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## Liquid Crystals

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# Synthesis of Schiff's base and azo liquid crystalline crown ethers

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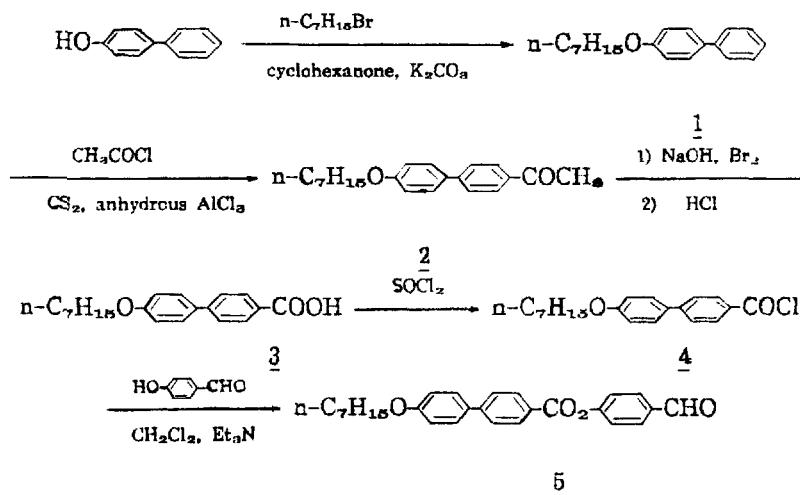
Six Schiff's base and azo liquid crystalline crown ethers were first synthesized via the intermediates *cis(trans)*-4,4'-diaminodibenzo-18-crown-6 and 4'-aminobenzo-15-crown-5, respectively. The structures were elucidated by elementary analysis, IR, <sup>1</sup>H NMR and MS. The mesomorphic properties were determined by DSC and optical microscopy. The relationship between the structures of the double-armed and mono-armed crown ethers, and the mesomorphic properties were studied. We realized that when the *L/D* ratio of the double-armed crown ether was in the range of 4-8 and that of the mono-armed crowns was larger than 3, they would possess liquid crystallinity. This conclusion is consistent with our previous investigations.

## 1. Introduction

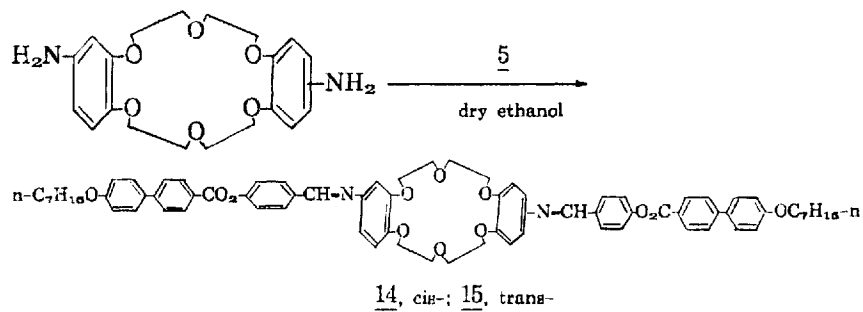
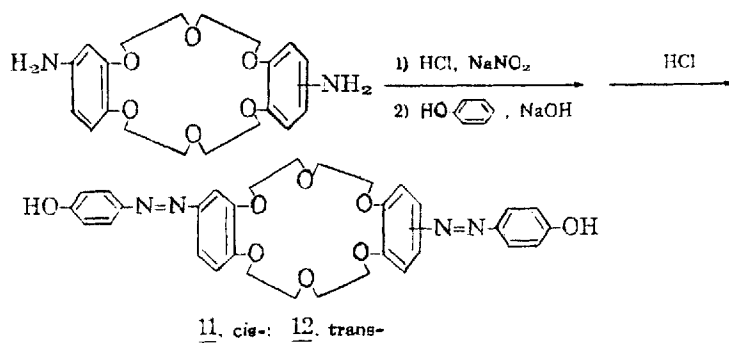
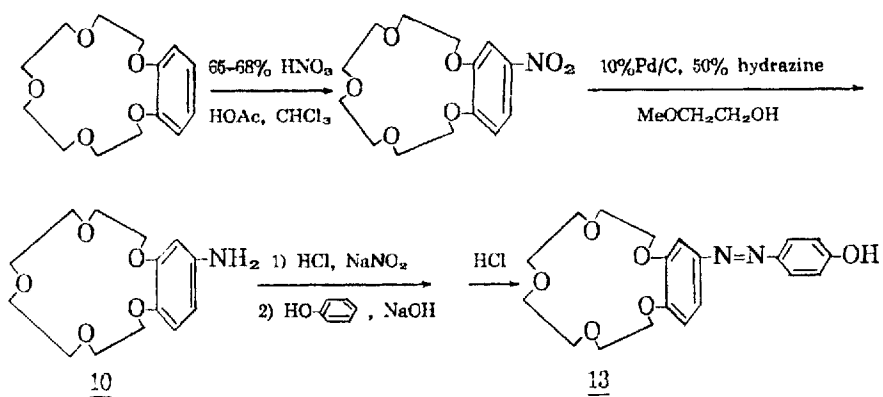
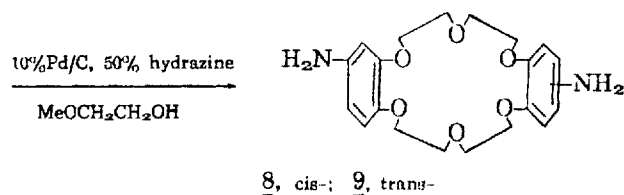
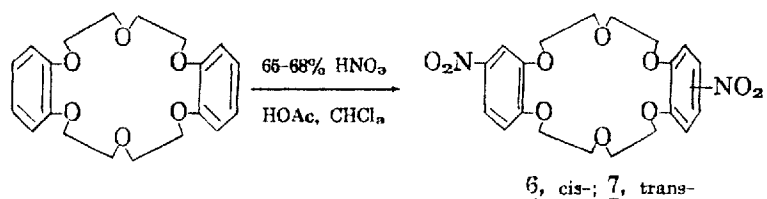
Liquid crystalline crown ethers, which possess the properties of both crown ethers and liquid crystals, have attracted extensive attention in the field of chiral recognition, ion transportation and functional membranes, etc., [1-3] since they were first reported in 1982. The synthesis of amide and ester-type liquid crystalline crown ethers, etc., have already been reported in the literature [4-6], but the synthesis of Schiff's base and azo type of liquid crystalline benzo crown ethers have not yet been successfully synthesized [6, 7].

