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Synthesis and Mesomorphic Properties of Novel Crown Ether Schiff Bases

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Different crown ether Schiff bases were prepared by condensing a different aldehyde with trans-diamino dibenzo 18-crown-6. The physical properties as well as the chemical formulations of these compounds were derived from microanalysis and spectroscopic methods. Phase transition temperatures and the thermal parameters were obtained from differential scanning calorimetry (DSC). The texture observation was performed under polarizing optical microscopy (POM) attached with a hot stage. Most of the compounds are thermotropic liquid crystals. Different phases such as smectic, (SmC, SmA) and nematic (N) were shown by crown ether Schiff bases containing azomethine and hydroxy group which are characteristic of rodlike liquid crystals.

Keywords: dibenzo 18-crown-6; liquid crystal crown ether (LCCE) Schiff base; nematic; smectic; thermotropic liquid crystal

INTRODUCTION

Recently, much interest has been generated in the application of new liquid crystalline materials in the field of chiral recognization, ion transportation, function membranes, transferring signal, and information at the molecular level [1,2]. Liquid crystals are also applied for photonics applications [3], as surfactants [4], elastomers [5], in electro-optic devices [6], video projectors, optical computing, nonlinear optics [7], thermographic measurements, temperature and pressure sensors, spectroscopy and chromatography [8, 9], batteries and sensor devices [10, 11], and building blocks [12].

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Percec et al. [13] have demonstrated that macrocyclization overrides the well-established polymer effect in the formation and stabilization of liquid crystalline (LC) phases. Therefore, contrary to what has been considered for over 100 years, it was proved that the cyclic and not the linear architecture is the most powerful in the design of molecular and macromolecular LCs. Liquid crystal crown ethers (LCCE) have potential applications because of their physicochemical properties and the utilization of crown ethers as selective ionophoric units in other functionalized compounds.

Predominantly nematic phases and also smectic layer structures were obtained from calamitic compounds with a crown ether moiety at one of the termini of an extended, rod-like, rigid core [14–18]. It was found that the isotropization temperatures of low molecular mass liquid crystals containing crown ethers decrease upon complexation with alkali metal salts [19], whereas smectic mesophases of polymeric liquid crystal crown ethers can be stabilized by complexation [17, 20]. On the other hand, columnar mesophases were induced by the interaction of heavy metal cations with multiarmed azacrowns [21].

Only a few LCCE Schiff bases were reported [22–26] which showed the smectic and nematic phases. The threaded texture and schlieren texture were observed in Schiff base type polymer LCCEs [27–28].

In this article, we report some crown ether Schiff bases prepared from various aldehydes (salicylaldehyde, 5-bromo salicylaldehyde, 2,3-dihydroxy benzaldehyde, 2-hydroxy naphthaldehyde, Thiophene 2-carbaldehyde, Pyrrole 2-carbaldehyde, o-vanillin, 3,4,5-trimethoxy benzaldehyde) condensed with *trans*-diamino dibenzo-18-crown-6.

All compounds thus isolated were purified and characterized by Fourier Transform Infrared Spectroscopy (FTIR), ¹H NMR, FAB-MS along with thermal analysis and texture observation using Differential Scanning Calorimetry (DSC) and Polarizing Optical Microscopy (POM), respectively. Effects of the changes in the polar substituents in aldehyde on the mesophases are also discussed.

EXPERIMENTAL

Reagents and Chemicals

All the chemicals used were of A.R. grades of E. Merck, or Aldrich unless otherwise specified.

Apparatus

The melting points were taken in a sealed capillary tube using Toshniwal India melting point apparatus and are uncorrected. The FT-IR spectra were recorded on JASCO-410 FT-IR Spectrometer as KBr pellets. Electronic spectra were recorded on JASCO V-530 double beam UV-Vis spectrophotometer in chloroform.

Transition temperatures and enthalpies were scanned in Shimadzu DSC 60, differential scanning calorimeter with a heating rate of 10.0°C min⁻¹ in air and it was calibrated with indium (156.6°C , $28.45\,\text{Jg}^{-1}$). The temperatures were read as the maximum of the endothermic peaks.

