Iron(III) metallomesogen of \([\text{N}_2\text{O}_2]\) donor Schiff base ligand containing 4-substituted alkoxy chains

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ABSTRACT
Two new square pyramidal iron(III)-complexes of ‘salen’-type Schiff base ligands containing 4-substituted long alkoxy arms on the aromatic rings, \([\text{Fe}(4-\text{C}_{16}\text{H}_{33}\text{O})_2\text{salcn})\text{Cl}\] and \([\text{Fe}(4-\text{C}_{16}\text{H}_{33}\text{O})_2\text{salophen})\text{Cl}\] \(\text{salcn} = \text{N,N’-cyclohexanebis(salicylideneiminato)}\) and \(\text{salophen} = \text{N,N’-phenylenebis(salicylideneiminato)}\), have been successfully synthesised, and their mesomorphic property investigated. The ligands and complexes were characterised by elemental analyses, UV–Vis, FT-IR, ESI–MS, \(^1\)H and \(^13\)C NMR (for ligands only). The phase behaviour of the iron(III) complexes were ascertained by differential scanning calorimetry, polarising optical microscopy and variable temperature PXRD study. Ligands are non-mesomorphic, however, mesomorphism got induced upon complexation with the iron(III) centre. X-ray diffraction study revealed a layer-like arrangement of the five coordinated mesomorphic iron(III) complexes. The mesophase is stable over a wide range of temperature. The density functional theory calculations were carried out using Gaussian 09 program at B3LYP level using unrestricted 6–31G (d, p) basis set to obtain the optimised geometry of the iron(III) complexes.

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1. Introduction
Multifunctional soft materials with organised molecular motifs exhibiting several different physical and chemical properties in a single material are a vibrant field for future technological applications. The design of such multifunctional materials with specific structure–property relationship is a challenging task. The synthesis of liquid crystal (LC) based on metal coordination with suitably designed
ligand provides an useful route for obtaining new multifunctional soft material combining anisotropic fluidity with those properties imparted by the metal component, such as optical, electronic and magnetic properties.[1–8] The coordination geometry of the complex determines the overall molecular shape and in turn the mesomorphic behaviour.[9–15] Factors such as substituent on the ligand structure and the type of spacer linkage also tend to influence the phase behaviour of such compounds. Due to their flexibility in coordination, unique geometrical disposition, easy functionalisation and chemical stability of the imine linkages, ‘salen’-type fragments have widely been used as building block for the design of metallomesogens.[16–29] Such ligands have also been exploited as efficient catalyst for a wide variety of chemical transformations [30–33] and for the design of nonlinear optical (NLO) materials.[34–36] Unlike most other d-block elements, mesomorphic Schiff base complexes of iron in either of its oxidation state are less known.[37–43] Furthermore, the combination of spin crossover and liquid crystalline properties of mesomorphic iron Schiff base compound renders them to be novel advanced materials.[39–43] Iron(III) complexes of Schiff base ligands are also important as synthetic models for magnetic materials.[44–46] catechol dioxygenase[47,48] and as catalyst for olefin epoxidation.[49] In this article we report the synthesis, characterisation and mesomorphic behaviour of iron(III) complexes of ‘salen’-type N$_2$O$_2$ donor Schiff base ligands containing 4-substituted long alkoxy chain on the side aromatic rings. The free ligands are devoid of mesomorphism; however on coordination with the metal centre, induction of mesomorphic character occurs due to conformational change in the ligands. The mesophases were stable for a fairly wide temperature range.

2. Experimental

2.1. Materials

All solvents were purified and dried using standard procedures. All the chemicals used were of analytical grade, obtained from commercial sources and used without further purification. Silica (60–120 mesh) from Spectrochem was used for chromatographic separation. Silica gel G (E-Merck, India) was used for thin-layer chromatography (TLC).

2.2. Physical measurement

The C, H and N analyses were carried out using PE2400 elemental analyzer. The $^1$H NMR spectra were recorded on a Bruker DPX-400 MHz spectrometer in CDCl$_3$ (chemical shift in $\delta$) solution with TMS as internal standard. UV–Vis absorption spectra of the compounds in CH$_2$Cl$_2$ were recorded on a Shimadzu UV-160PC spectrophotometer. Infrared spectra were recorded on a Perkin–Elmer L 120-000A spectrometer on KBr disc. The optical textures of the compounds were studied using a polarising microscope (Nikon optiphot-2-pol) attached with Instec hot and cold stage HCS302, with STC200 temperature controller of 0.1°C accuracy. The thermal behaviour of the compounds were studied using a Perkin–Elmer differential scanning calorimeter (DSC) Pyris-1 spectrometer with a heating or cooling rate of 5°C/min. Variable temperature powder X-ray diffraction (PXRD) study of the samples were carried out on a Bruker D8 Discover instrument using Cu Ka radiation.

