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## Liquid Crystals

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### Liquid-crystalline compounds with lateral aromatic branches

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## Liquid-crystalline compounds with lateral aromatic branches†

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We present the synthesis of 2-substituted hydroquinone-bisbenzoates which have large 2-substituents containing aromatic and other ring systems. Contrary to the general accepted opinion these large lateral substituents which cause remarkable deviations from the rod-like shape of the molecules do not prevent the liquid-crystalline properties, the compounds are nematic and smectic. The influence of different chemical groups on the liquid-crystalline properties is investigated systematically. The compounds tend to exhibit the glassy nematic state above room temperature. This property may be used for the construction of thermo-electrooptic devices.

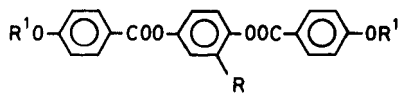
### 1. Introduction

In previous papers [1–8, 15] we reported the influence of lateral long chain substituents on the liquid-crystalline behaviour of three-ring compounds. The anomalous trend of the clearing temperature  $T_{NI}$  with increasing lateral alkyl chain, namely the convergence of  $T_{NI}$  after a strong decrease for the first members, may be explained by a tendency of the alkyl chains to an orientation parallel to the molecular long axes. There are several arguments which support this explanation; especially the behaviour of the length-to-breadth ratio which is consistent with the molecular-statistical theories of nematic liquid crystals [10]. By X-ray investigations we were able to prove the existence of cybotactic groups in the 1,4-bis-(4-*n*-substituted-benzoyloxy)-2-*n*-alkyl-benzenes (I), however, the compounds did not exhibit smectic behaviour [7]. Now we change the lateral substitutions in such a manner that substituted or unsubstituted aromatic rings (supplemented by some other ring systems) are attached by a spacer group *X* at the middle aromatic ring of the basic molecule according to formula (VI). According to the generally accepted opinion [20] these bulky substituents should reduce the mesogeneity to a large extent or even prevent it completely.

Former investigations have shown that an additional branching and therefore enhanced bulkiness of the lateral substituents causes further depression of the clearing temperature. For example, the  $T_{NI}$  of the *t*-butyl compounds (II) and the esters of 2-methyl-butanol (III) are lower than those of the *n*-alkyl isomers. However, even two alkyl chains in the oxime esters (IV) do not completely prevent the occurrence of nematic phases. According to Cox *et al.* [12] the esters of the 2-phenylhydroquinone

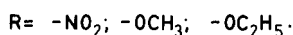
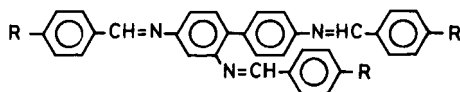
† Thermotropic liquid-crystalline compounds with lateral long chain substituents: Part IX.

(V) are liquid-crystalline. Unfortunately the temperatures in the tables do not agree with those in the graphical plot [12], and so a clear interpretation of the results is difficult.



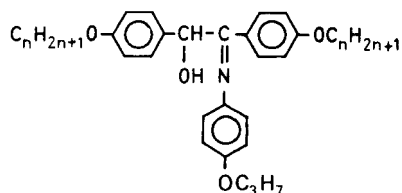
- |       |  |      |
|-------|--|------|
| (I)   | $C_nH_{2n+1}$  | [1]  |
| (II)  | tert.-butyl  | [11] |
| (III) | $COOCH_2(CH_2)_2C_2H_5$  | [4]  |
| (IV)  | $COO-N=C \begin{cases} C_nH_{2n+1} \\ C_nH_{2n+1} \end{cases}$ | [4]  |
| (V)   | $C_6H_5$   | [12] |
| (VI)  |  |      |

Already in the research group of Vorländer some liquid-crystalline compounds with lateral aromatic substituents have been synthesized by Mauerhoff and Kuhrmann. Mauerhoff [36] described three compounds of the type

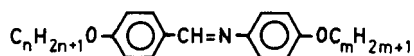


These substances are nematic with clearing temperatures above  $200^\circ C$ . Kuhrmann [13] prepared liquid crystals with lateral aromatic substituents. However close inspection of her results shows inconsistencies in the clearing temperatures and possibly an incorrect interpretation of the synthetic results (e.g. according to the reaction route described by Kuhrmann the formulae of compounds No. 2522–2527, 2589–2590 in [14] should bear two substituents attached to the middle benzene ring in reverse positions).

