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Synthesis and mesomorphic properties of naphth-2-yl 2-pyridylmethyl ketones and their copper(II) complexes

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The synthesis, characterization and mesomorphic properties of a homologous series of 6-alkoxy naphth-2-yl 2-pyridylmethyl ketones and their copper(II) complexes are reported. All the ligands and their copper complexes, with exception of the lowest homologues, exhibit enantiotropic mesophases. According to textural observations, the ligands display nematic and smectic transitions depending on the length of the alkoxy chain on the pyridine moiety, while the mesogenic complexes show exclusively smectic transitions. In comparison with the phenyl analogues, stabilization of the mesophase and a greater tendency to smectic ordering through incorporation of the naphthyl group into the molecules is confirmed.

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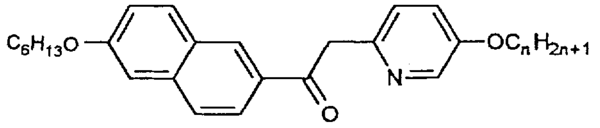
1. Introduction

There has been extensive research on the synthesis and characterization of metallomesogens due to the special combination of liquid crystalline properties and the presence of a transition metal in the structure of the molecules [1–3]. Previously, we have reported the synthesis and mesomorphic behaviour of a homologous series of alkoxy phenacyl pyridine ligands and their copper(II) complexes which exhibit monotropic nematic and enantiotropic smectic A phases, respectively [4]. As an extension of this work, we have replaced the phenyl moiety by a naphthyl group and prepared a homologous series of 6-hexyloxynaphth-2-yl 5-alkoxy-2-methylpyridyl ketones **I** and their copper(II) complexes **II**.

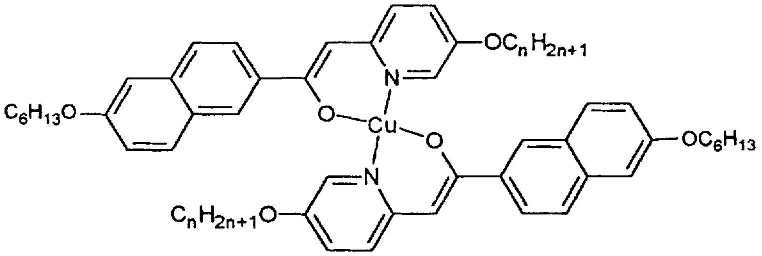
The naphthalene system, due to elongation of the π-system, increases the anisotropy of the electronic polarizability along the molecular long axes and favours mesophase formation [5, 6]. Therefore, stabilization of the mesomorphic properties of the new naphthyl derivatives compared to their phenyl analogues is to be expected. Coordination of a metal atom to the ligands with the naphthyl group is interesting not only with respect to the colour of the complex formed, but also with regard to the possible magnetic molecular interactions.

2. Synthesis and characterization

The synthesis of the ligands and their copper(II) complexes was carried out following scheme 1 according to the procedure described previously [4].