2.3. Synthesis and analysis

2.3.1. Hexadecyloxy salicyldehyde

4-(Hexyloxy)salicyldehyde was prepared using a general method as reported in the literature.[50] 2,4-Dihydroxybenzaldehyde (10 cm$^3$, 1.38 g), KHCO$_3$ (10 cm$^3$, 1 g), KI (catalytic amount) and 1-bromohexadecane (10 mmol, 1.6 g) were mixed in 250 cm$^3$ of dry acetone. The mixture was heated under reflux for 36 h, and then filtered to separate any insoluble solids present. Dilute HCl was added slowly to the warm solution, and the organic part was extracted with chloroform (100 cm$^3$). The combined chloroform extract was concentrated to give a purple solid product. The solid was purified by column chromatography (silica gel) using a mixture of chloroform and hexane (v/v, 1/1) as eluent. Evaporation of the solvent resulted in a white solid.

2.3.2. N, N'-Bis [4-(4'-hexadecyloxy)salicylidene]trans-1,2-diaminocyclohexane (16-dch)

An ethanolic solution (15 mL) of 2-hydroxy-(4-hexadecyloxy)salicylaldehyde (0.73 g, 2 mmol) was added slowly to an ethanolic solution (10 mL) of trans-1,2-diaminocyclohexane (0.12 g, 1 mmol) followed by addition of few drops of glacial acetic acid. The mixture was heated to reflux for 3 h to yield a yellow product. The compound was collected by filtration and recrystallized from absolute ethanol. Yield 0.58 g (72%); ESI–MS: m/z = 802.5. C$_{52}$H$_{96}$N$_2$O$_4$: calcd. C 77.8, H 10.7, N 3.6; found C 77.6, H 10.6, N 3.6. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ = 13.38 (s, 1H, ph-OH), 8.18 (s, 1H, N = CH), 7.32 (d, $J$ = 8.2 Hz, 1H, H$_2$), 6.34 (d, $J$ = 6.4 Hz, 1H, H$_1$), 6.28 (d, $J$ = 7.8 Hz, 1H, H$_2$), 3.14 (q, $J$ = 4 Hz, 1H, H$^3$), 3.92 (t, $J$ = 5.4 Hz, 2H, –OCH$_2$), 1.62–1.85 (m, 4H, cyclohexyl), 1.54 (m, 2H,
cyclohexyl), 1.42 (m, 2H, cyclohexyl), 0.92 (t, J = 6.7 Hz, 6H, –CH3), 0.86 (m, –CH2 in side chain) ppm. IR (νmax cm⁻¹, KBr): 3348 (νOH), 2926 (νas(C=H)), 2860 (νs(C=H)), 1621 (νC = N), 1560 (νC = C), 1275 (ν (C=O), phenolic), 1152 (ν(C=N)), 756 (δ(C=H)).

2.3.3. N, N'-Bis[4-(4'-hexadecyloxy)salicylidene]-1,2-phenylenediamine (16-opd)

An ethanolic solution (15 mL) of 2-hydroxy-(4-hexadecyloxy)salicylaldehyde (0.11 g, 1 mmol) was added slowly to a solution (10 mL) of 1, 2-phenylenediamine (0.11 g, 1 mmol). The reaction mixture was heated to reflux in the presence of a few drops of glacial acetic acid as catalyst for 3 h to yield a yellow product. The product was filtered followed by recrystallisation from absolute ethanol. Yield 0.06 g (73%); ESI–MS: m/z = 796.5. C52H66N2O4%; calcd. C 78.4, H 10.1, N 3.5; found C 78.2, H 10.2, N 3.6. 1H NMR (400 MHz, CDCl3): δ = 13.08 (s, 1H, ph-OH), 8.41 (s, 1H, N = CH), 7.31 (d, J = 8.2 Hz, 1H, H'), 7.23 (d, J = 8.4 Hz, 1H, H'), 7.14 (d, J = 8.5 Hz, 1H, H'), 6.32 (d, J = 6.3 Hz, 1H, H'), 6.27 (d, J = 8.1 Hz, 1H, H'), 3.92 (t, J = 6.7 Hz, 2H, –OCH2), 0.94 (t, J = 6.7 Hz, 6H, –CH3), 0.87 (m, –CH2 in side chain) ppm. IR (νmax cm⁻¹, KBr): 3400 (νOH), 2932 (νas(C=H)), 2852 (νs(C=H)), 1618 (νC = N), 1565 (νC = C), 1280 (ν(C=O), phenolic), 1150 (ν(C=N)), 760 (δ(C=H)).