The textures of the mesophases were studied with Leica DMLP polarizing microscope, equipped with a hot stage, Mavotherm 32 temperature display unit, and Leica MPS 32 Data back (display unit). The samples were investigated using cross polarizers at room temperature with a magnification of 2.5X and a magnification 10X or 20X was at elevated temperature. The samples were mounted on quartz slides of $26 \times 26 \,\mathrm{mm}$ with $1 \,\mathrm{mm}$ thickness. The film used was Kodak Max $400, 24 \times 36 \,\mathrm{mm}, 35 \,\mathrm{mm}$ color print.

Procedure: The substance to be examined is heated up to the isotropic liquid point on a microscopic slide and a cover slip is pushed into the melt in such a way that thin layer is formed between the slide and cover slip. The slide was cooled thoroughly and placed on the heating stage of the polarizing microscope. The temperature initially was raised rapidly (5°C min⁻¹) and the slide was then recooled. Afterwards the heating was then continued at a rate of 1°C min⁻¹ and cooling too was carried out at the same rate. The mesomorphic transition temperatures and the textures under the microscope were recorded for the compounds.

Synthesis

Dibenzo 18-crown-6 was synthesized by earlier reported method [29]. Nitration of dibenzo 18-crown-6 was also done by reported method [30].

Preparation of *trans*-diamino Dibenzo 18-Crown-6 (t-DADBC)

A mixture of 12.0 g (0.026 mol) *trans*-dinitro dibenzo 18-crown-6, 4 g Raney Nickel catalyst, and 300 mL ethanol was refluxed in a liter flask and added 20 mL hydrazine hydrate with stirring in about 20 min at a rate so as to maintain a vigorous reaction. The reaction mixture was refluxed until the effervescence ceased and continued for further 15 min. The catalyst was filtered hot with suction and the filtrate on cooling in ice-bath deposited t-DADBC (Fig. 1). Yield 6.5 g (63.5%), mp 199–201°C, reported [30] 201–203°C.

FIGURE 1 Preparation of trans-diamino dibenzo 18-crown-6.

General Synthesis of Crown Ether Schiff Bases

A solution of trans-diamino dibenzo 18-crown-6 (10 mmol) and aldehyde (20 mmol) (Fig. 2) in anhydrous ethanol (100 mL) in the presence of anhydrous MgSO₄ were stirred for 4 h under N₂ atmosphere at 80°C,

$$H_2N$$
 H_2N
 H_2N
 H_2N
 H_2N
 H_3N
 H_3C
 H_3C

FIGURE 2 Synthesis of crown ether Schiff base containing DB18C6.

and then the mixture was cooled. The different yellow colored precipitate was filtered and washed with ethanol. After recrystallization from ethanol, yellow crystals were obtained.

Synthesis of N,N'-bis(2-hydroxy-3-methoxybenzidene)-4,4'-diamino-2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxacyclooctadeca-2,11-diene (1)

A solution of *trans*-diamino dibenzo 18-crown-6 (3.90 g, 10 mmol) and 2-hydroxy 3-methoxy benzaldehyde (3.04 g, 20 mmol) in anhydrous EtOH (100 mL) in the presence of anhydrous MgSO₄ were stirred for 4 h under N₂ atmosphere at 80°C, and then the mixture was cooled. The yellow precipitate was filtered and washed with EtOH. After recrystallization from EtOH, yellow crystals (yield 91%) were obtained. mp 122°C. ¹H-NMR δ (ppm): 10.20 (s, 1H, OH), 9.25 (s, 1H, N=CH), 8.16 6.75(m, 14H, ArH), 4.38 4.15 (m, 14H, OCH₂, NCH₂Ar); FT-IR (KBr,cm⁻¹) $\nu_{\rm max}$: 3445, 2930, 1588, 1506, 1235,1129; FAB MS (m/z): 660 (M+1) 682 (M+Na); (Found: C 65.64, H 5.81, N 4.25. $C_{36}H_{38}N_2O_{10}$ calcd.: C 65.67, H 5.78, N 4.25%).

