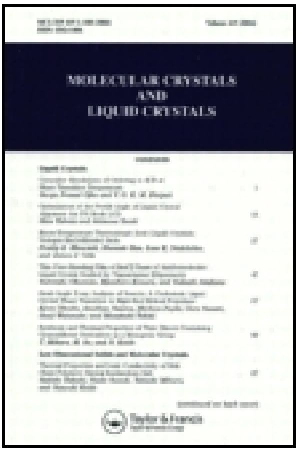

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New Pathways Towards Long-Chain Substituted Dibenzotetraaza[14]annulenes

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New Pathways Towards Long-Chain Substituted Dibenzotetraaza[14]annulenes

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The synthesis of nickel complexes dibenzotetraaza[14]annulenes bearing four, six or eight long paraffinic chains is described. Preliminary results concerning the mesomorphic properties of such systems show that those compounds exhibit a wide range hexagonal columnar phase.

Keywords: Metallomesogens, liquid crystals, synthesis.

INTRODUCTION

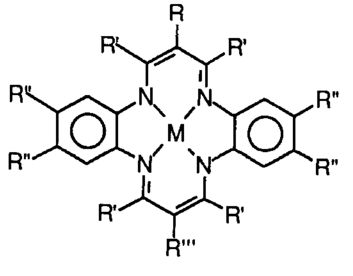
In the past, we have studied the synthesis of mesomorphic (columnar) organometallic π-acceptors. Indeed, we thought that the strong intracolumnar interactions between the π-acceptor molecules would have led to the same columnar arrangement of the crystalline π-donor and consequently to a segregated charge-transfer complex. Such an arrangement is necessary to get a unidimensional organic conductor and was found in the first mesomorphic organic conductor, $DIPSAr_4$/TCNQ.[1]

To obtain a columnar organometallic π-acceptor, we chose to synthesize long-chain substituted nickel bis-dithiolene complexes. Unfortunately, the introduction of two or four alkoxy chains lowered the electronic affinity of the system[2] and failed to provide mesomorphic complexes: nickel bis-dithiolene complexes with two or four alkoxy chains are *crystalline materials.*[3]

The aim of this paper is to present the synthesis and the mesomorphic properties of π-donor metallomesogens which could lead to mesomorphic organic conductors (change-transfer complexes or cationic radical salts), even with a non-mesomorphic partner.

For that purpose, we chose as the π-donor, dibenzotetraaza[14]annulene complexes.

Such compounds, whose general formula is **1**, are known to exhibit characteristics in common with porphyrins and phthalocyanines.[4]



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They can be used as oxygen carriers,[5] redox catalysts[6] and good π-donors.[7] Highly conducting cationic radical salts were obtained by doping complexes **1** (R, R‴ = H or *p*-tolyl for example) with iodine.[4,8,9]

The synthesis of such complexes implies the condensation of *o*-phenylenediamine with, for example, 4-phenyl-1,2-dithiolium salts,[10,11] β-alkoxy (or β-(N,N-dialkylamino)) α-acroleins,[12,13] propynal,[14] malondialdehyde,[9] vinamidinium salts,[15,16] using template[17] or non template[18] conditions. In the case of vinamidinium salts, both symmetrical[13] (R = R‴) and unsymmetrical[15] (R ≠ R‴) complexes were described.

On the other hand, metallomesogens gave rise to an increasing interest during the last decade, due to properties that could appear with the presence of a metal atom.[19–21] Discotic properties were claimed, without any experimental evidences, in a patent on dibenzotetraaza[14]annulenes **1**, in which R, R″ and R‴ were long-chain substituents.[13]

Consequently it was tempting to synthesize the compounds presented in Figure 1 in which mesomorphic properties and an enhanced π-donor character could be expected.

We present here the synthesis of the above complexes together with some preliminary results concerning their mesomorphic behaviour.

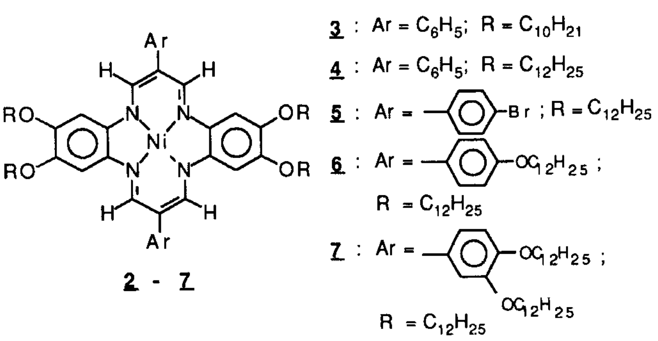
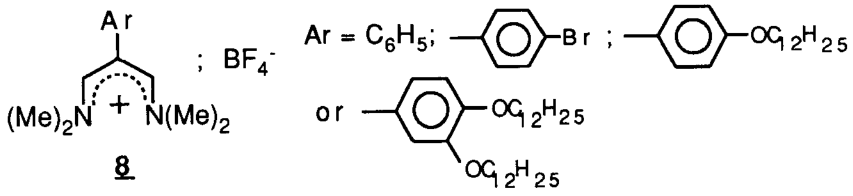