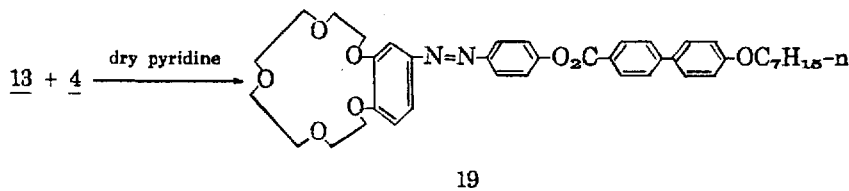
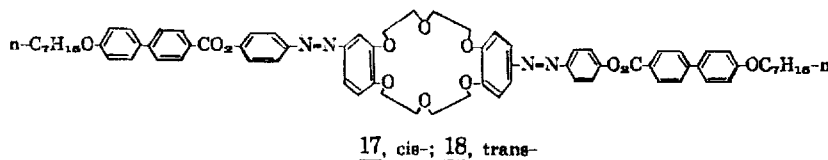
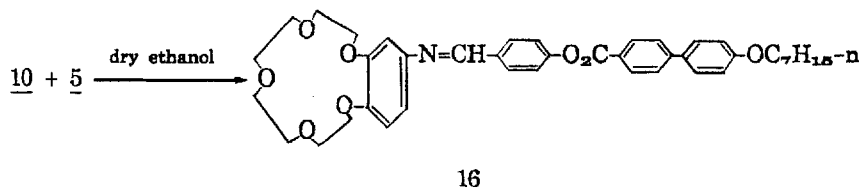
In our opinion, in order to acquire mesomorphic crown ethers, some additional factors, as well as the generally considered properties, should be involved. They are polarity and the molecular length to diameter ratio (*L/D*). Under such direction, mono-armed and double-armed Schiff's base type (14, 15, 16) and azo type (17, 18, 19) crown ethers were designed with the aid of CPK atomic models. Six novel crown ethers and two intermediates demonstrated mesomorphic properties.

The scheme of synthesis is shown as follows.



\*Author for Correspondence.