Synthesis of N,N'-bis(3,4,5-trimethoxybenzidene)-4,4'-diamino-2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxacyclooctadeca-2,11-diene (2)

Compound **2** was prepared by following the general procedure as above by taking a solution of trans-diamino dibenzo 18-crown-6 (3.90 g, 10 mmol) and 3,4,5-trimethoxy benzaldehyde (3.92 g, 20 mmol). After recrystallization from, EtOH, yellow crystals (yield 82%) were obtained. mp 125–126°C. $^1\text{H-NMR}$ δ (ppm): 8.14 (s, 1H, N=CH), 7.70 6.48 (m, 14H, ArH), 3.84 3.65 (m, 14H, OCH₂, NCH₂Ar); FT-IR (KBr,cm $^{-1}$)v_{max}: 2884, 1588, 1260, 1130; FAB MS (m/z): 770 (M+Na); (Found: C 64.33, H 6.21, N 3.75. $\text{C}_{40}\text{H}_{46}\text{N}_2\text{O}_{12}$ calcd.: C 64.30, H 6.20, N 3.80%).

Synthesis of N,N'-bis-thiophene-2-carbalidiene-4,4'-diamino(2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxa cycloocta deca-2,11-diene (3)

Compound **3** was prepared by taking a solution of *trans*-diamino dibenzo 18-crown-6 (3.90 g, 10 mmol) and thiophene-2-carbaldehyde (2.24 g, 20 mmol). After recrystallization from EtOH, yellow crystals (yield 84%) were obtained. Mp 187–189°C. $^1\text{H-NMR}$ δ (ppm): 8.34 (s,1H, N=CH), 8.43(s, 1H, N=CH), 7.39 6.42 (m, 14H, ArH), 4.23 4.04 (m, 14H, OCH₂, NCH₂Ar); FT-IR (KBr,cm $^{-1}$) ν_{max} : 2928, 1588, 1246, 1134; FAB MS (m/z): 579 (M+); (Found: C 62.28, H 5.88, N 4.84. $C_{30}H_{30}N_2O_6S_2$ calcd.: C 62.30, H 5.85, N 4.79%).

Synthesis of N,N'-bis-pyrrole-2-carbalidiene-4,4'-diamino(2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxa cycloocta deca-2,11-diene (4)

By taking a solution of *trans*-diamino dibenzo 18-crown-6 (3.90 g, 10 mmol) and pyrrole-2-carbaldehyde (1.90 g, 20 mmol) compound 4 was prepared. After recrystallization from, EtOH, yellow crystals (yield 76%) were obtained. Mp 170–172°C. ¹H-NMR δ (ppm): 8.95 (s,1H,N=CH), 7.94 6.90(m, 14H, ArH), 4.23 4.04 (m, 14H, OCH₂, NCH₂Ar); FT-IR (KBr,cm⁻¹) $v_{\rm max}$: 2932, 2861, 1612, 1508, 1254,1127; FAB MS (m/z): 568 (M+Na); (Found: C 66.16, H 5.92, N 10.29. $C_{30}H_{32}N_4O_6$ calcd.: C 66.05, H 6.00, N 10.35%).

Synthesis of N,N'-bis-salicylidiene-4,4'-diamino(2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxa cycloocta deca-2,11-diene (5)

In the case of compound **5** a solution of *trans*-diamino dibenzo 18-crown-6 (3.90 g, 10 mmol) and salicylaldehyde (2.44 g, 20 mmol) were reacted as above method. After recrystallization from EtOH, yellow crystals (yield 92%) were obtained. Mp 212–214°C. H-NMR δ (ppm): 10.97 (s, 1H, OH), 9.12 (s, 1H, N=CH), 8.02 6.93 (m, 14H, ArH), 4.23 4.06 (m, 14H, OCH₂, NCH₂Ar); FT-IR (KBr,cm⁻¹) $v_{\rm max}$: 3410, 1595, 1513, 1227, 1049; FAB MS (m/z): 600 (M+); (Found: C 68.29, H 5.65, N 4.64. $C_{34}H_{32}N_2O_8$ calcd.: C 68.26, H 5.67, N 4.62%).