I



II

$n = 2,4,5,6,7,8,10,11$

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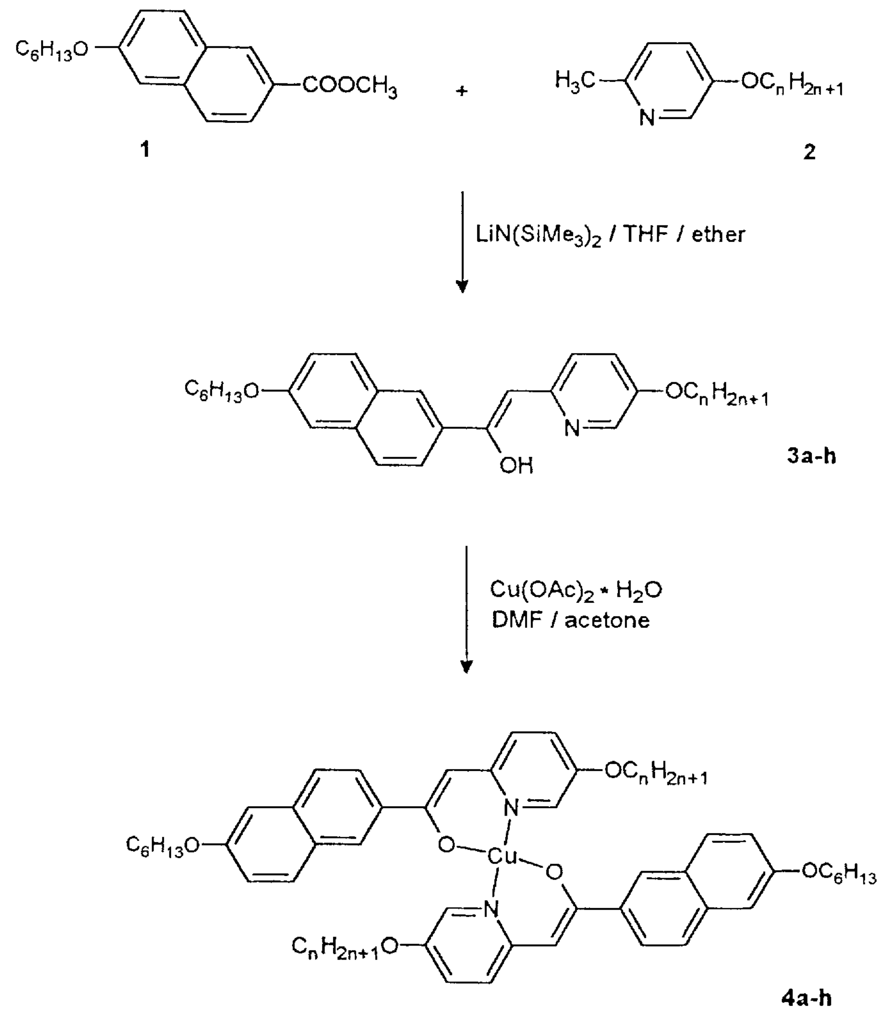


	a	b	c	d	e	f	g	h
n	2	4	5	6	7	8	10	11

Scheme 1.

From the structural viewpoint the naphthyl derivatives behave in the same way as the phenacyl pyridine derivatives; the enolic form is present in the solid state, while the keto-form is present in chloroform solution. The enolic form was confirmed by the presence of absorption bands which correspond to the O–H stretch (weak, 3600–3200, 1060 cm^{-1}) and C=C stretch (doublet, 1625, 1615 cm^{-1}) in the IR spectrum. Very weak absorption bands for the O–H stretch and the intense absorption for the C=C stretch reveal a resonance stabilized intramolecular hydrogen bond [7]. The presence of the signals at 6·15 ppm in the ^{1}H NMR spectrum corresponding to the proton of the methylene group, and at 197·2 ppm in the ^{13}C NMR spectrum corresponding to the carbon atom of the carbonyl group confirm the keto-form in chloroform solution. The UV-VIS spectra of the naphthyl derivatives are characterized by five absorption bands. More pronounced structuring of the spectrum and a bathochromic shift of the long wavelength band by 10 nm is observed compared to the phenacyl pyridine ligands, and this is a consequence of the extension of conjugation in the naphthyl system. The electronic spectra of the copper(II) complexes are characterized by three broad bands. The long wavelength band has a bathochromic shift to 402 nm due to ligand planarity in the deprotonated enol form, and the molar extinction coefficient is slightly increased as compared to the ligands. A very weak, hardly visible d-d band appears as a slight shoulder at 472 nm. The UV-VIS spectra of the ligand **3b** and its copper complex **4b** are shown in figure 1.

3. Results and discussion

The mesomorphic behaviour of the compounds prepared was studied by optical polarizing microscopy and differential scanning calorimetry (DSC). The DSC data were taken at the onset of the signal and correspond to the first heating scan. Tables 1 and 2 summarize the mesomorphic transition temperatures, enthalpies and entropies for the homologous series of free ligands and their copper complexes with $n = 2, 4, 5, 6, 7, 8, 10, 11$, where n denotes the number of carbon atoms in the 5-alkoxy chain of the pyridine sub-unit in formulae **I** and **II**.