2.3.4. Synthesis of Iron(III) complexes

In a typical procedure, a methanolic solution of FeCl3.6H2O (0.03 g, 0.1 mmol) was added slowly to a solution of synthesised ligands, 16-dch (0.08 g, 0.1 mmol) or 16-opd (0.08 g, 0.1 mmol) in a minimum volume of absolute ethanol and refluxed for ca. 2 h. The volume of the reaction mixture was reduced to one-third, whereupon a brown colour solid precipitated out. The product was isolated by filtration, washed several times with diethyl ether and recrystallised from chloroform/ethanol (1:1) to obtain a pure compound.

[Fe(16-dch)Cl] Yield 0.06 g (70%); C52H66N2O4ClFe; calcd. C 70.0, H 9.4, N 3.1; found C 69.8, H 9.3, N, 3.2. ESI–MS: m/z = 891.8 [M]+. IR (νmax cm⁻¹, KBr): 3095 (νas(C=H)), 2854 (νs(C=H)), 1606 (ν(C = N)), 1554 (ν(C = C)), 1256 (ν(C=O), phenolic), 1148 (ν(C=N)), 758 (δ(C=H)), 560 (ν (Fe=N)), 452 (ν(Fe=O)).

[Fe(16-opd)Cl] Yield 0.06 g (70%); C52H66N2O4ClFe; calcd. C 70.5, H 8.8, N 3.1; found C 70.4, H 8.7, N, 3.2. ESI–MS: m/z = 885.8 [M]+. IR (νmax cm⁻¹, KBr): 3098 (νas(C=H)), 2857 (νs(C=H)), 1605 (ν(C = N)), 1548 (ν(C = C)), 1254 (ν(C=O), phenolic), 1145 (ν(C=N)), 757 (δ(C=H)), 556 (ν (Fe=N)), 455 (ν(Fe=O)).

3. Results and discussion

3.1. Synthesis and characterisation

Following a simple synthetic strategy given in scheme 1, condensation of hexadecyloxy salicylaldehyde with appropriate diamine resulted in the isolation of N, N’-bis[4-(4'-hexadecyloxy)salicylidene]-trans-1; 2-diaminocyclohexane (16-dch) and N, N’-bis[4-(4'-hexadecyloxy)salicylidene]-1,2-phenylene diamine (16-opd) in quantitative yield. The iron(III) complexes were prepared by refluxing the tetradeinate [N2O2] donor Schiff base ligands with FeCl3.6H2O (1:1 molar ratio) in methanol. The complexes so obtained are deep brown solids in good yields and were recrystallised from chloroform/ethanol. The ligands and the corresponding iron(III) complexes were characterised with the help of analytical data and spectroscopic study such as FT–IR, UV–Vis, 1H and 13C NMR and ESI–MS. The results of elemental analysis indicate a good match with the calculated values, which corroborates the proposed formulae of the compounds. Comparison of FT–IR spectra of the ligands and their corresponding iron(III) complexes reveal some important characteristics features. The ligands show a strong absorption band around ~1620 cm⁻¹ due to C = N stretching attesting the formation of Schiff base. This band shifted to lower wave numbers by ~15 cm⁻¹ in the complexes due to reduction of the double-bond character of the C = N bond indicating the involvement of imine nitrogen in coordination to the metal centre.[51,52] The infrared spectra of the ligands showed a broad absorption band at ca. 3350 cm⁻¹ due to phenolic–OH. The absence of absorption band corresponding to the –OH group in the metal complexes indicate phenolic-O coordination with the metal centre. The Fe–N and Fe–O stretching frequency of the complexes located at ~560 cm⁻¹ and ~455 cm⁻¹, respectively, lends further confirmation of the coordination of the azomethine-N and phenolic-O to the metal centre (Fig S1 and S2). The ESI–MS of the compounds are concordant with their formula weights. The 1H NMR spectra of the free ligands showed characteristic signals at 13.1–13.4 ppm, corresponds to the -OH proton, and at 8.2–8.4 ppm, corresponds to the imine proton.

