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Mesomorphic properties of phenyl 4-(5-alkyl-1,3,2-dioxaborin-2-yl)benzoates

Influence of terminal and lateral substitution

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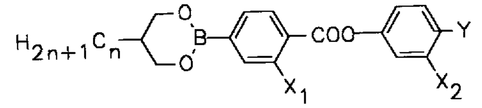
Thirty-eight non-substituted and laterally fluoro-substituted 4-nitro-, 4-isothiocyanato-, 4-fluoro-, 4-trifluoromethyl- and 4-trifluoromethoxyphenyl 4-(5-alkyl-1,3,2-dioxaborin-2-yl) benzoates have been synthesized and their phase transition temperatures and enthalpies of transition measured. The influences of both terminal and lateral substituents in both benzene rings (in the acidic and phenolic moieties) on the phase transition temperatures in general and on the stability of the smectic A phase in particular are compared. The clearing points of the compounds (*a*), the thermal stability of the smectic A phase (*b*) and the tendency to form a smectic A phase (*c*) decrease in the following way:

(*a*) $NCS > NO_2 > OCF_3 > CF_3 > F$,
(*b*) $NO_2 > NCS > OCF_3 > CF_3 > F$,
(*c*) $CF_3 \geq OCF_3 \geq NO_2 > NCS > F$.

Substitution of the central benzene ring by a fluorine atom effectively depresses the smectic A phase. The influence of 4-(5-alkyl-1,3,2-dioxaborin-2-yl)benzoates as components of nematic mixtures on their viscosity, optical and dielectric anisotropy and electro-optical properties is discussed.

1. Introduction

Continuing our interest in compounds containing the 1,3,2-dioxaborinane ring [1–3], we have synthesized compounds of formula **I**:



I

wherein the terminal group *Y* is a fluorine atom or a NO_2, NCS, CF_3, or OCF_3 group, and X_1 and X_2 are hydrogen or fluorine atoms. Other compounds with the terminal substituents: F, CF_3, and OCF_3 have been intensively studied lately [4, 5], but the properties of 1,3,2-dioxaborinanes are not yet as well known as those of, for example, the analogous 1,3-dioxans. Only a few papers describing achiral [1–3, 6, 7] and chiral compounds [8, 9] have been published, but there are further patent publications that recommend dioxaborinane derivatives for improving the threshold and multiplex drive properties of mixtures for TN-LCD [10–13] or PD-LCD [14]. We think that the absence of *cis*- and *trans*-isomers and the lower susceptibility of these compounds to humidity and oxygen in comparison to dioxanes give them a greater prospect in applications.

2. Synthesis

Esters of structure **I** were prepared by direct heating of a 4-(5-alkyl-1,3,2-dioxaborin-2-yl)benzoic acid [1–3] with a 4- or 3,4-substituted phenol (from Aldrich) in the presence of $POCl_3$. Structure I with $Y = NO_2$ is an intermediate for preparing structure I with $Y = NCS$. A good result was obtained when the NCS group was introduced in the following two stages: (*a*) reducing the NO_2 group to the NH_2 group by hydrogen in the presence of palladium catalyst deposited on charcoal; (*b*) treating the NH_2 compound with thiophosgene. The structures of I prepared in this way are consistent with

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Table 1. Mesomorphic properties of structures **I**. Phase transition temperatures (°C) and phase transition enthalpies (cal mol^{-1}) in italics.