All the ligands are thermally stable and, with the exception of the lowest homologue ($n = 2$), show enantiotropic mesophases. The homologues with $n = 4$, 5 and 6 display an enantiotropic nematic phase. The nematic phase was clearly identified by its very fluid schlieren texture and by the typical nematic droplets observed on cooling from the isotropic liquid [8]. It is interesting to note that homologue **3d** exhibits two mesophases, an enantiotropic nematic and a monotropic smectic A phase. The N–S_A transition was clearly observed on cooling, when the nematic very fluid schlieren texture was transformed into a smectic domain texture and then into simple fans. On further cooling, the smectic A phase crystallized. Upon heating the same sample, only the nematic phase was observed. The phase behaviour of homologue **3d** is sketched in scheme 2.

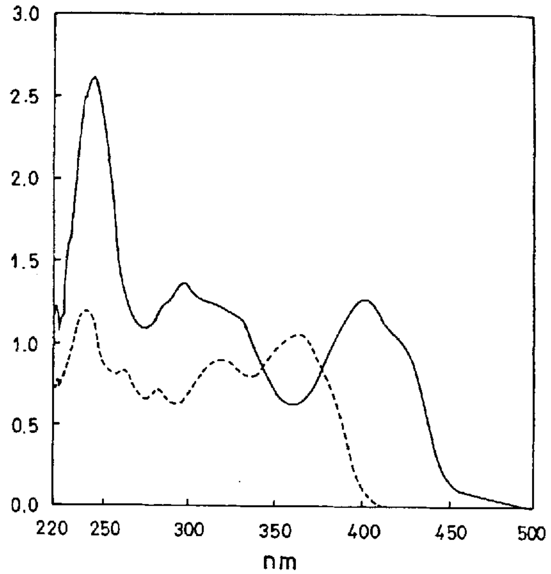


Figure 1. UV-VIS spectra of **3b** (dotted line, $c = 4{\cdot}8 \times 10^{-5}$ mol dm^{-3}) and **4b** (solid line, $c = 4{\cdot}9 \times 10^{-5}$ mol dm^{-3}) in THF as solvent.

Table 1. Transition temperatures, enthalpies and entropies for naphthyl ligands **3a–h**.

Ligand	n	Transitions	T/°C	ΔH/kJ mol^{-1}	ΔS/JK^{-1} mol^{-1}
3a	2	Cr–I	115	44·31	114·17
		I–N[c]	101	−3·62	−9·73
3b	4	Cr–Cr_2	57	15·10	45·74
		Cr_2–N	92	18·63	51·02
		N–I	104	3·30	8·75
3c	5	Cr–Cr_2	67	0·18	0·53
		Cr_2–N	107	42·96	113·00
		N–I	111[b]	—	—
3d	6	Cr–Cr_2	76	0·72	2·06
		Cr_2–N	110	46·01	120·08
		N–I	119	0·35	0·89
		N–S_A[c]	93	−0·78	−2·13
3e	7	Cr–Cr_1	64	5·59	16·58
		Cr_1–Cr_2	80	0·79	2·24
		Cr_2–S_C	103	34·50	91·72
		S_C–I	111	6·85	17·83
3f	8	Cr–Cr_1	53	0·86	2·64
		Cr_1–Cr_2	74	4·67	13·45
		Cr_2–S_C	99	35·12	94·37
		S_C–I	111	9·60	24·99
3g	10	Cr–Cr_1	56	10·06	30·56
		Cr_1–Cr_2	68	0·29	0·85
		Cr_2–S_C	99	36·76[a]	—
		S_C–I	112	—	—
3h[d]	11	Cr–Cr_2	95	0·27	0·73
		Cr_2–S_C	112	38·41	99·73
		S_C–I	118	10·26	26·23

Cr crystal; N nematic; S_A smectic A; S_C smectic C; I isotropic liquid.

[a] low resolution of the peaks–combined enthalpies.

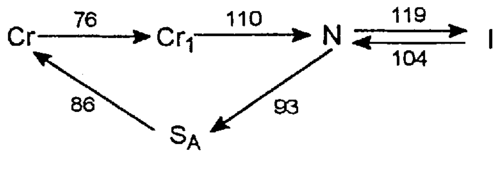
[b] determined by optical method.