FIGURE 1

SYNTHESIS

In order to obtain the complexes **2**–**7**, we chose the vinamidinium salts **8** as precursor:

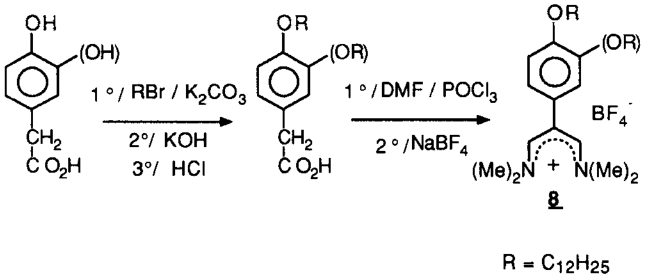


Indeed, in the course of our studies concerning mesomorphic properties of heteroaromatic[22,24] or heteroatomic salts[25,26] we have prepared long-chain substituted vinamidinium salts.

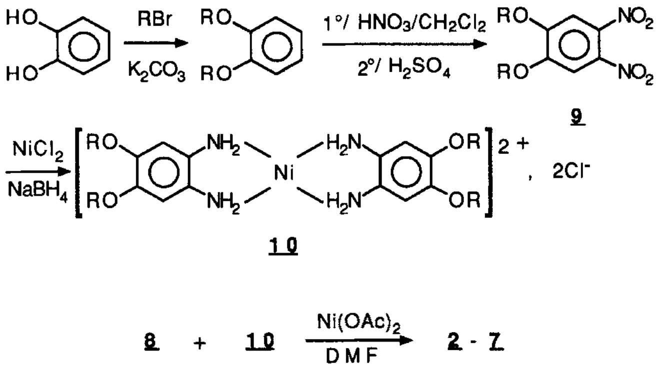
They are easily obtained starting from the suitable aryl acetic acid by a Vilsmeier-Haack reaction according to Scheme **1**.

The yields and melting points of the compounds **8** are reported in Table 1.

The salts **8** have been used to obtain the desired nickel complexes, **2** to **7**, according to the Scheme **2**.



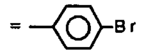
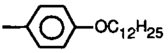
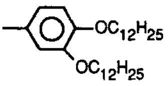
SCHEME 1



SCHEME 2

TABLE 1

Yields and physical characteristics of compounds **8** and **9**

		Yields %	Melting points (°C)	λ_{max} (nm) CH_2Cl_2
8	Ar = C_6H_5	64	185	317
	Ar = 	44	175	316
	Ar = 	55	103-104	318
	Ar = 	67	85	315
9	n=8	82	86	340
	n=10	84	84	340
	n=12	88	81	338

In a first step, pyrocatechol was dialkylated using an alkyl bromide and potassium carbonate according to published procedures.[27]

The pyrocatechol ethers obtained were then dinitrated to compounds **9** in very high yields (see Table 1), using biphasic conditions (to avoid chain cleavage). First, a solution of pyrocatechol diether in dichloromethane was added dropwise to nitric acid. Under such conditions, the mononitro derivative was obtained (its formation was followed by thin layer chromatography (tlc) on silica gel, using CH_2Cl_2 as eluent). Then, to the resulting biphasic mixture, was slowly added sulphuric acid, leading to compound **9**. It is noticeable that if the mononitro derivative is isolated, the subsequent nitration is not complete and yields are lowered.

Dinitro compounds **9** were then reduced using $NiCl_2/NaBH_4$ as described by Nose.[28] This reagent is supposed to generate a form of nickel boride,[29] which is responsible for the selective reduction of aromatic nitro groups. Usually, the hydrolysis of the reaction mixture leads to the desired aniline. In our case, a very unstable compound, the 4,5-dialkoxy *o*-phenylenediamine, is formed. As no hydrolysis of the mixture was performed, the precipitate obtained, which has not yet been identified, was tentatively formulated as the complex **10**; given that it is very insoluble in common organic solvents, no data concerning its structure could be obtained by NMR. Since the synthesis of dibenzotetraaza[14]annulenes from the same type of complex **10** (prepared according to Hieber[30]) was described by Hunziker,[9] this product was used without further purification in the work subsequently described.