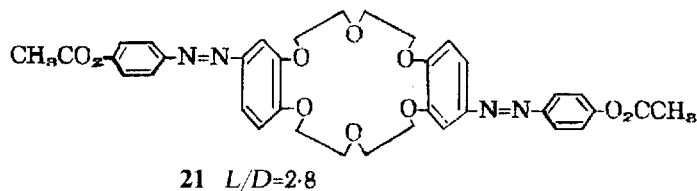
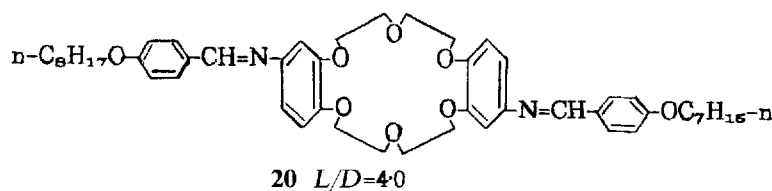
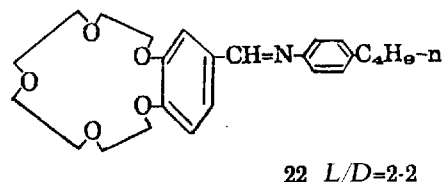


## 2. Results and discussion

Crown ether compounds **14–19** and intermediates **2, 5** designed referring to CPK atomic models were synthesized. Mesomorphic properties were determined by DSC and polarizing microscopy (see figures 1–9). Their phase transition temperatures and  $L/D$  values are listed in the table.

The  $L/D$  ratio of the compounds played an important role in the formation of liquid crystallinity. In general, rod-shaped liquid crystal molecules have  $L/D$  values larger than 4 [8]. Xie *et al.*, reported some analogous Schiff's base and azo crown ether derivatives **20** and **21**, in an attempt to get a mesomorphic crown ether but failed.

rigid enough; that mesomorphic properties were absent was understandable and reasonable. Nor did the following mono-armed Schiff's base crown ether **22** with the  $L/D$  ratio of 2:2 reported by He [5] show mesomorphic properties.



As we realized, the  $L/D$  values of the double-armed crown ethers **20** and **21** shown above did not exceed 4, and in addition, their molecular backbones were not

The double-armed crown ether compounds **14, 15, 17** and **18** we designed had  $L/D$  values greater than 4, and mono-armed compounds **16** and **19** were bigger than 3.

As a result, all of these were liquid crystals. This indicated that mono-armed liquid crystalline crown ethers could have smaller  $L/D$  values than the usual rod-shaped liquid crystal molecules (where  $L/D$  must be greater than 4), but still be larger than 3. Double-armed liquid crystalline crown ethers must comply with the  $L/D$  value requirement of usual rod-shaped liquid crystals. This conclusion is consistent with our previous results concerning the liquid crystals containing diaza-18-crown-6 and 15-crown-5 units [9, 10].

As indicated in the table, the Schiff's base liquid crystalline crown ethers **14**, **15** and **16** have higher mesomorphic temperatures than the azo liquid crystalline crown ethers **17**, **18** and **19**. The phenomenon is contrary to usual rod-shaped liquid crystals. This is probably due to the effect of the interplay of the oxygen atoms of the crown ether ring and the hydrogen atom of the linkage  $-\text{CH}=\text{N}-$ , which enhances the rigidity of liquid crystalline crown ether molecules.

As a result, smectic and nematic phases appeared in both such Schiff's base and azo mono-armed liquid crystalline crown ethers; only a nematic phase was observed in the above mentioned double-armed liquid

crystalline crown ethers. This is probably due to the fact that the double-armed liquid crystalline crown ethers are of molecular structural symmetry, which leads to a stronger terminal intermolecular attraction than the lateral intermolecular attraction.

### 3. Experimental

#### 3.1. Instruments

IR: Nicolet FT-IR 170SX infrared spectrometer. NMR: JNM-FX90Q NMR spectrometer. MS: Finnigan-Mat 4510(EI) GC-MS and Hitachi M-80AG-MS(FD). Elementary analysis: Carlo Erba 1106. DSC: XPT-7 microscope with polariscope made by the Jiangnan Optical Instrument Factory (with an attached heating plate produced by the Scientific Instrument Plant of Sichuan University). Reagents: All reagents used were chemical purity and analytical grade.

Table. Phase transition temperatures<sup>a</sup> and  $L/D$  values of the liquid crystalline crown ethers.