[c] monotropic transition.

[d] scanning rate 2° min^{-1}.

The higher homologues, **3e–h** show enantiotropic smectic C phases characterized by schlieren and broken fan-shaped textures. Almost all the ligands exhibit solid state polymorphism.

The copper complexes, with exception of the lowest homologue **4a**, exhibit enatiotropic smectic mesomorphism. The smectic A phase with focal-conic/homeotropic textures, was the only liquid crystal phase observed for the complexes **4b–f**. According to the



Scheme 2.

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Table 2. Transition temperatures, enthalpies and entropies for naphthyl copper(II) complexes **4a–h**.

Complex	n	Transitions	T/°C	ΔH/kJ mol^{-1}	ΔS/JK^{-1}mol^{-1}
4a	2	Cr–I	170^a	—	—
4b	4	Cr–Cr$_1$	83	0·51	1·43
		Cr$_1$–Cr$_2$	119	26·95	68·72
		Cr$_2$–S$_A$	153	0·27	0·63
		S$_A$–I	171	17·86	40·21
4c	5	Cr–Cr$_2$	140	14·62	35·38
		Cr$_2$–S$_A$	155	54·45	127·17
		S$_A$–I	164^a	—	—
4d	6	Cr–Cr$_2$	127	58·80	146·94
		Cr$_2$–S$_A$	143	0·14	0·34
		S$_A$–I	175	19·42	43·33
4e	7	Cr–Cr$_2$	126	25·61	64·16
		Cr$_2$–S$_A$	155	1·10	2·57
		S$_A$–I	178	19·20	42·57
4f	8	Cr–Cr$_2$	127	18·31	45·76
		Cr$_2$–S$_A$	152	67·86^b	—
		S$_A$–I	160	—	—
4g	10	Cr–S$_B$	97	39·29	106·15
		S$_B$–S$_C$	144	16·26^b	—
		S$_C$–I	149	—	—
4h	11	Cr–S$_B$	102	47·71	127·16
		S$_B$–S$_C$	139	2·65	6·43
		S$_C$–I	148	9·02	21·42

Cr crystal; S$_A$ smectic A; S$_C$ smectic C; S$_B$ smectic B; I isotropic liquid.

[a] determined by optical method.

[b] low resolution of the peaks–combined enthalpies.

calorimetric data, these compounds display crystal polymorphism. For the compounds **4b, d, e** the Cr–Cr$_2$ transition is accompanied by a higher enthalpy change than the Cr$_2$–S$_A$ transition. However, the sum of these two values, which represents the total melting enthalpy, is higher than the isotropization enthalpy. Similar behaviour has been noticed for some copper(II) complexes derived from 2,5-dihydroxybenzaldehyde [9]. None of these solid phase transitions could be observed by microscopy. Although the isotropization enthalpies in **4b, d, e** are high, the S$_A$ phase was proven by preparing a contact preparation with a known substance; the smectic A phase was totally miscible with the S$_A$ phase of ethyl *p*-ethoxybenzal-*p*-aminocinammate [10].

Homologues **4g** and **4f** display smectic polymorphism. According to the schlieren/broken fan-shaped texture, the higher temperature mesophase was identified as smectic C. To examine the low temperature mesophase and to avoid partial decomposition of the complexes in the isotropic state, the compounds were heated only to the S$_C$ mesophase and then cooled. During the cooling cycle, the smectic C type schlieren texture transformed into a different, more viscous, schlieren texture like that shown for *N*-4-*n*-pentyloxybenzylidene-4-*n*-pentyl-aniline [8]. In certain regions a mosaic texture is observed as well. Upon standing for several hours at 120°C the schlieren C texture was transformed completely into the mosaic texture. A contact preparation showed complete miscibility with the S$_B$ phase of ethyl *p*-ethoxybenzal-*p*-aminocinammate [10] for the low temperature mesophase in **4g** and **4f**. Therefore this low temperature phase was identified as smectic B.