3.1.1. UV-visible absorption studies

The electronic spectra of the compounds were recorded in dichloromethane at room temperature (Table 1). Both the ligands (16-dch and 16-opd) exhibited two absorption bands in the region ~278–332 nm, assigned to π→π* transitions of the molecular orbitals essentially localised on the phenyl ring. In addition, ligand 16-opd exhibited a shoulder band at ca. 364 nm
corresponds to $\pi \rightarrow \pi^*$ transition of the azomethine chromophore. Whereas, ligand 16-dch showed a low-intensity band at ca. 390 nm, attributed to the $n \rightarrow \pi^*$ transition of the imine nitrogen lone pair to the $\pi^*$ orbital of the $C = N$ fragment. The complexes showed one intense band ~282–312 nm originating from $\pi \rightarrow \pi^*$ transitions of the aromatic rings (Figure 1). Another low-intensity band observed in the region ~410–450 nm corresponds to the ligand to metal charge transfer (LMCT) transition.

### 3.2. POM and DSC study: mesomorphic behaviour

The thermal behaviour of the compounds was examined using differential scanning calorimetry and polarised optical microscopy. The thermal data of the complexes are summarised in Table 2. The free ligands did not show any mesomorphic behaviour probably due to their greater conformational flexibility with small molecular anisotropy. However, on coordination with the metal centre mesomorphism in the ligand was induced, which indicates conformational modification of the ligands with enhanced structural anisotropy in

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**Table 1. UV–Vis spectra of the compounds.**

| Compounds | $\pi \rightarrow \pi^*$ (Lmol$^{-1}$ cm$^{-1}$) | $n \rightarrow \pi^*$ (Lmol$^{-1}$ cm$^{-1}$) | CT (Lmol$^{-1}$ cm$^{-1}$) |
|-----------|---------------------------------------------|---------------------------------------------|---------------------------|
| 16-dch    | 278 (23500)                                 | ~390 (1800)                                 | –                         |
| 16-opd    | 292 (16400)                                 | –                                           | ~450 (3500)                |
| [Fe(16-dch)Cl] | 282 (24800)                              | –                                           | ~410 (6800)                |
| [Fe(16-opd)Cl] | 312 (19200)                              | –                                           | ~450 (3500)                |

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**Scheme 1.** (i) $C_n H_{2n+1}$ Br, KHCO$_3$, KI, dry acetone, $\Delta$, 36 h, (ii) glacial AcOH, absolute EtOH, $\Delta$, 3 h and (iii) FeCl$_3$.6H$_2$O, EtOH, $\Delta$, 2 h.
the presence of metal centre. The [Fe(16-dch)Cl] complex exhibited enantiotropic mesomorphic behaviour. Examination of the sample under polarised optical microscopy revealed that on cooling the sample from isotropic melt, a viscous mesophase (Figure 2) is developed at 152.6°C, which is identified as lamellar columnar phase. The DSC thermogram of the complex [Fe(16-dch)Cl] exhibited three sharp transitions in heating as well as cooling (Figure 3). The transition at 158.3°C ($\Delta H = 15.6$ KJ mol$^{-1}$) is due to I-ColL phase transition.

The [Fe(16-opd)Cl] complex showed a fan-like texture (Figure 4) at 126.5°C characteristic of typical SmA mesophase. The DSC thermogram of [Fe(16-opd)Cl] showed two transition in the heating run but no transition could be detected in the cooling run (Figure 5). The transition at 82.3°C ($\Delta H = 2.3$ KJ mol$^{-1}$) is due to crystal to SmA phase transition. The thermal stability of the complexes confirmed on subsequent heating and cooling scans, although the complexes tend to decompose at higher temperature after reaching the isotropic state. It is important to note that [Fe(16-dch)Cl] showed high enthalpy values for the phase transition in comparison to [Fe(16-opd)Cl]. The larger enthalpy change for [Fe(16-dch)Cl] during transition from mesophase to isotropic liquid indicated a higher lamellar ordering of the mesophase, that is, lamellar columnar (ColL) phase.