Compound	Phase transition temperatures/ <sup>o</sup> C	$L/D$
14	Cr 245 S 302 I	6.7:1
15	Cr 241 N 350 I	6.8:1
16	Cr 229.1 S 257.3 N 310.4 I	3.7:1
17	Cr 88.4 N 192.3 I	6.7:1
18	Cr 91.6 N 192.0 I	6.8:1
19	Cr 75.7 S 173.4 N 229.4 I	3.7:1
2	Cr 102.2 N 137.5 I	
5	Cr 88.9 S <sub>1</sub> 97.6 S <sub>2</sub> 109.6 I	

<sup>a</sup>Cr, crystal; S, smectic; N, nematic; M, mesomorphic; I, isotropic.

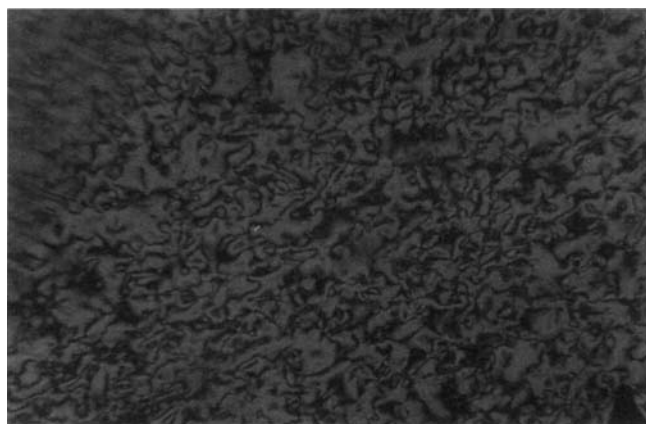


Figure 2. Texture of **15**,  $T=299^{\circ}\text{C}$ ,  $\times 140$ .

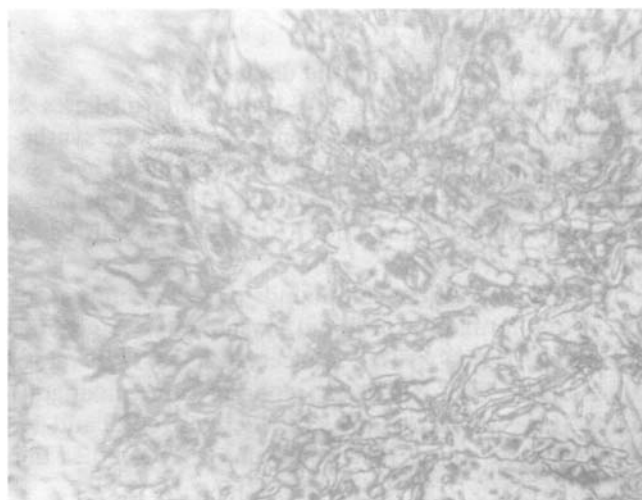


Figure 3. Texture of **16**,  $T=276^{\circ}\text{C}$ ,  $\times 120$ .

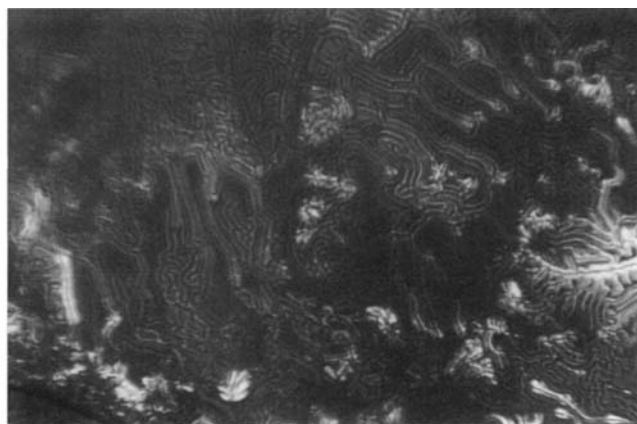


Figure 4. Texture of **17**,  $T=210^{\circ}\text{C}$ ,  $\times 140$ .



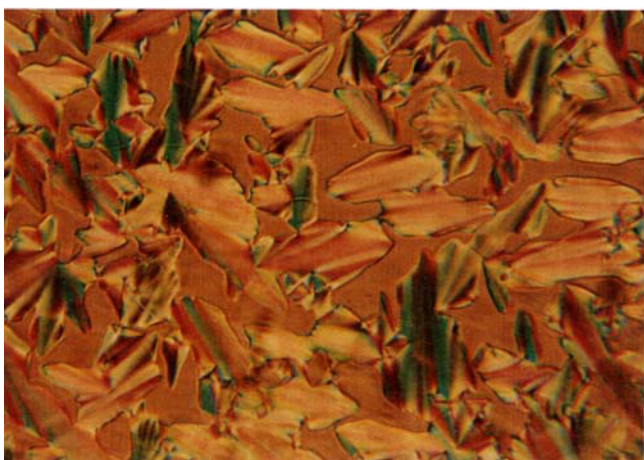


Figure 1. Texture of **14**,  $T=258^{\circ}\text{C}$ ,  $\times 130$ .