Gallardo and Müller [16] have described the reaction of 4-*n*-alkoxybenzoins with 4-*n*-propylaniline which are claimed to yield compounds of the type



The compounds are nematic, their nematic–isotropic transition temperatures of above  $100^\circ C$  are anomalously high for a branched two ring compound and do not seem to be compatible with the proposed structure. In fact, the  $T_{NI}$  are very similar to those of the 4-*n*-alkoxy-*N*-(4-*n*-alkoxy-benzylidene)-anilines (see [14, p. 97])



and there are hints that the reaction did not follow the expected route. Detailed work concerning this discrepancy is under progress and will be published elsewhere.

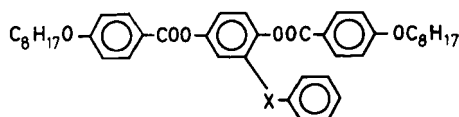
## 2. Results and discussion

We have synthesized 47 compounds of the general structure (VI); details of the synthesis are given in §4. The transition temperatures of the new compounds are given in tables 1–4. In order to have a better comparison of the influence of the lateral substituents on the liquid-crystalline behaviour we have used alkyloxy groups of constant length ( $C_8$ ) in the reference molecule.

A 2-benzoyl or phenylester group in compounds **1** and **2** (cf. table 1) in comparison to the 2-phenyl-hydroquinone bis-(4-*n*-octyloxybenzoate) of Cox *et al.* [12] (C 81 N 77 I) decreases  $T_{\text{NI}}$ . This decrease is enhanced further by substitution of the lateral phenyl ring in the 4-position for compounds **25–27** (cf. table 3).

Esterification of different  $\omega$ -phenylalkanols with 2,5-bis-(4-*n*-octyloxy-benzoyloxy)-benzoyl chloride yields compounds **3–8** (cf. table 1). Their transition temperatures are plotted in figure 1. The insertion of methylene groups makes the spacer  $X$  more flexible and so allows the orientation of the lateral phenyl group more or less parallel to the reference molecule. Spacers with an odd number  $\geq 3$  of single segments (the  $-\text{CO}-$  and  $-\text{O}-$  of the ester group are included in the calculation) compensate the angle of the substituent made with the molecular long axis and cause the higher  $T_{\text{NI}}$  in the strongly alternating curve shown in figure 1. The regions of stable nematic existence of about 30 K in **13** and **15** and the  $T_{\text{NI}}$  with values above 90°C prove that

Table 1. The influence of the spacer  $X$  on the transition temperatures (in °C).



No.	$X$	C	N	I	
<b>1</b>	$-\text{CO}-$	●	85	● (58)	●
<b>2</b>	$-\text{COO}-$	●	91	● (65)	●
<b>3</b>	$-\text{COO}-\text{CH}_2-$	●	98	● 98	●
<b>4</b>	$-\text{COO}-(\text{CH}_2)_2-$	●	73	● 80.5	●
<b>5</b>	$-\text{COO}-(\text{CH}_2)_3-$	●	68	● 96	●
<b>6</b>	$-\text{COO}-(\text{CH}_2)_4-$	●	80	● (76)	●
<b>7</b>	$-\text{COO}-(\text{CH}_2)_5-$	●	62	● 92	●
<b>8</b>	$-\text{COO}-(\text{CH}_2)_6-$	●	70	● 77	●
<b>9</b>	$-\text{COS}-\text{CH}_2-$	●	78	● 80	●
<b>10</b>	$-\text{CO}-\text{NH}-\text{CH}_2-$	●	121	● (112)	●
<b>11</b>	$-\text{COO}-\text{CH}_2-\text{CH}=\text{CH}-$	●	107	● 130.5	●
<b>12</b>	$-\text{COO}-\text{CH}_2-\text{CH}_2-\text{O}-$	●	76	● 109	●
<b>13</b>	$-\text{COO}-\text{CH}_2-\text{CH}_2-\text{N}-$   $\text{C}_2\text{H}_5$	●	60	● 76	●
<b>14</b>	$-\text{COO}-\text{CH}_2-\text{CH}_2-\text{N}-$   $\text{C}_2\text{H}_4\text{CN}$	●	72	● (50)	●