TABLE 2

Yields and physical characteristics of nickel dibenzotetraaza[14]-annulenes **2** to **7**

	Yields %	λ (nm), (log ε) CH_2Cl_2	Transition Temperatures °C (transition enthalpies J/g)*
2	13	322 (4.78) 458 (4.87) 524 (3.88)	$\mathrm{Cr} \underset{41\,(5.3)}{\overset{112\,(13)}{\rightleftarrows}} \mathrm{D}_h \xrightarrow{358} \mathrm{I}$
3	15	322 (4.74) 458 (4.83) 524 (3.74)	$\underset{\xleftarrow[53\,(10.7)]{\hspace{8em}}}{\mathrm{Cr}_1 \xrightarrow{60\,(0.18)} \mathrm{Cr}_2 \xrightarrow{107\,(16.9)} \mathrm{D}_h} \xrightarrow{344} \mathrm{I}$
4	27	322 (4.76) 458 (4.85) 524 (3.89)	$\underset{\xleftarrow[27\,(31.6)]{\hspace{8em}}}{\mathrm{Cr}_1 \xrightarrow{46\,(32.0)} \mathrm{Cr}_2 \xrightarrow{99\,(9.4)} \mathrm{D}_h} \xrightarrow{335} \mathrm{I}$
5	3	320 (4.78) 458 (4.83) 524 (3.83)	$\underset{\xleftarrow[4\,(25.7)]{\hspace{8em}}}{\mathrm{Cr}_1 \xrightarrow{22\,(25.7)} \mathrm{Cr}_2 \xrightarrow{87\,(1.8)} \mathrm{M}} \xrightarrow{358} \mathrm{I}$
6	5	314 (4.73) 462 (4.83) 530 (3.88)	$\mathrm{Cr} \underset{16\,(2.74)}{\overset{24\,(27.4)}{\rightleftarrows}} \mathrm{D}_h \xrightarrow{349} \mathrm{I}$
7	3	316 (4.83) 462 (4.92) 532 (4.00)	$\mathrm{Cr} \underset{11\,(13)}{\overset{18\,(13.3)}{\rightleftarrows}} \mathrm{D}_h \xrightarrow{250} \mathrm{I}$

* Clarification with decomposition; all the transitions towards the isotropic phase have been determined by microscopic observations.

Finally, the crude complexes **10** were condensed with the aryl vinamidinium salts **8**, in dimethylformamide, in the presence of $[Ni(OAc)_2]$, and led to the desired dibenzotetraaza[14]annulenes, **2** to **7**, bearing four, six or eight long alkoxy chains. The yields are reported in Table 2.

MESOMORPHIC PROPERTIES

In Table 2, the results concerning the thermal behaviour of complexes **2** to **7** are given. It can be seen that the compounds bearing four and six long alkoxy chains have a very high clearing temperature (> 300°C) where decomposition occurs. If more chains are added to the central rigid core, the clearing temperature is lowered as expected (e.g. to 250°C for compound **7**).

The DSC curves of compounds **2**, **4**, **6**, **7** are presented in Figures 2 to 5. In all cases, they have been obtained without reaching the isotropic phase. The clarification temperatures have been observed by microscopic observations.

Typical textures exhibited by compounds **2**, **3**, **4**, **6** are given in plates 1 to 8 and have been obtained using the following procedure (since the clarification temperature should be avoided). A small quantity of complex is poured on a lamella and a drop of solvent is added (dichloromethane for plates **2**, **4**, **7**; toluene for plates **1**, **3**, **5**, **6**). A cover slide is put

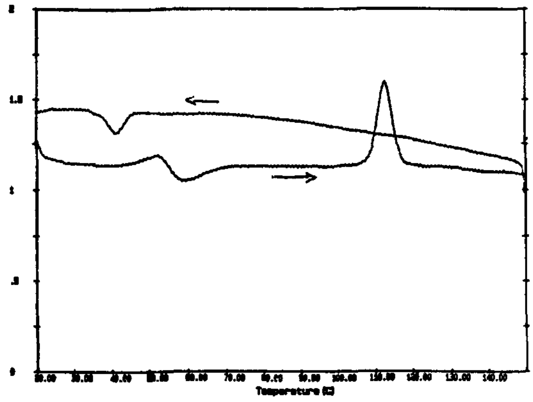


FIGURE 2 DSC curve of compound 2.

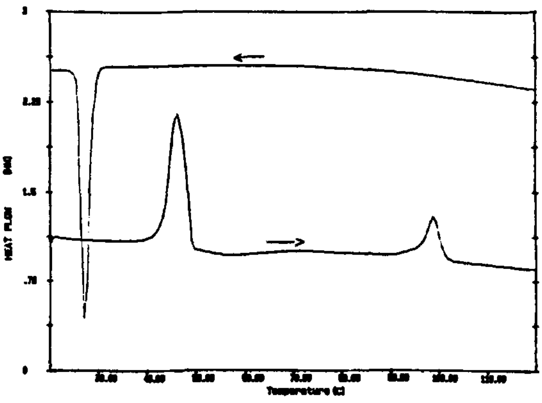


FIGURE 3 DSC curve of compound 4.

on the preparation. The sample is then slowly heated to 200°C (heating rate: 1°/min). By slow evaporation of the solvent, textures of columnar phase were obtained.