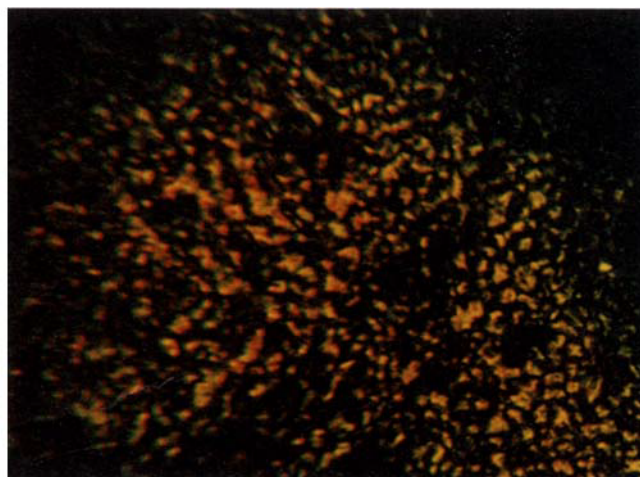


Figure 5. Texture of **19**,  $T=110^{\circ}\text{C}$ ,  $\times 130$ .

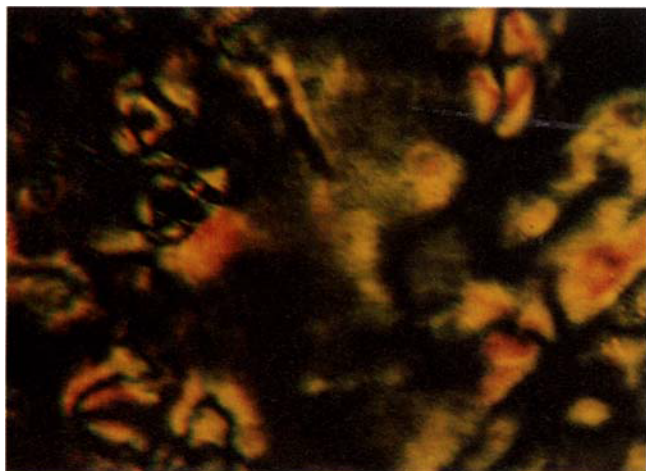


Figure 6. Texture of **19**,  $T=180^{\circ}\text{C}$ ,  $\times 130$ .



Figure 7. Texture of **5**,  $T=96^{\circ}\text{C}$ ,  $\times 150$ .

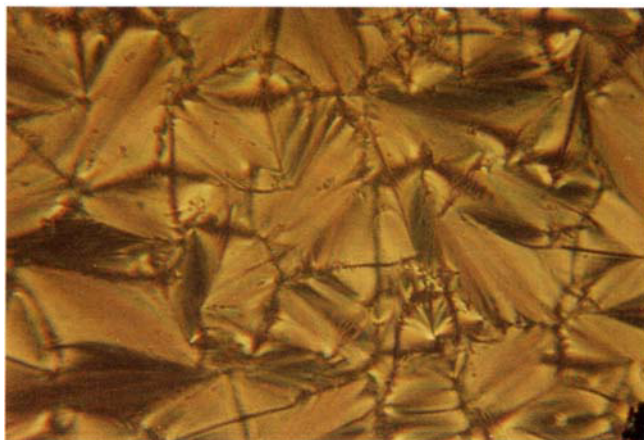


Figure 8. Texture of **5**,  $T=107^{\circ}\text{C}$ ,  $\times 150$ .



Figure 9. Texture of **2**,  $T=129^{\circ}\text{C}$ ,  $\times 150$ .

## 3.2. Synthesis of intermediates

## 3.2.1. 4-Heptoxybiphenyl (1)

For detailed synthesis see [12].