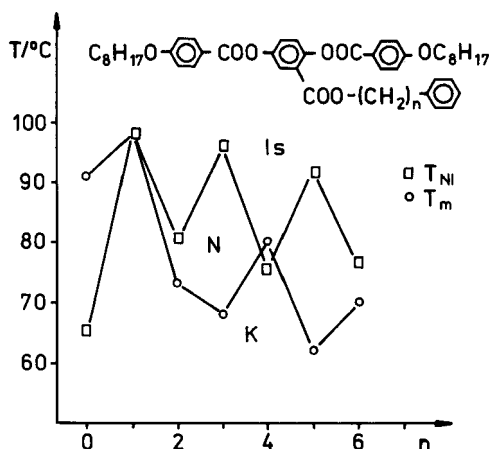
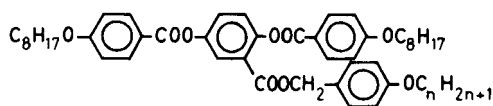


Figure 1. Transition temperatures versus spacer length in the homologous series of the  $\omega$ -alkylphenyl 2,5-bis-(4-*n*-octyloxybenzoyloxy)-benzoates.  $T_{NI}$ , nematic-isotropic transition temperatures;  $T_m$ , melting temperatures.

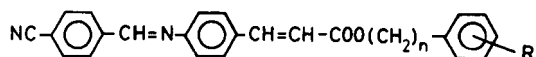
Table 2. The influence of the length of the substituent  $R^2$  on the liquid-crystalline properties (in °C).



No.	<i>n</i>	C	$S_C$	N	I
15	1	●	85	—	●
16	2	●	92	—	●
17	3	●	82	—	●
18	4	●	79	● (37)	●
19	5	●	75	● (44.5)	●
20	6	●	84	● (55)	●
21	7	●	80	● (61)	●
22	8	●	85	● (67)	●
23	9	●	93	● (71.5)	●
24	10	●	93	● (75)	●

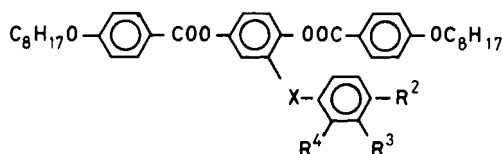
the bonding of lateral phenyl rings to the reference molecule by flexible spacers is a useful concept for the synthesis of liquid crystals.

The alternating behaviour of compounds 3–8 may be compared with that of the substances synthesized by Gray [37]



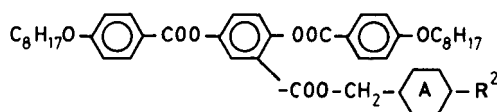
In this series the  $T_{NI}$  alternate very strongly due to the terminal ring of the ester moving in and out of line with the long axis of the rest of the molecule. The alternation does diminish in magnitude as the length of the spacer  $-(CH_2)_n-$  increases. This has

Table 3. The influence of different spacers and substitution of the lateral aromatic moiety (temperatures in °C).