No.	*n*	X_1	X_2	*Y*	C_2	C_1	C	S_A	N	I
1	3	F	H	F			● 91·8 *7640*	—	● 104·7 *200*	●
2	3	F	F	F			● 81 *8010*	—	● (61·9) *120*	●
3	3	F	H	OCF_3			● 68·8 *4370*	● 77·5 *40*	● 108·4 *150*	●
4	3	Cl	H	OCF_3			● 50	● (39)	—	●
5	3	F	F	OCF_3			● 23	● 40	● 90	●
6	3	F	H	CF_3		● 86·0 *130*	● 106·7 *6610*	● [76]	—	●
7	3	F	H	NCS			● 102·5 *4640*	—	● 158·5 *180*	●
	3	F	H	CN			● 89	—	● 174	●
8	4	H	H	F			● 99·5 *6620*	● (62·3)	● 112·0 *120*	●
9	4	F	H	F			● 65·0 *4180*	—	● 85·8 *140*	●
10	4	H	F	F			● 97·5 *6250*	● (73·4) *160*	● (92·5) *180*	●
11	4	F	F	F			● 67·4 *6260*	—	● (59·9) *90*	●
12	4	H	H	OCF_3			● 80·6 *4800*	● 134·8 *530*	● 137·7 *340*	●
13	4	F	H	OCF_3			● 73·5 *5640*	● 96·2 *240*	● 107·5 *190*	●
14	4	F	H	CF_3			● 93·0	● (87·0)	—	●
15	4	H	H	NO_2‡			● 128	● 145	● 183	●
16	4	F	H	NO_2‡			● 73	● 107	● 146·5	●
17	4	H	F	NO_2‡			● 119	● 120	● 149	●
18	4	F	F	NO_2‡			● 62	● 105	● 114	●
19	4	H	H	NCS		● 80·2 *1460*	● 130·2 *6140*	● (124·5)	● 186·1	●
20	4	F	H	NCS			● 96·1 *5520*	—	● 161·6 *230*	●
	4	F	H	CN			● 95	—	● 168	●
21	4	H	F	NCS		● 87·4 *810*	● 130·0 *5000*	● 139·4 *170*	● 175·8 *200*	●
	4	H	F	CN			● 103·0	—	● 164	●
22	4	F	F	NCS			● 69·4 *7180*	● 90·2 *50*	● 151·5 *250*	●
	4	F	F	CN			64	—	● 143	●
23	5	H	H	F	● 65·7 *190*	● 70·4 *1420*	● 75·3 *4140*	● 80·6 *170*	● 118 *230*	●
24	5	F	H	F		● 55·5 *1700*	● 61·6 *4850*	—	● 80·3 *110*	●
25	5	H	F	F			● 69·2 *3690*	● 85·0 *240*	● 99·2 *140*	●
26	5	F	F	F			● 50·1 *4860*	● (44·5) *90*	● 70·5 *140*	●

Table 1 (*continued*).

No.	n	X_1	X_2	Y	C_2	C_1	C	S_A	N	I
27	5	H	H	OCF_3			● 68·9 *3280*	● 138·1 *950*	—	●
28	5	F	H	OCF_3			● 48·4 *7410*	● 111·0 *510*	● 113·9 *385*	●
29	5	H	H	CF_3	● 71·5 *320*	● 75 *1340*	● 89·5 *3770*	● 135·2 *1250*	—	●
30	5	F	H	CF_3			● 74·4 *4540*	● 102·8 *440*	—	●
31	5	H	H	NO_2‡			● 118	● 155	● 180	●
32	5	F	H	NO_2‡			● 82	● 126	● 152	●
33	5	H	F	NO_2‡			● 98	● 125	● 147	●
34	5	F	F	NO_2‡			● 59	● 105	* 117·5	●
35	5	H	H	NCS			● 131·1 *7130*	● 145·7 *80*	● 188 *360*	●
36	5	F	H	NCS			● 104·0 *6850*	—	● 163·5 *310*	●
†	5	F	H	CN			● 94	—	● 167	●
37	5	H	F	NCS		● 51·0 *1200*	● 115·8 *4590*	● 151·1 *180*	● 178·0 *240*	●
38	5	F	F	NCS			● 49·4 *5920*	● 110·7 *70*	● 155·4 *280*	●
†	5	F	F	CN			● 63	—	● 142	●

†Cyano analogues according to [1]; ( ) denotes a monotropic transition temperature; [ ] denotes a virtual transition temperature. ‡Phase transitions of nitro compounds are given as rounded numbers because they were not purified as rigidly as the other compounds.

analytical data, including combustion analysis, and IR and ^1H NMR spectra. The spectral data of our compounds with the 2,5-disubstituted dioxaborinane ring are similar to those reported [6].

3. Results

3.1. *Phase transition temperatures and enthalpies*

Phase transition temperatures and enthalpies are given in table 1. They were measured using a Linkam heating stage, in conjunction with a polarizing PZO microscope, and also using a Setaram DSC92. Liquid crystalline phases were identified by the texture seen in the polarizing microscope and additionally by X-ray investigations.

X-ray diffraction patterns were registered at temperatures $T - T_{S_A-N} = -20°C$ for the compounds of structure **I** numbered in table 1 as **28** and **37**. Both a Guinier symmetrical focusing powder camera with Co_{K_α} radiation and a Laue flat plate camera with Cu_{K_α} radiation for samples oriented by a magnetic field were used. In each case the free surface of the sample was probed by the X-rays [15].