## 3.2.2. 4-Acetyl-4'-heptoxybiphenyl (2)

30.0 g (0.112 mol) **1** and 170 ml CS<sub>2</sub> were put into a 250 ml three-necked flask equipped with a condenser, a CaCl<sub>2</sub> drying tube and a magnetic stirrer. While stirring, 17.0 g (0.128 mol) anhydrous AlCl<sub>3</sub> was quickly added. 9.0 g (0.115 mol) acetyl chloride was added dropwise during reflux, and then stirred at reflux for a further 45 min. After cooling the mixture was poured into 18 ml of concentrated HCl and 120 ml iced water. This was stirred for 30 min the organic layer separated and washed with 150 ml (50 ml × 3) water, then dried over anhydrous sodium sulphate. The solvent was evaporated to yield a yellowish sticky crude product. 40 ml ether was added, stirred, filtered, and the unsolved solid recrystallized from isopropanol to leave white leaflike crystals 10.5 g (30.2 per cent). DSC: *T*<sub>CrN</sub> 102.2, *T*<sub>NI</sub> 137.5°C; IR (KBr): 1676 (s, C=O), 1602, 1526 (s, m, Ar), 1271 (s, Ar-O-C) cm<sup>-1</sup>. <sup>1</sup>H NMR (CD<sub>3</sub>COCD<sub>3</sub>): 8.09–6.99 (8 H, m, 2 × ArH<sub>4</sub>), 4.13–3.99 (2 H, t, CH<sub>2</sub>O), 2.60 (3 H, s, CH<sub>3</sub>CO), 1.81–1.38 (10 H, m, 5 × CH<sub>2</sub>), 0.9 (3 H, t, CH<sub>3</sub>) ppm. *m/z* (EIMS): 311 ([M + H]<sup>+</sup>, 20 per cent), 310 (M<sup>+</sup>, 91 per cent), 197 ([HOC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>CO]<sup>+</sup>, 100 per cent). Elementary analysis, calcd. (C<sub>21</sub>H<sub>26</sub>O<sub>2</sub>): C, 81.25; H, 8.44. Found: C, 81.69; H, 8.34.

## 3.2.3. 4'-Heptoxybiphenyl-4-carboxylic acid (3)

For a detailed synthesis see [13].

## 3.2.4. 4-[4'-Heptoxybiphenyl-4-carboxyl]benzaldehyde (5)

Into 50 ml three-necked flask equipped with a condenser and magnetic stirrer, 1.09 g (8.9 mmol) 4-hydroxybenzaldehyde, 8 ml dichloromethane and 1.3 ml triethylamine were added. The solution of 2.95 g (8.9 mmol) of 4'-heptoxybiphenyl-4-carbonyl chloride in 7.5 ml dichloromethane was added dropwise under a N<sub>2</sub> atmosphere. Stirring was continued for 3.5 h. After cooling a white precipitate formed. 70 ml dichloromethane were added. The resulting solution was washed with 10 ml water, 10 ml 5 per cent sodium hydroxide solution, 20 ml (10 ml × 2) water and dried over anhydrous sodium sulphate. The solvent was removed to yield a yellowish crude product 3.20 g (86.3 per cent). This was purified by recrystallization from alcohol. DSC: *T*<sub>CrM<sub>1</sub></sub> 88.9, *T*<sub>M<sub>1</sub>M<sub>2</sub></sub> 97.6, *T*<sub>M<sub>2</sub>I</sub> 109.6°C. IR (KBr): 1732, 1704 (s, C=O), 1601, 1500 (s, m, Ar), 1271, 1081 (s, Ar-O-C) cm<sup>-1</sup>. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 10.03 (1 H, s, CHO), 8.28–6.95 (12 H, m, 3 × ArH<sub>4</sub>), 4.09–3.95 (2 H, t, CH<sub>2</sub>O), 1.82–1.17 (10 H, m, 5 × CH<sub>2</sub>), 0.9 (3 H, t, CH<sub>3</sub>) ppm. *m/z* (EIMS): 416 (M<sup>+</sup>, 4 per cent), 295

([C<sub>7</sub>H<sub>15</sub>OC<sub>6</sub>H<sub>4</sub>C<sub>6</sub>H<sub>4</sub>CO]<sup>+</sup>, 100 per cent). Elementary analysis, Calcd. (C<sub>27</sub>H<sub>28</sub>O<sub>4</sub>): C, 77.88; H, 6.73. Found: C, 77.90; H, 6.82.

## 3.2.5. 4,4'-Dinitrodibenzo-18-crown-6 (6,7)

Synthesis according to the procedures described in [14].