	<i>X</i>	<i>R</i> <sup>2</sup>	<i>R</i> <sup>3</sup>	<i>R</i> <sup>4</sup>	<i>S</i> <sub>C</sub>	<i>S</i> <sub>A</sub>	<i>N</i>	<i>I</i>
25	-COO-	CN	H	H	● 107	-	- ● (50)	●
26	-COO-	CH <sub>2</sub> CN	H	H	● 91	-	- ● (48)	●
27	-COO-	CH <sub>2</sub> CH <sub>2</sub> CN	H	H	● 112	-	- ● (39)	●
28	-COO-N=C-	C <sub>5</sub> H <sub>11</sub>	H	H	● 74	-	- ● (22)	●
29	-C(=N)-N(CH <sub>3</sub> )-OOC-	C <sub>5</sub> H <sub>11</sub>	H	H	● 58	-	- ● (17)	●
30	-CH=N-OOC-	C <sub>5</sub> H <sub>11</sub>	H	H	● 75	-	- ● (70)	●
31	-COO-N=CH-	CN	H	H	● 110	-	- ● (66)	●
32	-COO-N=CH-	OC <sub>6</sub> H <sub>13</sub>	H	H	● 87	-	- ● 92	●
33	-COO-CH <sub>2</sub> -	Br	H	H	● 95	● (61)	- ● 121	●
34	-COO-CH <sub>2</sub> -	CH <sub>3</sub>	H	H	● 82	-	- ● 113	●
35	-COO-CH <sub>2</sub> -	iso-C <sub>3</sub> H <sub>7</sub>	H	H	● 75	-	- ● 95	●
36	-COO-CH <sub>2</sub> -	CN	H	H	● 91	-	● 152	- ●
37	-COOCH <sub>2</sub> -CH=CH-	NO <sub>2</sub>	H	H	● 164	-	● 195	- ●
38	-COO-CH <sub>2</sub> -	NO <sub>2</sub>	H	H	● 94	-	● 163	- ●
39	-COO-CH <sub>2</sub> -	H	NO <sub>2</sub>	H	● 80	● 82	● 96	● 112.5
40	-COO-CH <sub>2</sub> -	H	H	NO <sub>2</sub>	● 108	-	- ● (80)	●
41	-COO-CH <sub>2</sub> -	-O-CH <sub>2</sub> -O-	H	H	● 99	-	- ● 112	●
42	-COO-CH <sub>2</sub> -	OC <sub>8</sub> H <sub>17</sub>	OCH <sub>3</sub>	H	● 78	-	- ● 80.5	●
43	-COO-CH <sub>2</sub> -	OC <sub>4</sub> H <sub>9</sub>	OC <sub>4</sub> H <sub>9</sub>	H	● 99	-	- ● (73)	●

Table 4. The influence of the terminal ring on the mesomorphic behaviour (temperatures are in °C).



	<i>A</i>	<i>R</i> <sup>2</sup>	<i>K</i>	<i>S</i> <sub>C</sub>	<i>N</i>	<i>I</i>
44		H	●	55	-	● 103
45		H	●	75	-	● 86
46		C <sub>3</sub> H <sub>7</sub>	●	89	-	● 108
47		C <sub>4</sub> H <sub>9</sub>	●	106	● (84)	● 154

been considered as a hint to the greater ability of longer alkylene chains to bend into line with the molecular long axis [37].

Variants of the spacer in structure (VI) are possible; they are investigated preferably in compounds whose spacers consist of three or five segments. Substitution of an oxygen in the lateral ester group of **3** by sulphur in **9** yields a depression of  $T_{NI}$ , introduction of an amide group, **10**, enhances the transition temperatures probably by the formation of hydrogen bonds. The introduction of a carbon-carbon double bond, **11**, between segments 4 and 5 enhances  $T_{NI}$  by 35 K in comparison to the analogous compound with a single bond, **5**. Substitution of the phenyl neighboured methylene in **5** by oxygen yields the phenoxy compound **12**. The advantageous liquid-crystalline properties and the relatively easy synthesis of this moiety persuaded us to perform detailed investigations of the substituent effects in this molecule [19]. The examples **13** and **14** demonstrate that the spacer may be branched itself without loss of mesomorphic properties.

Only by strong supercooling in the isomeric ketoxime esters **28** and **29** can the nematic phases be detected. On the other hand the remarkably higher  $T_{NI}$  of **30–32** proves that a further systematic investigation of the influence of different spacers  $X$  and substituents  $R^2$  in structure (VI) is of interest. Aldoxime esters as **30–32** are suited for this purpose only with restriction since they tend to decompose at higher temperatures [17, 18].

Since spacers with three or five segments in structure (VI) affect especially advantageously the mesomorphic properties, the investigation of the substituent influence in the lateral phenyl ring may be performed systematically. The examples **15–24** (cf. table 2), **33–37** (cf. table 3) show the increase of  $T_{NI}$  by particular *p*-substituents, in contrast to the behaviour of **25–27**.

Figure 2 shows the transition temperatures of the homologous series **15–24**, in which only the length of the alkyloxy group at the lateral substituent is changed. All compounds exhibit enantiotropic nematic phases. It should be emphasized that the  $T_{NI}$  are influenced by only a small amount by the chain length of this substituent, indeed the  $T_{NI}$  of the six homologues **19–24** are nearly constant. The most remarkable feature of this series is the existence of monotropic smectic C phases, since there is a general rule that lateral substituents depress the smectogenic properties much more than the nematogenic ones [1–8, 20]. In lateral substituted three ring compounds only by substitution with small lateral groups such as F, Cl, CH<sub>3</sub>, CN, CHO have weak smectogenic S<sub>C</sub> properties been observed.