A survey of the phase behaviour for the free ligands and their copper complexes is given in figures 2 and 3, respectively.

Figures 2 and 3 show the effect of chain length on the type of mesophase. For the ligands, this effect is more pronounced than for the copper complexes. The lower ligand homologues display nematic phases. Increasing the chain length from $n=7$ to $n=11$, the nematic mesophase disappears and only the more ordered smec-

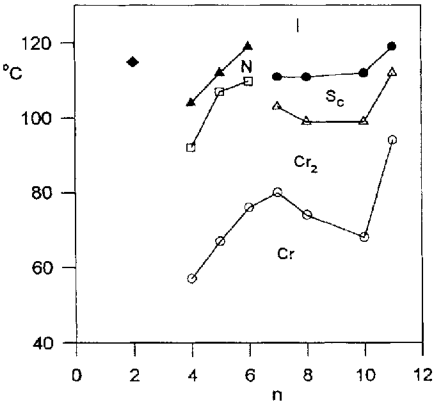

Figure 2. A plot of the phase transition temperatures versus the number of carbon atoms in the alkoxy chain for the ligands **3a–h**. For the ligands **3a** and **3d** monotropic phase transitions are not included. ◆ Cr–I; □ Cr–N; ▲ N–I; △ Cr$_2$–S$_C$; ● S$_C$–I; ○ Cr–Cr$_2$

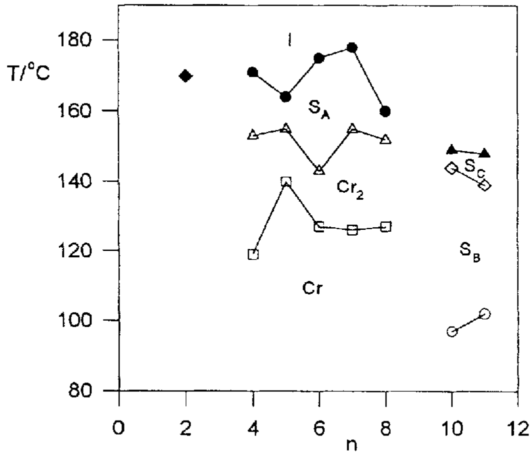

Figure 3. A plot of the phase transition temperatures versus the number of carbon atoms in the alkoxy chain for the copper(II) complexes **4a–h**. ◆ Cr–I; □ Cr–Cr$_2$; △ Cr$_2$–S$_A$; ○ Cr–S$_B$; ◇ S$_B$–S$_C$; ● S$_A$ I; ▲ S$_C$ I.

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tic C mesophase is observed. In the case of the copper complexes **4b–h**, the smectic A phase is present till the alkoxy chain reaches 8 carbon atoms. This can be explained by the nearly equal overall chain lengths of the complexes for $n \leqslant 8$ as determined by the length of the 6-hexyloxy chain used on the naphthyl moiety. Observation of Dreiding models and computer assisted modelling of these complexes confirmed as expected that increase in the alkoxy chain length on the pyridine moiety to 10 or 11 carbon atoms elongates the overall length of the molecule. Consequently the mesomorphic behaviour is changed, and a small range S_C and a wide range S_B phase are observed. Different mesomorphic behaviours for the free ligands and their copper complexes were regularly observed. Coordination of a metal ion to the ligand enhances transverse intermolecular association due to the greater polarity of the coordination bonds in CuL_2. Assuming planar coordination of the two ligands around the copper, a highly conjugated complex is formed which is capable of intermolecular π-π-interactions, forcing parallel alignment of the rod-shaped molecules in layers and therefore promoting the smectic phase.

Comparison of the mesomorphic properties of the naphthyl derivatives **3d** and **4d** with their phenyl analogues [4], presented in table 3, clearly shows that the influence of the naphthyl group is more pronounced for the free ligands. While naphthyl derivative **3d** displays enantiotropic nematic and monotropic smectic A transition phases, the phenyl analogue shows only a Cr–I transition [4]. The behaviour of the former ligand is a consequence of elongation of the central core unit and extension of the electronic conjugation. The copper complexes of both ligands show enantiotropic S_A phases, but the temperature range for the naphthyl derivative is broader than for the phenyl analogue [4].