The X-ray patterns of the compounds with $Y = OCF_3$ or NCS were typical for the smectic A_1 phase showing one pair of non-diffuse symmetrical spots and corresponding to a layer spacing equal to the length of the molecules [16]. For **28**, layer thickness $d = 25·0$ Å, molecular length $l = 24·4$ Å, $d/l = 1·02$, and for **37**, $d = 25·5$ Å, $l = 24·5$ Å, $d/l = 1·04$.

In the non-laterally substituted structure **I** ($X_1 = X_2 = H$; alkyl chain length $n = 4$ or 5) only nematic and smectic A phases are observed. The temperature ranges of these phases depend on the type of terminal substituent Y and the length of the alkyl tail. For $Y = CF_3$ a nematic phase is not observed and for $Y = OCF_3$ this phase is observed over a short temperature range, and only when $n = 4$. In the compound with a longer alkyl chain, a direct transition from the smectic A phase to the isotropic phase occurs. The thermal stability of the mesophase as given by the clearing point (T_{N-I} or T_{S_A-I}) decreases in the following way: $NCS > NO_2 > OCF_3 > CF_3 > F$. The thermal stability of the smectic A phase demonstrated by the phase transition temperature T_{S_A-N} or T_{S_A-I} decreases in the order: $NO_2 > NCS > OCF_3 > CF_3 > F$. A comparison of the ratio of the temperatures T_{S_A-I}/T_{N-I} and of the alkyl length in structures **I** for which the smectic A phase appears for the first time shows that the smectic tendencies decrease in the order: $CF_3 > OCF_3 > NO_2 > NCS > F$. The observed influence of the terminal substituents Y on the stability of the N and S_A phases in these 4-(5-alkyl-1,3,2-dioxaborin-2-yl)benzoates is similar to that observed in similar 4-*trans*-4-alkylcyclohexyl)benzoates [17, 18].

The introduction of a fluorine atom in the external benzene ring (phenolic moiety) in the *ortho*-position (position 3) to the terminal substituent Y only slightly changes the clearing point and the S_A–N transition temperature in these compounds. The decrease in the clearing points is in the range 0–20°C (greater for $Y = F$ and smaller for $Y = NCS$), and the increase in the S_A–N transition temperatures in the range 5–10°C, except for the compounds with $Y = NO_2$. In this case the clearing point, as well as the S_A–N transition temperature decreases more than 30°C. The nitro group has a large van der Waals radius and the presence of the *ortho*-substituent introduces a steric hindrance which prevents coplanarity with the adjacent benzene ring. Twisting of the NO_2 group then increases the repulsive forces between the molecules.

The introduction of a fluorine atom into the central benzene ring (acidic moiety) in the *ortho*-position to the carboxylic bridge ($X_1 = F$) strongly decreases the melting points, clearing points and S_A–N transition temperatures of structure **I**. In structure **I** with $Y = NCS$ and F, the smectic A phase is eliminated. In the case of structure **I** with $Y = NO_2$, the results suggest that the lateral substitution by the fluorine in the central benzene ring causes a similar steric effect to that in the external benzene ring when $Y = NO_2$. This confirms that a fluorine atom in the neighbourhood of the COO group as well the NO_2 group involves a similar steric disturbance.

The simultaneous introduction of fluorine atoms in the lateral positions of both benzene rings causes further decreases in the clearing points for all the structures **I**. Moreover, a further significant decrease in the melting points is observed for the structures **I** with the NCS terminal group. An opposite influence of disubstitution (to that mentioned above) is observed for the phase transition temperature S_A–N and the ratio $T_{S_A\text{–}N}/T_{N\text{–}I}$. These values are greater than in the compounds with one fluorine atom in the central benzene ring. The stability of the smectic A phase, as well as a tendency to form this phase, changes in structure **I** in the following way: $X_1 = H$, $X_2 = F > X_2 = X_1 = H > X_2 = X_1 = F > X_1 = F$, $X_2 = H$. This relationship was found for the compounds with $Y = NCS$ and NO_2. It is also probably true for the compounds with $Y = OCF_3$ and CF_3, but structure **I** with these substituents have not been investigated with all four variants of these lateral positions of substitution. The observed influence of the fluorine atoms as lateral substituents in structure **I** is similar to that found in other classes of compounds, for example, in biphenylcarboxylates [19] and terphenyl derivatives [20].