In addition the existence of smectic A phases in lateral substituted compounds such as **36–38** is very unusual. In a Preliminary Communication [9] we have described the intercalated structure of the S<sub>A</sub> phases in substances of this type. The publication of further results in similar compounds is in preparation [21].

Examples **38–40** demonstrate the positional isomerism of the nitro group at the phenyl ring without loss of mesomorphic properties. Compound **39** is the first lateral substituted substance with the polymorphism S<sub>C</sub> S<sub>A</sub> N. Some examples of a multiple substitution of the lateral ring are given in **41–43**, further material concerning this problem will be published elsewhere [19].

In table 4 we present the transition temperatures of compounds in which the lateral phenyl ring according to structure (VI) is exchanged by a heterocyclic aromatic ring in **44** or by cyclohexane moieties in **45–47**; these substances are also enantiotropic nematic.

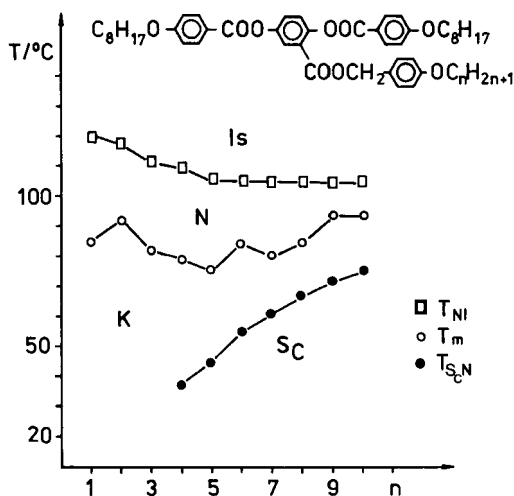


Figure 2. Transition temperatures versus alkyl chain length in the homologous series of the 4-*n*-alkyloxybenzyl 2,5-bis-(4-*n*-octyloxybenzoyloxy)-benzoates.  $T_{ScN}$ , smectic C-nematic transition temperature.

### 3. Conclusions

In a preliminary summary of the results we may conclude that substances of the general formula (VI) with suitably chosen spacer and substituents exhibit liquid-crystalline properties at unexpectedly high temperatures. The flexibility of the spacers is well known in side-chain polymers [26]. On the other hand, by the action of the nematic surrounding the possible rotational isomers of alkyl chains tend to a conformation which yields a stretched molecule [31]. Therefore we conclude, considering especially the influence of the different spacers displayed in figure 1, that the large lateral substituents in compounds with pronounced mesomorphic properties are oriented more or less parallel to the long axes of the reference molecules.

Considering the molecular structure revealed by space-filling models we can observe that the shape is neither rod-like nor disc-like (cf. figure 3), we may speak about a new type of lateral substituted liquid crystal. Compounds in which lateral two-ring or three-ring groups are linked by small spacers at the reference molecule, have some similarity with the ligated Siamese twin molecules [22–25]. On the other hand there exists a relationship to liquid-crystalline polymers in which mesogenic side groups are fixed as lateral substituents to the mesogenic moieties of rigid rod or semi-flexible main-chain polymers [32].

These compounds do not possess only unconventional molecular structure but also unconventional properties. Preliminary X-ray investigations of the  $S_A$  phase of compound **38** point at specialities [9]. The length of the basic molecule is 42 Å, however, the layer thickness of the  $S_A$  phase is only 34.2 Å. This difference has been explained by an intercalation of the layers [9], however, considering the results of additional investigations in several members of the homologous series [33] another structural model seems to be more probable. The peculiarities are due to the unsymmetrical shape of the molecules and the existence of the space filling lateral ring systems which are attached to the reference molecule by a spacer. As the cyclohexane derivatives **45–47** prove, for the mesomorphic properties special interactions between the aromatic parts of the basic molecule and the spacer are not necessary.