Preliminary X-ray results show that complexes **2**, **3**, **4**, **6** and **7** exhibit a columnar hexagonal phase (D_h) (**2**:$a = 23.70$ Å, $c = 3.42$ Å; **3**:$a = 25.44$Å, $c = 3.38$ Å; **4**:$a = 27.10$ Å, $c = 3.42$ Å; **6**:$a = 31.42$ Å, $c = 3.39$ Å). The structure of all those phases will be discussed in more detail later.

CONCLUSION

Here, we have described a new synthesis of long-chain substituted dibenzotetraaza[14]annulene nickel complexes. The most remarkable point of this synthesis is the preparation, in high yields, of 4, 5-dialkoxy 1,2-dinitrobenzene in a single step (usually, the dinitration of 1, 2-dialkoxybenzene is performed in several steps[31]). The cleavage of the chains can be avoided by using a biphasic medium. The reduction ($NaBH_4$–$NiCl_2$) of those dinitro compounds leads to masked *o*-phenylenediamines which are condensed

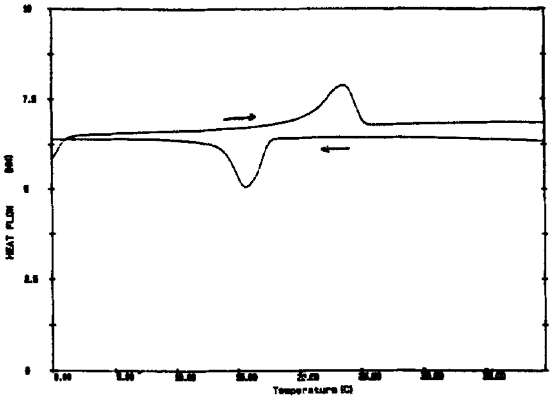


FIGURE 4 DSC curve of compound **6**.

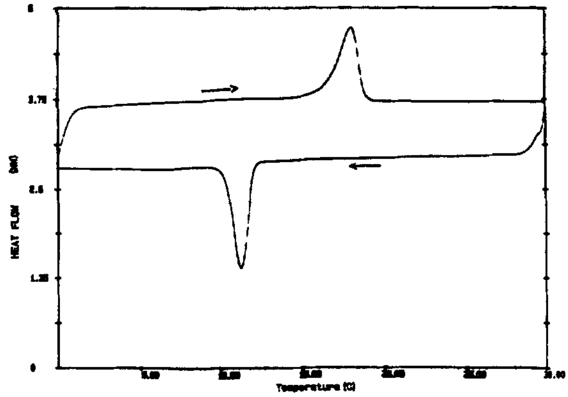


FIGURE 5 DSC curve of compound **7**.

with substituted vinamidinium salts leading to the desired nickel complexes.
Work is in progress to increase the yield of this last step (and to identify the nature of the intermediate **10**).

The dibenzotetraaza[14]annulene nickel complexes with four and six long chains have a very high clearing point and exhibit hexagonal columnar phases (identified by X-ray experiments and by microscopic observations). Increasing the number of long paraffinic chains leads, as expected, to the lowering of the clearing point.

EXPERIMENTAL

Pyrocatechol, 4-hydroxy, 3,4-dihydroxy and 4-bromophenyl acetic acids were purchased from Aldrich and used without purification.

PLATE 1 Compound **2**, $T = 214°\mathrm{C}$. See Color Plate XI.

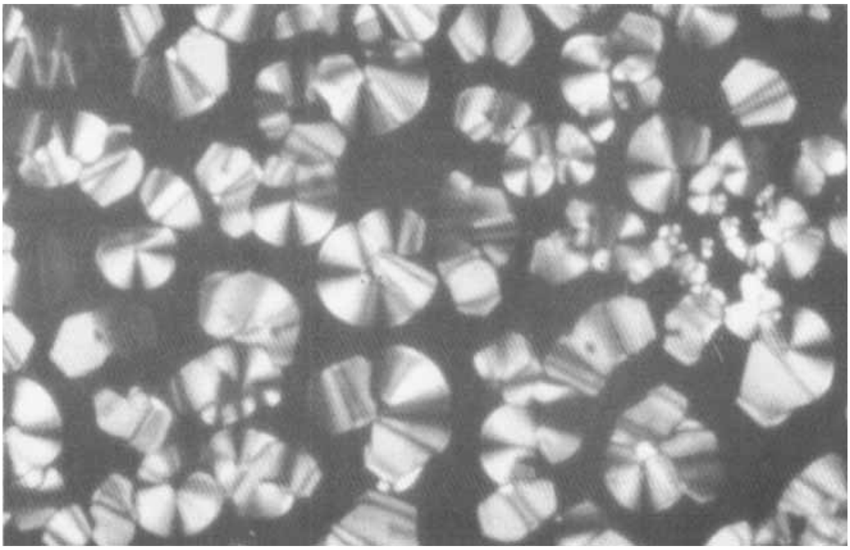


PLATE 2 Compound **3**, room temperature. See Color Plate XII.