Synthesis of N,N'-bis-2-hydroxy naphthalidiene-4,4'-diamino(2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxa cycloocta deca-2,11-diene (6)

Compound **6** was prepared by taking a solution of trans diamino dibenzo 18-crown-6 (3.90 g, 10 mmol) and 2-hydroxy naphthaldehyde (3.44 g, 20 mmol). After recrystallization from EtOH, yellow crystals (yield 83%) were obtained. Mp 226–228°C. ¹H-NMR δ (ppm): 10.03 (s, 1H, OH), 8.55 (s, 1H, N=CH), 8.06 6.90 (m, 14H, ArH), 4.23 4.06 (m, 14H, OCH₂, NCH₂Ar); FT-IR (KBr,cm⁻¹) ν_{max} : 3414, 1591, 1508, 1279, 1132, 1060; FAB MS (m/z): 698 (M); (Found: C 72.22, H 5.41, N 4.03. C₄₂H₃₈N₂O₈ calcd.: C 72.20, H 5.45, N 4.01%).

Synthesis of N,N'-bis-5-bromosalicylidiene-4,4'-diamino(2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxa cycloocta deca-2,11-diene (7)

Compound **7** was prepared by the same procedure as given above by taking a solution of *trans*-diamino dibenzo 18-crown-6 (3.90 g,10 mmol) and 5-bromo salicylaldehyde (4.02 g, 20 mmol) After recrystallization from EtOH, yellow crystals (yield 79%) were obtained. Mp 198–200°C. ¹H-NMR δ (ppm): 11.97 (s, 1H, OH), 9.89 (s, 1H, OH), 8.60

(s, 1H, N=CH), 8.43 (s, 1H, N=CH), 7.78 6.56 (m, 14H, ArH), 4.16 3.73 (m, 14H, OCH₂, NCH₂Ar), 2.86 (t, J = 5.3 Hz, 4H, NCH₂); FT-IR (KBr,cm⁻¹) $v_{\rm max}$: 3354, 1595, 1507, 1252, 1124, 1051; FAB MS (m/z): 756(M+); (Found: C 54.01, H 4.20, N 3.70, Br 21.15. $C_{34}H_{32}N_2O_8Br_2$ calcd.: C 53.96, H 4.23, N 3.70, Br 21.16%).

Synthesis of N,N'-bis-2,3-dihydroxybenzalidiene-4,4'-diamino(2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxa cycloocta deca-2,11-diene (8)

Compound **8** was prepared by taking a solution of *trans*-diamino dibenzo 18-crown -6 (3.90 g, 10 mmol) and 2,3-dihydroxy benzaldehyde (2.76 g, 20 mmol). After recrystallization from EtOH, yellow crystals (yield 81%) were obtained. Mp 187–189°C. ¹H-NMR δ (ppm): 13.24 (s, 1H, OH), 10.92 (s, 1H, OH), 9.84 (s, 1H, N=CH), 7.60 6.70 (m, 14H, ArH), 4.23 4.04 (m, 14H, OCH₂, NCH₂Ar); FT-IR (KBr,cm⁻¹) ν_{max} : 3445, 1588, 1506, 1235, 1129; FAB MS (m/z): 631 (M+1); (Found: C 64.75, H 5.4, N 4.46. $C_{34}H_{34}N_2O_{10}$ calcd.: C 64.76, H 5.39, N 4.44%).

RESULTS AND DISCUSSION

Physical Characterization

All the crown ether Schiff bases thus obtained are found to be thermally stable. The spectroscopic methods (FTIR, ¹H NMR and FAB-MS) have been employed to elucidate the structures of the compounds (1–8). FTIR data show that the diagnostic bonds, which can be assigned to the stretching of imine (C=N) of all compounds were observed at the frequencies of 1588 to $1622\,\mathrm{cm}^{-1}$. The bands appearing at 3354 to $3450\,\mathrm{cm}^{-1}$ in the spectra of compounds except 2, 3, and 4 can be ascribed to the presence of hydroxy (–OH) group. A band assigned to the stretching of C–O–C of the ether linkage is also observed in the FTIR data of all compounds.

