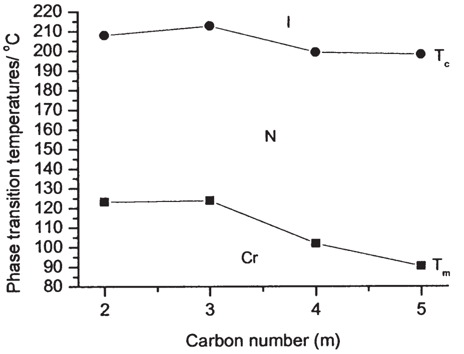


Figure 2. T_m and T_c of $\mathbf{B}_{3,m}$ compounds.

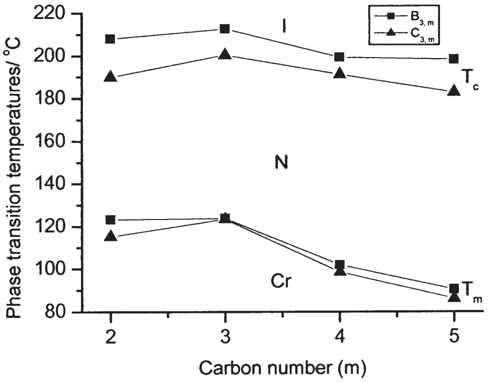
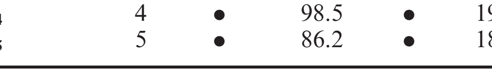


Figure 3. T_m and T_c of $\mathbf{B}_{3,m}$ and $\mathbf{C}_{3,m}$ compounds.

terminal alkyl groups, the symmetry of the molecular core in series **A** might also influence the melting point; that is, the compound that contains a symmetric core show a higher T_m value.

Lateral methyl substitution in the middle phenyl ring significantly reduces the melting temperatures of bistolane liquid crystals [17]. The phase transition temperatures of series **C** compounds are listed in table 2. Figure 3 shows that the clearing points of compounds $\mathbf{B}_{3,m}$ are higher than those of compounds $\mathbf{C}_{3,m}$, while their melting points are more similar. Thus, introducing a methyl group into the middle phenyl ring of a bistolane or replacing a phenyl of a bistolane with a *trans*-1,4-disubstituted cyclohexane ring has the same effect of reducing the melting point.

Table 2. Transition temperatures (°C) for series **C** [17].



Compound	m	Cr		N		I
$\mathbf{C}_{3,2}$	2	●	115.3	●	190.0	●
$\mathbf{C}_{3,3}$	3	●	123.4	●	200.4	●
$\mathbf{C}_{3,4}$	4	●	98.5	●	191.2	●
$\mathbf{C}_{3,5}$	5	●	86.2	●	182.8	●

In comparison with other high birefringence liquid crystals, the series **A** and **B** show small heats of fusion, and are expected to be useful components in liquid crystal mixtures for LCDs from the point of view of solubility. The birefringence of series **B** compounds is around 0.33, which is higher than that of series **A** (only 0.14) and tolane liquid crystals, but slightly lower than that of bistolane liquid crystals. This is not difficult to understand because the birefringence value is determined mainly by the molecule's conjugation length.

4. Conclusions

The synthesis and characterization of two series of three-ring phenylacetylene-based liquid crystals containing one or two cyclohexyl rings have been described. All the compounds show enantiotropic nematic phase except $\mathbf{A}_{2,2}$. The symmetry of the molecule and the relative lengths of alkyl and aromatic segments, and have an important influence on the phase transition temperatures of these compounds. The compounds containing two cyclohexyl rings have moderate Δn values of around 0.14. The compounds containing one cyclohexyl ring show a broad nematic range, high clearing points and high Δn values of around 0.33.

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