^{1}H NMR Spectra were recorded on a Varian EM390 spectrometer at 300 MHz, in $CDCl_3$, with TMS as reference. IR spectra were recorded on a FT-IR Perkin-Elmer spectrometer in KBr pellets. UV-Visible spectra were obtained from a Kontron-Uvikon spectrophotometer in CH_2Cl_2. Melting points were determined using a Leitz Orthoplan microscope equipped with a Mettler FP80 hot stage.

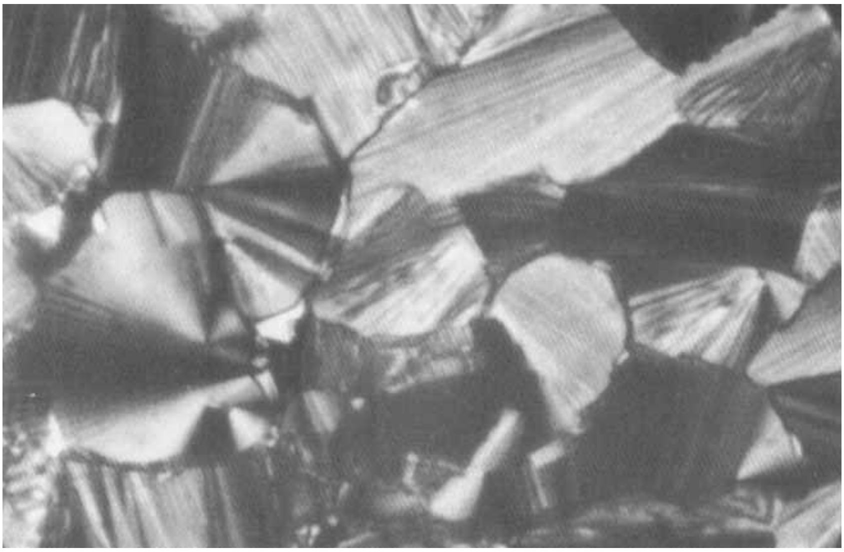


PLATE 3 Compound **3**, $T = 81.2°C$. See Color Plate XIII.

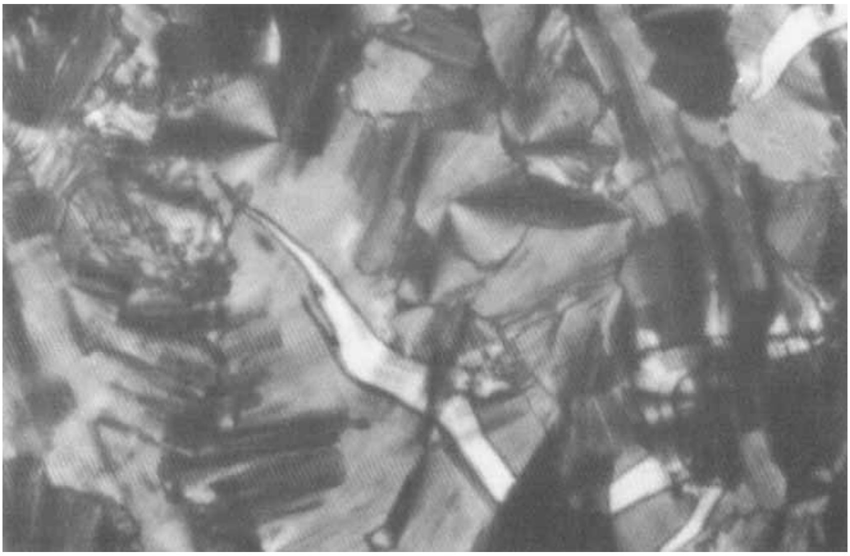


PLATE 4 Compound **3**, $T = 120°C$. See Color Plate XIV.

The etherification of pyrocatechol[27] and the synthesis of the vinamidinium salts have already been described.[26]

1,2-Dinitro 4,5-dialkoxybenzene 9

The pyrocatechol ether (22 mmol) in dichloromethane (60 cm^3) was added dropwise to 70% nitric acid (132 mmol). After 5 min stirring, sulfuric acid (88 mmol) was