 1 H NMR data of all the compounds showed that a peak due to the presence of azomethine proton (CH=N) was observable at the chemical shift $\delta=8.2$ to 9.0 ppm. While the multiplet owing to the presence of aromatic protons was observed within the range of $\delta=6.4$ to 8.0 ppm, the phenolic protons was observable within the range of $\delta=10.0$ to 11.0 ppm. The appearance of a peak within the range of $\delta=3.5$ to 4.3 is due to the presence of the proton of ether $(-OCH_2)$ group.

The formulations and molecular structure of compounds **1–8** in Figure 2 are also supported by the data obtained from FAB-MS and microanalysis, wherein the percentage of C, H, and N from the analysis conform the calculated values.

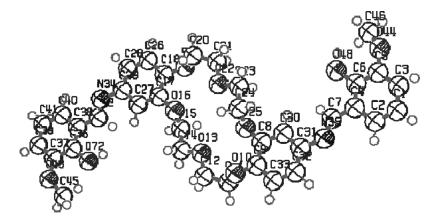


FIGURE 3 ORTEP-3 drawing of compound N,N'-bis(2-hydroxy-3-methoxy-benzidene)-4,4'-diamino-2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxacycloocta deca-2,11-diene (1).

ORTEP-3 drawing of compound 1 and 2 are given in Figures 3 and 4, respectively.

Phase Transition Behavior and LCCE Schiff Bases

All the newly synthesized crown ether Schiff bases 1–8 were studied for the liquid crystalline properties. Melting points of selected crown ether Schiff bases given in Table 1 were taken in open capillaries for approximate evaluation of the transition temperatures. The transition

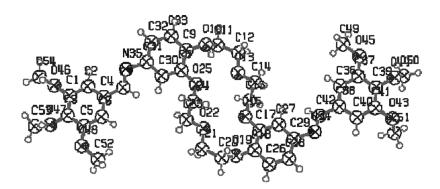


FIGURE 4 ORTEP-3 drawing of compound N,N'-bis(3,4,5-trimethoxybenzidene)-4,4'-diamino-2,3,11,12-dibenzo-1,4,7,10,13,16-hexaoxacycloocta deca-2,11-diene (2).

Compound no.	Compound name	${ m Mp}^{\circ}{ m C}$	*cp°C
1	5	212	_
2	6	227	_
3	7	198	_
4	8	187	_
5	3	154	196
6	4	170	208
7	1	121	245
8	2	125	248

TABLE 1 Capillary Data of Crown Ether Schiff Bases

from the solid state to the first mesophase is considered as the melting point. Heating the compound in a capillary tube showed viscous turbid melt sticking to the walls of the tube, followed by a turbid mobile state that ultimately yielded the isotropic liquid state. The thermal and liquid crystal behavior of these compounds were investigated by the combination of DSC and POM.

Transition temperatures and thermal behavior of crown ether Schiff bases recorded under the POM and DSC are shown in Table 2. The results show that most of the compounds are mesomorphic in

TABLE 2 Thermal Transition Data of Compounds **1–8** Upon Cooling Process and Corresponding Enthalpy Changes $(J\,g^{-1})$

Compound	Transition	$Temperature \ (^{\circ}C)$	$\Delta T/^{\circ}C$	$\Delta H \; (J g^{-1})$
1	Cr-SmC	122.62		2.11
	SmC-SmA	153.07	30.45	35.90
	SmA-N	225.05	71.98	4.07
	N-I	243.99	18.94	1.63
2	Cr-SmA	91.89		15.20
	SmA-N	131.51	71.14	9.64
	N-I	300.17	50.76	11.78
3	Cr-SmC	154.7		5.04
	SmC-I	196.4	41.7	23.80
4	Cr-N	171.2		16.46
	N-I	209.6	38.4	2.72
5	Cr-I	212.3	_	_
6	Cr-I	227.2	_	_
7	Cr-I	198.6	_	_
8	Cr-I	187.5	_	_

Cr - crystal; SmA - smectic A; SmC - smectic C; I - isotropic.