## 3.2.6. 4,4'-Diaminodibenzo-18-crown-6 (8, 9)

Synthesis according to reference [15].

## 3.2.7. 4-Aminobenzo-15-crown-5 (10)

Synthesis according to literature [16].

## 3.2.8. 4,4'-bis(4-Hydroxyphenylazo)dibenzo-18-crown-6 (11, 12)

Synthesis according to literature [17].

## 3.2.9. 4-(4-Hydroxyphenylazo)benzo-15-crown-5 (13)

Synthesis according to literature [16].

## 3.3. Synthesis of liquid crystalline crown ethers

3.3.1. *cis*-4,4'-bis[(4'-heptoxybiphenyl-4-carboxyl)benzylideneamino]dibenzo-18-crown-6 (14)

Into a 100 ml three-necked flask equipped with a condenser and magnetic stirrer, 0.27 g (0.69 mmol) **8**, 0.69 g (1.63 mmol) **5** and 20 ml alcohol were placed. The mixture was heated to reflux under an atmosphere of nitrogen for 4 h during which time a yellow precipitate appeared. The mixture was cooled and filtered. The crude product was recrystallized from chloroform and DMF (1:1) to yield a yellowish powder 0.3 g (36.6 per cent). DSC: *T*<sub>CrN</sub> 245, *T*<sub>NI</sub> 302°C. IR (KBr): 1729 (s, C=O), 1261 (w, C=N). 1608, 1511 (s, Ar), 1262, 1071 (s, Ar-O-O), 1136 (s, O-O-O) cm<sup>-1</sup>. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 8.49–6.87 (32 H, m, 6 × ArH<sub>4</sub> + 2 × ArH<sub>3</sub> + 2 × CH=N), 4.21–3.93 (20 H, m, 10 × CH<sub>2</sub>O), 1.81–1.36 (20 H, m, 10 × CH<sub>2</sub>), 0.91 (6 H, t, 2 × CH<sub>3</sub>) ppm. *m/z* (FDMS): 1210 ([M + Na]<sup>+</sup>, 100 per cent), 1187 ([M + H]<sup>+</sup>, 28 per cent), 1186 (M<sup>+</sup>, 20 per cent). Elementary analysis, Calcd. (C<sub>74</sub>H<sub>78</sub>N<sub>2</sub>O<sub>12</sub>): C, 74.85; H, 6.62; N, 2.36. Found: C, 74.33; H, 6.66; N, 2.40.

3.3.2. *trans*-4,4'-bis[(4'-heptoxybiphenyl-4-carboxyl)benzylideneamino]dibenzo-18-crown-6 (15)

The procedure was similar to that given for **14**. Yield, 80.3 per cent. DSC: *T*<sub>CrN</sub> 241, *T*<sub>NI</sub> 265°C. IR (KBr): 1732 (s, C=O), 1621 (w, C=N), 1602, 1510 (s, Ar), 1264, 1072 (s, Ar-O-C), 1133 (s, C-O-C) cm<sup>-1</sup>. <sup>1</sup>H NMR (CDCl<sub>3</sub>): 8.50–6.88 (32 H, m, 6 × ArH<sub>4</sub> + 2 × ArH<sub>3</sub> + 2 × CH=N), 4.25–3.95 (20 H, m, 10 × CH<sub>2</sub>O), 1.80–1.25 (20 H, m, 10 × CH<sub>2</sub>), 0.91 (6 H, t, 2 × CH<sub>3</sub>) ppm. *m/z* (FDMS): 1211 ([M + 2H + Na]<sup>+</sup>, 39 per cent), 1188 ([M + 2H]<sup>+</sup>, 73 per cent), 1187 ([M + H]<sup>+</sup>, 100 per cent), 1185

( $[M - H]^+$ , 74 per cent). Elementary analysis, Calcd. ( $C_{74}H_{78}N_2O_{12}$ ): C, 74.85; H, 6.62; N, 2.36. Found: C, 74.43; H, 6.698; N, 2.34.