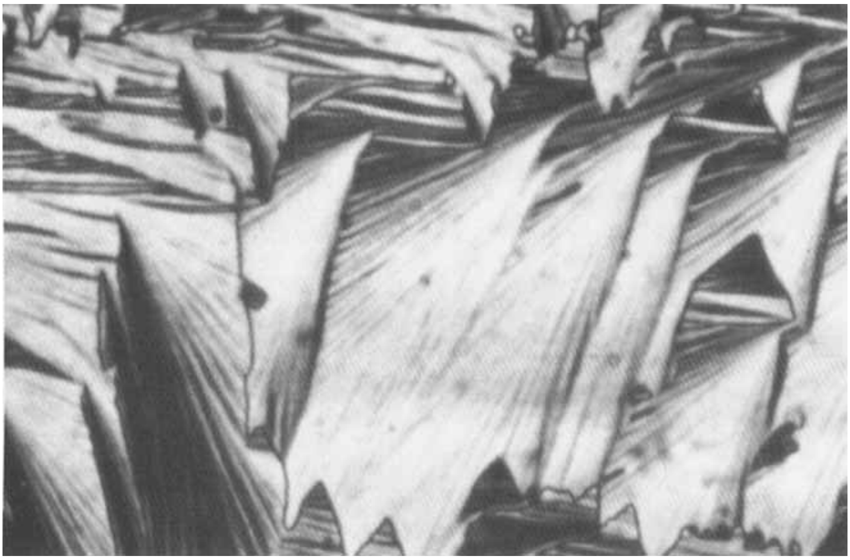


PLATE 5 Compound **4**, $T = 179°C$. See Color Plate XV.

PLATE 6 Compound **4**, $T = 179°C$. See Color Plate XVI.

PLATE 7 Compound **6**, $T = 100°C$. See Color Plate XVII.

added dropwise. The mixture was then stirred for 90 min at room temperature, and poured into crushed ice. The dinitro compound was extracted with CH_2Cl_2, the organic phases were washed until neutral and dried over Phase Separator (PS) paper (Whatman). After evaporation of the solvent under vacuum, the yellow solid was recrystallized from ethanol.

Yields, melting points and λ_{max} are given in Table 1.

4-Alkoxy and 3,4-dialkoxyphenylacetic acids

The etherification and esterification of mono (or di) hydroxyphenylacetic acid were performed as already described,[23] using two (or three) equivalents of alkyl bromide. The obtained esters were saponified with an ethanolic solution of potassium hydroxide.

4-dodecyloxyphenylacetic acid:

Yield: 85%.

Melting point: 87°C.

IR $\upsilon(cm^{-1})$, (KBr): 2953 (*s*, CH_3); 2850 (*s*, CH_2); 1703 (*m*, C=*O*); 1618 and 1517 (*m*, aromatics); 1252 (*s*, C—O—C) and 1037 (*w*, C—O—C).

NMR ^1H δ(ppm), ($CDCl_3$/TMS): 0.85 (*t*, 3H, CH_2CH_3); 1.2 (*m*, 18H, $(CH_2)_9$); 1.6 (*q*, 2H, —OCH_2—CH_2); 3.5 (*s*, 2H, CH_2COO); 3.9 (*t*, 2H, OCH_2); 6.8 (*d*, 2H aromatic); 7.2 (*d*, 2H aromatic); 10.0 (broad *s*, OH).

3,4-didodecyloxyphenylacetic acid:

Yield: 67%.

Melting point: 72°C.

IR $\nu(cm^{-1})$, (KBr): 2920(*s*,CH_3); 2850(*s*,CH_2); 1720(*s*,C=O); 1591 and 1519 (*m*, aromatics); 1394(*s*,C—O); 1266(*w*,C—O ether).

NMR ^{1}H δ(ppm), ($CDCl_3$/TMS): 0.85 (*t*,6H, CH_2CH_3); 1.2 (*m*, 36H, 2(CH_2)$_9$); 1.6 (q, 4H,—OCH_2—CH_2); 3.5 (*s*, 2H, CH_2COO); 3.9 (2*t*, 4H, OCH_2); 5.9 (broad s, OH); 6.8 (*s*,3H, aromatic).

Vinamidinium Salts

1,1,5,5-tetramethyl 3-(4′-dodecyloxyphenyl) 1,5-diazapentadienium tetrafluoroborate:

NMR ^{1}H δ(ppm), ($CDCl_3$/TMS):0.8 (*t*, 3H, CH_3); 1.2 (*m*, 18H, (CH_2)$_9$); 1.8 (*s*, 2H, CH_2); 2.5 (*s*, 6H, N(CH_3)$_2$); 3.5 (*s*, 6H, N(CH_3)$_2$); 3.8 (*t*, 2H, OCH_2); 6.9 (d, 2H, aromatic); 7.8 (*s*,2H, diazapentadienium).

Melting point: 103–104°C.

IR υ(cm^{-1}), (KBr):3437 and 3356 (*m*, CH_2)$_{11}$; 2938 (*s*, CH_3); 1646 (*w*,diazapentadienium); 1590 (*s*, C_6H_5); 1397 (*s*, C-C_6H_5); 1084 (*s*, BF_4^-).