### 3.3. X-ray diffraction studies

To confirm the results as obtained from optical microscopy and DSC experiment, temperature-dependent XRD studies was carried out for the [Fe(16-dch)Cl] and [Fe(16-opd)Cl] complexes in the mesophase (Table 3). For [Fe(16-dch)Cl] complex at 150°C, three sharp Bragg peaks were observed at 30.3, 15.15 and 10.1 Å in the small angle region (Figure 6). The sharp nature of the small angle reflection clearly indicates towards the existence of a long-range interaction of the structure. These reflections do not attest to either

![Figure 1. UV–Vis absorption spectra of iron(III) complexes in dichloromethane (10$^{-4}$M solution).](image1)

![Figure 2. (colour online) Fan-like texture of [Fe(16-dch)Cl] at 152°C on cooling.](image2)

![Figure 3. DSC thermogram of [Fe(16-dch)Cl].](image3)

![Figure 4. (colour online) Fan-like texture of [Fe(16-dch)Cl] at 152°C on cooling.](image4)

![Figure 5. DSC thermogram of [Fe(16-dch)Cl].](image5)

![Figure 6. DSC thermogram of [Fe(16-dch)Cl].](image6)
a purely lamellar or purely columnar structure. The d-spacing ratio of the three reflections turns out to be 1:2:3, which corresponds to (001), (002) and (003) plane. The presence of these equidistant reflections shows that the molecules are packed in a regularly spaced layer-like arrangement in the mesophase with a layer distance of 30.3 Å. Two other less intense but relatively sharp reflections at 5.0 and 4.6 Å overriding a broad peak observed in the wide-angle region corresponds to (010) and (100) plane. The diffuse peak corresponds to the short-range liquid-like positional ordering of molten alkoxy chains within the layers. The Miller index of (010) and (100) plane affords the presence of short columns within the layers.

As the intensity of the peaks are less than primary layer peak, they can be ascribed to 2D columnar modulation of the layer. Therefore, it may be inferred that the observed mesophase is not a simple SmA phase due to the presence of short-range columnar order within the layers. The XRD data clearly indicates towards lamello-columnar phase (Col$_L$) with two-dimensional ordering of molecules within the smectic layers. A model (Figure 7) could be deduced for the organisation of the molecules. In the present case, the non-discoid molecules tend to organise in a face-to-face antiparallel dimeric fashion within the layer. The one-dimensional stacking distance of 4.6 Å with the aliphatic alkoxy chains diverged in the opposite direction of the column. However, it is relevant here to mention that the report related to metallomesogen exhibiting Col$_L$ mesophase are not ubiquitous in literature.[16–19,23,53,54] Molecules with branched chains where columnar morphology often transcends into a lamellar structure have been well elaborated by Lattermann et al. [55,56].

The diffraction pattern of [Fe(16-opd)Cl] at 120°C exhibit four sharp reflections in the low-angle region.

### Table 3. XRD data of iron(III) complexes.

| Compound        | Mesophase parameters$^a$ | $d_{\text{obsd}}$/Å$^b$ | Miller indices$^c$ |
|-----------------|--------------------------|--------------------------|-------------------|
| [Fe(16-dch)Cl]  | $a = 4.63$ Å             | 30.29 (30.30)            | 001               |
| $T = 150^\circ$C| $b = 5.01$ Å             | 15.15 (15.15)            | 002               |
|                 | $c = 30.30$ Å            | 10.10 (10.10)            | 003               |
|                 |                          | 5.01                     | 010               |
|                 |                          | 4.63                     | 100               |
|                 |                          | ca. 4.52                 |                   |
| [Fe(16-opd)Cl]  | $d = 30.3$ Å             | 45.65 (45.52)            | 001               |
| $T = 120^\circ$C| $V_m = 1470$ Å$^3$       | 22.82 (22.75)            | 002               |
|                 |                          | 15.20 (15.24)            | 003               |
|                 |                          | 11.38 (11.36)            | 004               |
|                 |                          | ca. 4.60                 |                   |

![Figure 4](colour online) Fan-like texture of [Fe(16-opd)Cl] at 126°C on cooling.

![Figure 5](DSC thermogram of [Fe(16-opd)Cl].)