In conclusion, these results confirm that incorporation of the more polarizable naphthalene unit instead of the phenyl unit promotes stronger and wider ranging molecular interactions resulting in conversion of the non-mesogenic phenyl materials into liquid crystalline materials with a range of mesophases.

4. Experimental

^1H- and ^{13}C-NMR spectra were recorded in $CDCl_3$ solutions on a Varian Gemini XL 300 spectrometer; shifts are given in ppm downfield from TMS as internal standard. IR spectra were recorded on a Perkin-Elmer 297 spectrophotometer and UV-VIS spectra on a Phillips PU 8700 UV/Visible spectrophotometer. The optical observations were made using a Leitz polarizing microscope (× 150) equipped with hot stage. The phase transition temperatures were determined using a Perkin-Elmer DSC-7 differential scanning calorimeter operated at a scanning rate of $10^\circ \mathrm{min}^{-1}$ on heating and cooling.

Ethyl *p*-ethoxybenzal-*p*-aminocinammate was used in contact preparations as a standard substance with the known phase sequence; Cr–S_B 77·1°C, S_B–S_A 115·6°C, S_A–N 155·4°C, N–I 157·6°C [10].

Methyl 6-hexyloxynaphth-2-oate (**1**) was prepared in 86% yield from methyl 6-hydroxynaphth-2-oate and hexyl bromide in acetone using potassium carbonate as base [11]. The series of 5-alkoxy-2-methylpyridines (**2a–h**) was prepared as reported previously [4]. The structure of the products was confirmed by their IR and ^1H NMR spectra.

Acylation of the carbanion of the 5-alkoxy-2-methyl pyridines with methyl 6-hexyloxynaphth-2-oate, after regioselective metallation of the methyl group with lithium bis-trimethylsilylamide, according to the procedure described in [4], gave the ketones (**3a–h**) in 35–46% yield. Crude products were purified by column

Table 3. Comparison of the mesomorphic behaviour of naphthyl ligand **3d** and its Cu(II) complex **4d** with the phenyl analogues [4].

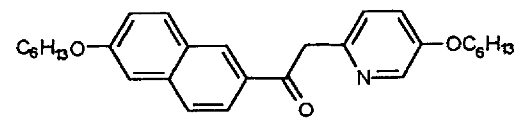
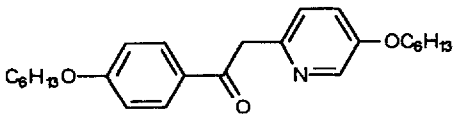
	Phase transitions	
Compound type		
Ligands	$\begin{tikzcd} Cr \arrow[r, "76"] & Cr_1 \arrow[r, "110"] & N \arrow[r, shift left, "119"] \arrow[dl, "93"] & I \arrow[l, shift left, "104"] \\ & S_A \arrow[ul, "86"] & & \end{tikzcd}$	$Cr \xrightarrow{96} I$
Cu(II) complexes	$Cr \xrightarrow{127} Cr_1 \xrightarrow{143} S_A \xrightarrow{175} I$	$Cr \xrightarrow{145} S_A \xrightarrow{166} I$

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Table 4. Elemental analyses of the new compounds.