Microanalysis ; Found (calculated):**C**:63.22 (63,23);**H**: 9.26 (9.15); **N**: 5.89 (5.74).

1,1,5,5-tetramethyl 3-(3′,4′-didodecyloxyphenyl) 1,5-diazapentadienium tetrafluoroborate:

NMR ^{1}H δ(ppm), ($CDCl_3$/TMS):0.8 (*t*, 6H, CH_3); 1.2 (*m*, 36H, (CH_2)$_9$); 1.8 (*s*, 4H, CH_2); 2.5 (*s*, 6H, N(CH_3)$_2$); 3.5 (*s*, 6H, N(CH_3)$_2$); 3.8 (*t*, 4H, OCH_2); 6.9 (*d*, 2H, aromatic); 7.2 (*d*, 2H, aromatic); 7.8 (*s*, 2H, diazapentadienium).

Melting point: 85°C.

IR υ(cm^{-1}), (KBr): 3437 and 3356 (*m*, CH_2)$_{11}$; 2938 (*s*, CH_3); 1646 (w, diazapentadienium); 1590 (*s*, C_6H_5); 1397 (*m*, C—C_6H_5); 1084 (*s*, BF_4^-).

1,1,5,5-tetramethyl 3-(4′-bromophenyl) 1,5-diazapentadienium tetrafluoroborate:

Yield: 44%.

NMR ^{1}H δ(ppm), ($CDCl_3$/TMS): 0.8 (*t*, 6H, CH_3); 1.2 (*m*, 36H, (CH_2)$_9$); 1.8 (*s*, 4H, CH_2); 2.5 (*s*, 6H, N(CH_3)$_2$); 3.4 (*s*, 6H, N(CH_3)$_2$); 7.2 (d, 2H, aromatic); 7.6 (d, 2H, aromatic); 7.8 (*s*,2H, diazapentadienium).

Melting point: 175°C.

IR υ(cm^{-1}), (KBr):2932 (*m*, CH_3); 1614 (*w*, diazapentadienium); 1590 (*s*,C_6H_5); 1404 (*s*,C—C_6H_5); 1084 (*s*,BF_4^-).

Dibenzotetraaza[14]Annulene Nickel Complexes 2–7

General procedure

The appropriate dinitro compound (3,8 mmol), nickel chloride hexahydrate (15,2 mmol) and methanol (40 cm^3) were placed in a three-necked flask. Sodium borohydride (62,2 mmol) was added gradually, as an exothermic reaction took place. The reaction was followed by tlc (CH_2Cl_2 as eluent) and stopped after the complete disappearance of the dinitro compound. A black, insoluble precipitate was filtered, washed with methanol and used without further purification. This compound **10** and the vinamidinium salt **8** (3,8 mmol) in dimethylformamide (10 ml) were refluxed for 15h. After cooling, the precipitate was filtered, washed with water and extracted with hot dichloromethane. The complex was purified by chromatography on silica gel (eluent: CH_2Cl_2/pentane 1:1) and recrystallized twice from ethyl acetate and once from toluene. Compound **5** was purified by chromatography and crystallisation from dichloromethane.

Yields and characterizations are given in Table 2.

Mass spectrum of compound **4**, m/z:1233 (MH^+), 1262 ($MC_2H_5^+$), 1231 (M – 1).

NMR ^{1}H δ(ppm), ($CDCl_3$/TMS):0.85 (*t*,6H, CH_3); 1.25 (*m*,2(n – 2)H, $(CH_2)_{n-2}$); 1.8 (*s*, 4H, CH_2—CH_2—O); 3.8 (*s*, 4H,—CH_2O); 6.5 if *n* = 8, 6.6 if *n* = 10, 6.7 if *n* = 12 (*s*, 4H,—N—CH = C(Ar)—CH = N—), 6.8–7.3 (*m*,14H, $C_6H_5 + C_6H_2(OR)_2$).

IR ν(cm^{-1}), (KBr):3200 (*w*,CH_2) and 2900 (*s*,CH_3) 1608 (*w*,—N—C=C—C=N—); 1590 (*s*,C_6H_5).

Microanalysis:Found (calculated)

Compound **3**:C: 74.72 (74.91); **H**: 8.96 (9.16); **N**: 4.99 (4.99).
Compound **4**:C: 75.59 (75.89); **H**: 9.43 (9.63); **N**: 4.42 (4.54).
Compound **5**:C: 67.29 (67.45); **H**: 8.40 (8.19); **N**: 4.02 (4.05).
Compound **6**:C: 75.63 (76.42); **H**: 10.23 (10.44); **N**: 3.54 (3.49).