![Figure 6](XRD profile of [Fe(16-dch)Cl].)
with d-spacings of 45.6, 22.7, 15.2 and 11.3 Å which are in the ratio 1:2:3:4. These equidistant reflections correspond to (001), (002), (003) and (004) plane with a lamellar periodicity of 45.6 Å (Figure 8). A diffuse peak centred at ca. 4.6 Å is also observed in the wide-angle region corresponding to the short-range liquid-like positional ordering of the alkoxy chains within the layer plane. However, as opposed to [Fe(16-dch)Cl], there is no long-range correlation order observed within the layers in this complex, as evident from the absence of any sharp peak in the wide-angle region. Therefore, a SmA mesophase with a layer-like arrangement of the molecules is conjectured. The molecules are arranged in a dimeric head-to-head organisation (Figure 9) within the layer. Pertinent here is to mention that, similar d-block metallomesogen of ‘salen’-type ligands with square planar geometry reported by us, predominantly shows columnar mesophase with hexagonal or rectangular ordering.[24–29] The five coordinated square pyramidal geometry of the resulting iron(III) complex is believed to be responsible for the layer-like arrangement of the molecules in the present case. Thus, the packing of molecules in the mesophase, we believe, are promoted by pi-stacking. The spacer group in [Fe(16-dch)Cl] is non aromatic while it is aromatic in [Fe(16-opd)Cl]—this might have a bearing on inter molecular ‘pi-stacking’ interaction causing slight variation in the molecular packing.

### 3.4. DFT studies

DFT calculations were performed at B3LYP level using the Gaussian 09 package.[57] The 6-31G (d, p) basis set was used for the computation. The gas-phase ground state geometry of the iron(III) complex was fully optimised with tight convergence criteria excluding any symmetry constrain. The vibrational frequency calculation was performed using second-derivative analytic methods to verify the attainment of the energy minima at the same level of theory that confirmed the absence of imaginary Eigen values. A five coordinated square pyramidal structure around the metal ion was conjectured (Figures 10 and 11). The N₂O₂ core forms an equatorial plane by means of the O and N atoms, and the axial site is occupied by Cl atom. The central metal ion is located in the equatorial least square plane. Some of the significant geometric parameters of the optimised iron(III) complexes are shown in Table 4. The Fe–N and Fe–O bond lengths were 2.12–2.14 Å and 1.89 Å, implying the presence of regular σ and dative coordination.
bonding, respectively. The calculated bond length of Fe—Cl was 2.38 Å. The bond angle O1—Fe—O2, O1—Fe—N1, O1—Fe—N2, N1—Fe—O2, N1—Fe—N2, N2—Fe—O2 value reveals a deviation from planarity of the equatorial plane. The dihedral angle, N1—O1—O2—N2 of [Fe(16-dch)Cl] was found to be 12°, whereas for [Fe(16-opd)Cl] the value of dihedral angle is close to zero. The cyclohexane spacer acquires a somewhat non-planar conformation than phenylene spacer, and is believed to have caused a greater distortion from a square pyramidal geometry. The molecular length of the iron(III) complexes measured from the two terminal end of the side alkyl chain in the fully extended form was found to be around 41.2 Å.

4. Conclusion

Iron(III) complexes of tetradentate 'salen'-type Schiff base ligands containing alkoxy chains in 4-position of the terminal aromatic ring and a central cyclohexane/phenylene spacer have been successfully synthesised and characterised. While the Schiff base ligands do not show any liquid crystalline behaviour, but mesomorphism is induced upon coordination with the iron(III) centre. The [Fe(16-dch)Cl] complex exhibits enantiotropic lamello columnar mesomorphism, whereas the [Fe(16-opd)Cl] complex exhibits SmA mesomorphism. Based on spectral and DFT studies, a five coordinated distorted square pyramidal geometry have been proposed.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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Table 4. Calculated bond distances (Å), angles (°) and dihedral angles (°) of the iron(III) complexes.

| Geometrical parameters | [Fe(16-dch)Cl] | [Fe(16-opd)Cl] |
|------------------------|--------------|--------------|
| Fe—O1                  | 1.89         | 1.89         |
| Fe—O2                  | 1.89         | 1.89         |
| Fe—N1                  | 2.12         | 2.14         |
| Fe—N2                  | 2.14         | 2.14         |
| O1—Fe—O2              | 94.2         | 93.8         |
| O1—Fe—N1              | 85.9         | 85.9         |
| O1—Fe—N2              | 76.1         | 74.9         |
| N1—Fe—O2              | 86.3         | 85.9         |
| N1—Fe—O3              | 153.8        | 144.7        |
| O1—Fe—N3              | 136.7        | 144.8        |
| N1—O1—O2—N2           | 12           | 0            |
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