In the case of structure **I** with $Y = NCS$, substitution by one fluorine atom in the acidic moiety or by two fluorine atoms (in the acidic and phenolic moieties) is most advantageous, because the decreases in the melting points and S_A–N transition temperatures are greater than the decreases in the clearing points, so that the range of the nematic phase is enhanced. In the case of $Y = F$, the introduction of two fluorine atoms in the lateral positions involves a greater decrease in the clearing points than in the melting points; these compounds are only monotropic nematics. The mesomorphic properties of structure **I** with $Y = NO_2$, NCS, F, OCF_3, or CF_3 are different from those of structure **I** with $Y = CN$, the latter having only a nematic phase for the members with $n = 3$–7 in the alkyl chains, see [1] and table 1.

3.2. *1,3,2-Dioxaborin-2-yl esters as dopants for nematic mixtures*

The potential of structure **I** for use in adjusting the properties of nematic mixtures was tested by preparing 10 wt % solutions in a eutectic mixture A containing three 4-(*trans*-4-alkylcyclohexyl)benzenoisothiocyanates —see table 2.

For these mixtures, the dielectric anisotropy ($\Delta\varepsilon$), birefringence (Δn), bulk viscosity η, threshold voltage V_{10} and saturation voltage V_{90} were measured at 20°C. The results are given in table 2.

Not all of the dioxaborinyl esters **I** were readily soluble in the base mixture A. Only the disubstituted structures **I**, the monosubstituted compounds with $Y = OCF_3$ and F, and unsubstituted compounds with $Y = OCF_3$ had a sufficient solubility (about 10 per cent by weight). The viscosity of mixture A after being doped with 10 wt % of structure **I** increases from 13 mPa s to 15–16 mPa s. Mixtures with short response times usually have viscosities lower than 20 mPa s, so the amount of dioxaborinanes in such a mixture should not be greater than 20 wt % to fulfill this condition.

The dielectric and optical properties and the threshold voltage of the doped mixtures change in the following way in terms of the terminal substitution of the dopants (A refers to the undoped mixture):

$\Delta\varepsilon$ $\quad CN > NCS > OCF_3 > F > A,$

$\Delta\varepsilon/\varepsilon_\perp$ $\quad NCS > CN > A > F > OCF_3,$

Δn $\quad NCS > A \geq CN > OCF_3 > F,$

V_{10} $\quad A > NCS \sim OCF_3 \sim F > CN.$

Structure **I** with an NCS, OCF_3, or F terminal group decrease the threshold voltage of mixture A to a similar degree, but less than structure **I** with $Y = CN$. In most cases, the steepness of the electro-optical curves is lowered (greater values of the ratio V_{50}/V_{10}-1). Therefore

Table 2. Physical and electro-optical properties of mixtures containing 90 wt % of mixture A: 4-(*trans*-4-propylcyclohexyl) benzenoisothiocyanate—42 wt %, 4-(*trans*-4-hexylcyclohexyl)benzenoisothiocyanate—40 wt % and 4-(*trans*-4-octylcyclohexyl) benzenoisothiocyanate—18 wt % and 10 wt % of a compound **I** (at 20°C).

n	X_1	X_2	Y	V_{10} /volt	V_{50} /volt	V_{90} /volt	T_{N-I} /°C	η /mPa s	Δn	$\Delta\varepsilon$	$\Delta\varepsilon/\varepsilon_\perp$	Sol.† /mol %	V_{90}/V_{10}-1
Mixture A													
				1·69	1·95	2·38	42·0	12·9	0·1680	8·1	1·98	—	0·411
Mixture A containing 10 wt % of compound I													
4	F	H	F	1·24	1·52	1·76	42·0	16·0	0·1614	8·6	1·91	22·0	0·423
4	H	F	F	1·39	1·56	1·82	41·5	16·0	0·1644	9·3	1·86	25·1	0·312
4	F	F	F	1·31	1·58	2·05	40·0	16·1	0·1603	7·6	1·94	10·2	0·561
4	H	H	OCF_3	1·33	1·60	2·10	45·5	15·3	0·1659	8·8	2·00	12·2	0·575
4	F	H	OCF_3	1·49	1·69	2·23	41·0	15·5	0·1622	8·9	1·89	11·0	0·496
4	F	H	NCS	1·48	1·71	2·15	48·8	15·6	0·1782	9·5	2·11	7·0	0·452
4	F	F	NCS	1·30	1·56	1·95	47·5	15·7	0·1756	8·6	2·10	6·8	0·496
5	F	F	NCS	1·36	1·61	1·98	49·0	15·2	0·1782	9·0	2·19	18·8	0·451
5	F	F	CN	1·15	1·43	1·70	47·0	16·1	0·1685	9·4	2·04	17·0	0·480

†Solubility at 0°C calculated from CSL's equation: $\ln x_k = -\frac{\Delta H_k^\circ}{R}\left(\frac{I}{T}-\frac{1}{T_k^\circ}\right)$, where x_k, ΔH_k°, T_k° are the mol ratio, melting enthalpy and melting temperature of the k-component.