References

1. V. Gionis, H. Strazlecka, M. Veber, R. Kormann and L. Zuppiroli, *Mol. Cryst. Liq. Cryst.*; **137**, 365–372 (1986); and references therein.
2. M. Veber, R. Fugnitto and H. Strzelecka, *Mol. Cryst.; Liq. Cryst*, **96**, 221–227 (1983).
3. M. Veber, P. Davidson, C. Jallabert, A. M. Levelut and H. Strzelecka, *Mol. Cryst. Liq. Cryst. Letters*, **5(1)**, 1–7 (1987).
4. F. A. Cotton and J. Czuchajowska, *Polyhedron*, **9(21)**, 2553–2566 (1990), and references therein.
5. J. D. Koola and J. K. Kochi, *Inorg. Chem.*, **26**, 908 (1987).
6. H. Behret, W. Clauberg and G. Sanstede, *Z. Phys. Chem.*, **113**, 97 (1978).
7. L. S. Lin, T. J. Marks, C. R. Kannewurf, J. W. Lyding, M. S. McClure, M. T. Ratajack and T. C. Whang, *J. Chem. Soc. Chem. Commun.*, 954–955 (1980).
8. M. Hunziker, B. Hilti and G. Rihs, *Helv. Chim. Acta*, **64(1)**, 82–89 (1981).
9. M. Hunziker, H. Lœliger, G. Rihs and B. Hilti, *Helv. Chim. Acta*, **64(8)**, 2544–2554 (1981).
10. S. C. Tang, G. N. Weinstein and R. H. Holm, *J. Amer. Chem. Soc.*, **95(2)**, 613–614 (1973).
11. S. C. Tang, S. Koch, G. N. Weinstein, R. W. Lane and R. H. Holm, *Inorg. Chem.*, **12(11)**, 2589–2595 (1973).
12. Akashi Kageyasu and Suga Nobuhiko, *Eur. Pat. Appl.*, EP 73,456; CA, **99**, 88241g (1983).
13. M. Hunziker, *Eur. Pat. Appl.*, EP 162,804; CA, **106**, 41716p (1987).
14. R. Müller and D. Wöhrle, *Makromol. Chem.*, **176**, 2775–2795 (1975).
15. R. Gompper, C. Illek and K. Polborn, *Tetrahedron. Lett.*, **33(28)**, 3989–3992 (1992).
16. a/F. Adams, R. Gompper and E. Kujath, *Angew. Chem. Int. Ed. Engl.*, **28**, 1060–1062 (1989); b/F. Adams and R. Gompper, *Angew. Chem. Int. Ed. Engl.*, **28**, 1063–1065 (1989).
17. A. R. Cutler and D. Dolphin, *J. Coord. Chem.*, **6**, 59–61 (1976).
18. C. W. G. Ansell, M. F. H. Y. J. Chung, M. Mc Partlin and P. A. Tasker, *J. Chem. Soc. Dalton Trans.*, 2113–2116 (1982).
19. a/A. M. Giround-Godquin and P. M. Maitlis, *Angew. Chem. Int. Ed. Engl.*, **30**, 375–402 (1991), and references therein ; b/ S. A. Hudson and P. M. Maitlis, *Chem. Rev.*, **93**, 861–885 (1993) and references therein.
20. P. Espinet, M. A. Esteruelas, L. A. Oro, J. L. Serrano and E. Sola, *Coord. Chem. Rev.*, **117**, 215–274 (1992), and references therein.
21. A. P. Polishchuk, T. V. Timofeeva, *Russ. Chem. Rev.*, **62(4)**, 291–321 (1993).
22. H. Strzelecka, C. Jallabert and M. Veber, *Mol. Cryst. Liq. Cryst.*, **156**, 355–359 (1988).
23. M. Veber, P. Sotta, P. Davidson, A. M. Levelut, C. Jallabert and H. Strzelecka, *J. Phys. France*, **51**, 1283–1301 (1990).
24. M. Veber, C. Jallabert, H. Strzelecka, O. Jullien and P. Davidson, *Liq. Cryst.*, **8(6)**, 775–785 (1990).
25. A. Zinsou, M. Veber, H. Strzelecka, C. Jallabert and P. Fourré, *New J. Chem.*, **17**, 309–313 (1993).
26. A. Zinsou, M. Veber, H. Strzelecka, C. Jallabert and A. M. Levelut, *Liq. Cryst.*, accepted for publication.
27. H. Strzelecka, C. Jallabert, M. Veber and J. Malthête, *Mol. Cryst. Liq. Cryst.*, **156**, 347–353 (1988).
28. A. Nose and T. Kudo, *Chem. Pharm. Bull.*, **29(4)**, 1159–1161 (1981).
29. H. H. Seltzman and B. D. Berrang, *Tetrahedron. Lett.*, **34(19)**, 3083–3086 (1993).
30. W. Hieber, C. Schliessmann and K. Ries, *Z. Anorg. Allg. Chem.*, **180**, 89–104 (1929).
31. G. M. Coppola, G. E. Hardtmann and B. S. Huegi, *J. Heterocycl. Chem.*, **11**, 51 (1974).