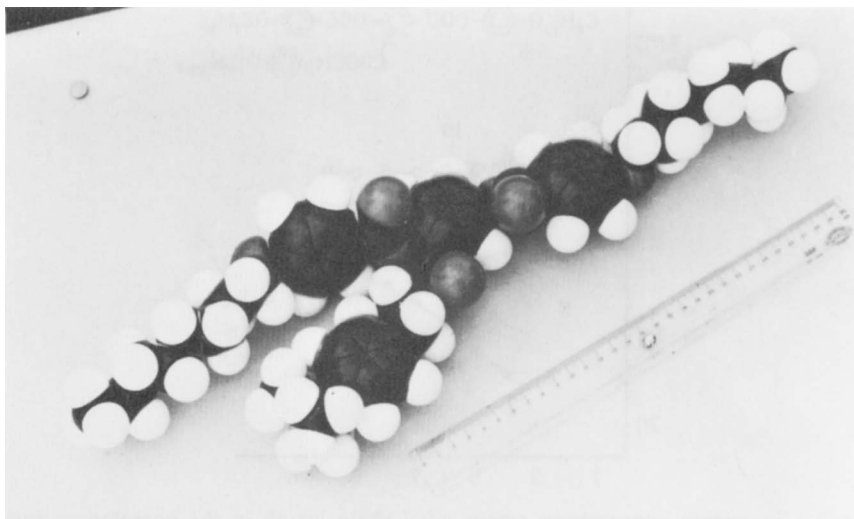


Figure 3. Space-filling model of compound (VI) with the spacer  $X = -\text{COO}(\text{CH}_2)_2-$ .

Consideration of the length-to-breadth ratios of the molecules does not lead to a satisfactory explanation of the high clearing temperatures. This may be demonstrated by the example of the nitro compound **37**. The smectic A–isotropic transition temperature is exactly equal to the  $T_{\text{NI}} = 195^\circ\text{C}$  of the reference molecule hydroquinone-bis-(4-*n*-octyloxybenzoate) [1]. Since the length of  $42 \text{ \AA}$  is equal for both compounds, the space-filling model for **37** shows at least double the breadth for the molecular part which contains the lateral substituent. In the frame of the molecular-statistical theories the smaller length-to-breadth ratio should cause a drastic decrease of the clearing temperature. The possibility of association by the strongly polar nitro group, however, could complicate this simple picture.

It should be emphasized that generally lateral substituted compounds tend to have very high shear viscosities [2]. This behaviour has also been observed qualitatively in the reported new compounds and quantitative measurements are in progress. We want to illustrate this by the behaviour of compound **35**. Rapid cooling of the nematic phase of this substance to temperatures of between  $0$  and  $5^\circ\text{C}$  yields a glassy nematic structure which is metastable without crystallization for several months. The quenched glassy structure is able to preserve an orientation or information which has been impressed on the nematic phase at temperatures above the glass temperature by the action of electric or magnetic fields or interaction with the walls. Using nematic liquid crystals with glass temperatures above room temperature we were able to construct thermo-electrooptical displays which have several advantages compared with similar constructions using smectic A phase [27, 28] or polymeric liquid crystals [29]. Details of this new type of display will be published elsewhere.

#### 4. Synthesis of compounds VI

The reaction of 2-benzoylhydroquinone with 4-*n*-octyloxybenzoylchloride yielded **1**. Esterification of 2,5-bis-(4-*n*-octyloxybenzoyloxy)-benzaloxime (C 110 N 132 I) or -acetophenoxime respectively (C 111 (N 83) I) with 4-*n*-pentylbenzoylchloride produced substances **7** and **8**. All the other compounds were obtained by acylation of

the respective hydroxythiol or amino-substituted reaction partners with 2,5-bis-(4-*n*-octyloxybenzoyloxy)-benzoylchloride. The latter may be produced by oxidation of the 2,5-bis-(4-*n*-octyloxybenzoyloxy)-benzaldehyde [30] with chromium-(VI)-oxide in acetic acid and reaction of the carboxylic acid (C 172–175 I) with thionylchloride. Esterifications were performed in toluene with triethylamine as a base. The necessary  $\omega$ -phenylalkanols [34], substituted phenols [35], oximes [17, 18], N-(2-hydroxyethyl)-anilines, cinnamic alcohols and benzyl alcohols [34] are commercial products or were synthesized by use of well-known procedures.

We thank Dr. C. Tschierske for providing the 4-substituted trans cyclohexylcarbinols necessary for the synthesis of **46** and **47**. Experimental contributions of Dr. G. Pelzl and W. Wedler are gratefully acknowledged.

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