### 3.3.3. 4-(4'-Heptoxybiphenyl-4-carboxylbenzylideneamino)benzo-15-crown-5 (16)

The procedure was similar to that given for 14. Yield, 41.3 per cent. DSC:  $T_{CrS}$  229.1,  $T_{SN}$  257.3,  $T_{NI}$  310.4°C. IR (KBr): 1736 (s, C=O), 1654 (s, C=N), 1603, 1497 (s, Ar), 1273, 1084 (s, m, Ar-O-C), 1162 (m, C-O-C)  $cm^{-1}$ .  $m/z$  (EIMS), 295 ( $[C_7H_{15}OC_6H_4C_6H_4CO]^+$ , 100 per cent), 197 ( $[C_6H_5C_6H_4CO_2]^+$ , 20 per cent). Elementary analysis, Calcd. ( $C_{41}H_{47}NO_8$ ): C, 72.5; H, 6.90; N, 2.06. Found: C, 72.45; H, 6.66; N, 2.00.

### 3.3.4. cis-4,4'-bis(4'-Heptoxybiphenyl-4-carboxylphenylazo)dibenzo-18-crown-6 (17)

Into a 100 ml three-necked flask, 0.58 g (0.97 mmol) 11, 30 ml pyridine and 4 (obtained by 0.69 g 3 reacted with thionyl chloride) were added. This was stirred for 5 h under a nitrogen atmosphere at room temperature. The temperature was raised to 75°C and stirred for another hour. The mixture was cooled and the brown liquid poured into 120 ml cold water to yield a precipitate. This was filtered, washed with water to obtain the crude orange product 1.12 g (97.1 per cent). The product was purified by column chromatography (silica gel H), using ether and methanol (V/V 1:1) as eluant to leave an orange powder. DSC:  $T_{CrN}$  88.4,  $T_{NI}$  192.3°C. IR (KBr): 1695 (s, C=O), 1600, 1508 (s, Ar), 1256, 1059 (s, m, Ar-O-C), 1133 (s, C-O-C)  $cm^{-1}$ .  $^1H$  NMR ( $d_6$ -DMSO): 8.03–6.86 (30 H, m, 6 × ArH<sub>4</sub> + 2 × ArH<sub>3</sub>), 4.16–3.93 (20 H, m, 10 × CH<sub>2</sub>O), 1.71–1.32 (20 H, m, 10 × CH<sub>2</sub>), 0.87 (6 H, t, 2 × CH<sub>3</sub>) ppm.

### 3.3.5. trans-4,4'-bis(4'-Heptoxybiphenyl-4-carboxylphenylazo)dibenzo-18-crown-6 (18)

Prepared by the same procedure as with 17, Yield, 95.0 per cent. Column chromatography (silica gel H), using ether and chloroform (V/V 10:1) as eluant to obtain an orange powder. DSC:  $T_{CrN}$  91.6,  $T_{NI}$  192.0°C. IR (KBr): 1686 (s, C=O), 1603, 1508 (s, Ar), 1256, 1061 (s, Ar-O-C), 1131 (s, C-O-C)  $cm^{-1}$ .  $^1H$  NMR ( $d_6$ -DMSO): 7.94–6.86 (30 H, m, 6 × ArH<sub>4</sub> + 2 × ArH<sub>3</sub>), 4.16–3.93 (20 H, m, 10 × CH<sub>2</sub>), 1.75–1.23 (20 H, m, 10 × CH<sub>2</sub>), 0.87 (6 H, t, 2 × CH<sub>3</sub>) ppm.

### 3.3.6. 4-(4'-Heptoxybiphenyl-4-carboxylphenylazo)benzo-15-crown-5 (19)

Prepared in the same way as 17. Yield, 85.0 per cent. Column chromatography (basic aluminium oxide), using chloroform and acetone (V/V 15:1) as eluant to obtain a yellow powder. DSC:  $T_{CrS}$  75.7,  $T_{SN}$  173.4,  $T_{NI}$  229.4°C. IR (KBr): 1725 (s, C=O), 1575, 1490 (s, Ar), 1256, 1061 (s, Ar-O-C), 1131 (s, O-C-O)  $cm^{-1}$ .  $^1H$  NMR (CDCl<sub>3</sub>): 8.26–6.79 (15 H, m, 3 × ArH<sub>4</sub> + ArH<sub>3</sub>), 4.10–3.68 (16 H, m, 8 × CH<sub>2</sub>O), 2.05–1.26 (12 H, m, 6 × CH<sub>2</sub>), 0.81 (3 H, t, CH<sub>3</sub>) ppm.  $m/z$  (EIMS): 295 ( $[C_7H_{15}OC_6H_4C_6H_4CO]^+$ , 100 per cent), 183 ( $[aminobenzo-15-crown-5]^+$ , 40 per cent), 214 ( $[O_2OC_6H_4C_{16}H_4O]^+$ , 70 per cent).

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