^{*}cp = clearing point.

character. The mesophases were identified from their optical textures. Focal conic fan texture of smectic phase and schlieren nematic phase are easily distinguishable from their typical textures and features. Both smectic and nematic mesophases were observed in most of the compounds. It has been observed that the crystal-to-smectic and smectic-to-nematic transitions overlapped in some cases.

Compound 1 shows SmC phase at 122°C and SmA mesophase at 153°C (Fig. 5a), which on further heating at 225°C transforms into nematic (Fig. 5c) and finally at 244°C (Fig. 5d) into isotropic phase. The textures are taken in cooling condition. After cooling nematic phase gave schlieren and broken focal-conic fan texture of the SmC phase and focal-conic fan texture of the SmA Phase.

Compound **2** shows smectic mesophase at 92°C (Fig. 6a), which on further heating at 131°C transforms into nematic phase (Fig. 6b). After cooling the Schlieren nematic phase the compound gave focal-conic fan texture of the SmA phase.

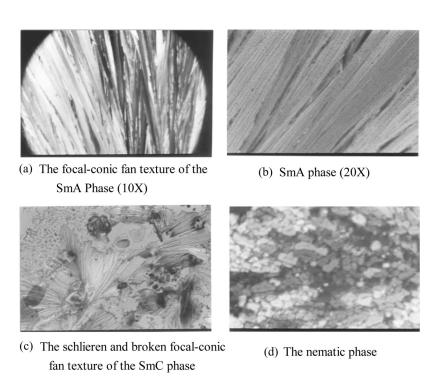
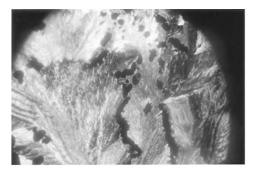
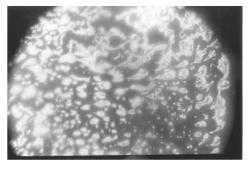


FIGURE 5 POM study of the liquid crystals textures of compound 1.



(a) The focal-conic fan texture of the SmA



(b) The schlieren Nematic phase

FIGURE 6 POM study of the liquid crystals textures of compound **2**.

Compound **3** also shows smectic phaseat 154.7°C which may be due to the presence of the thiophene ring in a lateral position within the structure of the compound [31].

Compound 4 shows Nematic phase at 171.2°C. Compound 5 to 8 does not exhibit any mesophases. Presumably this compound is too rigid to display mesomorphic behavior as it does not have a terminal alkoxy or alkyl group.

CONCLUSION

By substituting the parent dibenzo-18-crown-6 with mesogenic units (e.g., cyano, nitro, amino, alkoxy, olefin, azomethine, nitrone, azoxy, azo, ester, etc.) newer compounds with greater mesomorphic range can be designed and hence in the present work molecules have been

synthesized by attaching azomethine and methoxy units to the crown ether base. Most of the compounds exhibit liquid crystalline behavior on the basis of results obtained from POM and DSC. The relatively higher transition temperature in 1 is perhaps due to the presence of an additional Schiff base unit and structural rigidity. Micrographs show birefringence due to the mesomorphic nature of the compounds. The order of the phase transition: Spherulitic texture—Smectic texture—Nematic texture—Isotropic Texture.

Compounds 1 to 4 show liquid crystalline behavior at higher transition temperature which can be minimized by substituting long ester chain or by branched alkanes. LCCE Schiff bases can be utilized as one of the indicative tools for the complexation study as well as for the applying both the properties of crown ether and liquid crystal. The compounds are showing focal-conic fan texture of SmA phase, schlieren, and broken focal-conic texture of SmC phase, mosaic texture of SmC phase threaded texture of the nematic phase, schlieren nematic phase, and mobile texture of the nematic phase.

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