Compound	Molecular formula	Found(calc) C/%	Found(calc) H/%	Found(calc) N/%
3a	$C_{25}H_{29}NO_3$	76·83(76·70)	7·59(7·47)	3·58(3·58)
3b	$C_{27}H_{33}NO_3$	77·19(77·29)	8·10(7·93)	3·28(3·34)
3c	$C_{28}H_{35}NO_3$	77·67(77·56)	8·13(8·14)	3·11(3·23)
3d	$C_{29}H_{37}NO_3$	77·68(77·82)	8·19(8·33)	3·07(3·13)
3e	$C_{30}H_{39}NO_3$	78·16(78·05)	8·76(8·52)	3·08(3·03)
3f	$C_{31}H_{41}NO_3$	78·35(78·28)	8·77(8·69)	3·16(2·95)
3g	$C_{33}H_{45}NO_3$	78·59(78·69)	8·78(9·01)	2·78(2·78)
3h	$C_{34}H_{47}NO_3$	78·90(78·87)	9·01(9·15)	2·63(2·71)
4a	$C_{50}H_{56}N_2O_6Cu$	70·95(71·11)	6·93(6·68)	3·27(3·32)
4b	$C_{54}H_{64}N_2O_6Cu$	71·97(72·01)	7·34(7·16)	3·29(3·11)
4c	$C_{56}H_{68}N_2O_6Cu$	72·22(72·42)	7·37(7·38)	3·20(3·02)
4d	$C_{58}H_{72}N_2O_6Cu$	73·02(72·81)	7·32(7·59)	3·07(2·93)
4e	$C_{60}H_{76}N_2O_6Cu$	73·28(73·18)	7·66(7·78)	2·85(2·84)
4f	$C_{62}H_{80}N_2O_6Cu$	73·40(73·52)	7·72(7·96)	2·90(2·77)
4g	$C_{66}H_{88}N_2O_6Cu$	74·25(74·16)	8·54(8·30)	2·76(2·62)
4h	$C_{68}H_{92}N_2O_6Cu$	74·28(74·45)	8·60(8·45)	2·60(2·55)

chromatography on silica gel with toluene/di-isopropyl ether as eluent and then by crystallization from cyclohexane or *n*-hexane. The ligands were characterized by their IR, ^{1}H, ^{13}C and UV-VIS spectra and by elemental analysis. Characteristic data for **3b** are given as an example. IR (KBr, ν): 3600–3200 (weak, O–H), 2940–2870 (C–H, alkyl), 1625, 1615 (d, C=C), 1190 (C–O) cm^{-1}. ^{1}H NMR (CDDl$_3$, δ): 0·90–1·02 (m, 6H, alkyl), 1·35–1·65 (m, 8H, alkyl), 1·71–1·90 (m, 4H, alkyl), 3·97 (t, $J = 6·4$ Hz, 2H, –Py–OC$\underline{\mathrm{H}}_2$–), 4·09 (t, $J = 6·6$ Hz, 2H, –naph–OC$\underline{\mathrm{H}}_2$–), 4·53 (s, 2H, –C$\underline{\mathrm{H}}_2$–CO–), 7·09–7·24 (m, 4H, 2H Py, 2H naph), 7·72 (d, $J = 8·6$ Hz, 1H naph), 7·85 (d, $J = 8·8$ Hz, 1H naph), 8·05 (d, $J = 7·1$ Hz, 1H, naph), 8·25 (d, $J = 2·8$ Hz, 1H Py), 8·54 (s, 1H naph) ppm. ^{13}C NMR (CDDl$_3$, δ): 13·5, 13·8, 18·9, 22·4, 25·5, 28·9, 31·0, 31·4, 47·3, 68·0, 68·1, 77·1, 106·4, 119·9, 121·9, 124·2, 124·9, 127·0, 127·7, 130·7, 131·2, 131·8, 137·4, 147·2, 154·1, 159·4, 197·1 ppm. UV-VIS (THF, λ_{max}): 239(24 750), 263(17 250), 283(14 850), 319(18 660), 365(22 000) nm(ε).

Copper(II) complexes (**4a–h**) were prepared from the corresponding free ligands and copper(II) acetate monohydrate by heating in the molar ratio 1 : 1·5 in the solvent mixture acetone/DMF. Upon cooling, greenish-brown crystals separated from the solution. The crystals were collected by filtration, washed several times with cold acetone and dried under reduced pressure to yield complexes **4a–h** in 87–97% yield. They were characterized by their IR and UV-VIS spectra and by elemental analysis. Complexation of two ligands to one copper(II) atom was confirmed by the method described in ref. [12]. Characteristic data for **4b** are given as an example. IR (KBr, ν): 2940 (C–H, alkyl), 1590 (C=C), 1190 (C–O), cm^{-1}. UV-VIS (THF, λ_{max}): 245(53 580), 298(27 910), 402(26 090) nm(ε).

Elemental analyses data given in table 4 confirm the proposed structures for all the newly prepared compounds.

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