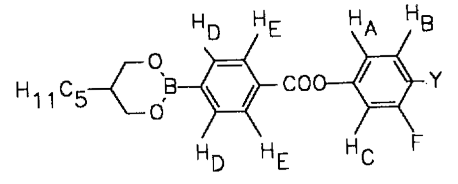
these compounds would be less useful as components of TN mixtures, where mixtures with a steep electro-optical curve are usually used, but may be used as components for STN mixtures. The results of investigations of structure **I** as dopants for STN mixtures have been described by us [21]. We found that mixtures containing structure **I** enabled us to obtain STN cells with 180° or even higher twist angles with a high multiplexibility and short response times.

4. Experimental

Typical procedures illustrative of the preparation of the esters and of structure **I** with Y=NCS are given below.

4.1. *3-Fluoro-4-nitrophenyl 4-(5-pentyl-1,3,2,-dioxaborin-2-yl)benzoate*

3·5 g (12·7 mmol) of 4-(5-pentyl-1,3,2-dioxaborin-2-yl)benzoic acid, 2 g (12·7 mmol) of 3-fluoro-4-nitrophenol, 0·78 g (5·1 mmol) of $POCl_3$ and 5 cm^3 benzene were heated under reflux during 5 h. Then this mixture was poured into water and 50 cm^3 of benzene were added. The layers were separated and the benzene layer was washed and then dried over $MgSO_4$. The benzene was evaporated and the solid was crystallized from methanol; the yield of the ester was 47 per cent (2·5 g) with phase transitions: C 98 S_A 125 N 147 I. IR (KBr) 1258 cm^{-1} (C-O-C); 1317 cm^{-1} (B-O); 1350 cm^{-1} (NO_2 sym); 1530 cm^{-1} (NO_2 asym); 1737 cm^{-1} (C=O); ^{1}H NMR δ=0·94 (3 H, t, J=6 Hz, CH_3), 1·34 (8 H, m, $(CH_2)_4$), 2·1 (1 H, m, O-CH_2-CH), 3·77 and 3·82 (2 H, dd, J=11 and 11 Hz O-CH_2-CH, ax), 4·17 and 4·23 (2 H, dd, J=4 and 4 Hz, O-CH_2-CH, eq), 7·25 and 8·2 (3 H, main components of a complex multiplet belonging to H_C, H_A and H_B—see formula **II**; the proton spectrum of this benzene ring is similar to that of 3-fluoro-4-nitrophenol [22]), 7·92 (2 H, d, J=8 Hz, H_D), 8·12 (2 H, d, J=8 Hz, H_E).



II

4.2. *3-Fluoro-4-isothiocyanatophenyl 4-(5-pentyl-1,2,3-dioxaborin-2-yl)benzoate*

2·5 g (6 mmol) of 3-fluoro-4-nitrophenyl 4-(5-pentyl-1,2,3-dioxaborin-2-yl)benzoate, 0·3 g of Pd/C (10 per cent) and 50 cm^3 of ethyl acetate were heated at 50°C in a hydrogen atmosphere. After consuming 430 cm^3 of H_2, the reaction stopped; the reaction vessel was flushed with nitrogen and then the catalyst was separated and the solvent removed by distillation. The solid amine was dissolved in 200 cm^3 of chloroform, and the solution was dripped into a mixture of 50 cm^3 of chloroform, 20 cm^3

of water, 0·76 g (6 mmol) of $CSCl_2$ and 1·2 g (12 mmol) of $CaCO_3$, keeping the temperature between 0–3°C. After completion of the addition, stirring was continued as the temperature rose to room temperature. The chloroform layer was separated and dried over $MgSO_4$. Then the chloroform was distilled off and the solid residue crystallized from a mixture of acetone:methanol (3:1). The product was next dissolved in hexane and the solution passed through a heated column filled with silica gel. The product obtained was repeatedly crystallized from hexane until constant transition temperatures were obtained. The yield of 3-fluoro-4-isothiocyanatophenyl 4-(5-phenyl-1,2,3-dioxaborin-2-yl)benzoate was 1 g (39 per cent) with phase transitions: C 115·8 S_A 151·1 N 178 I. Analysis for $C_{22}H_{23}NO_4SF$; Calculated: C, 61·84; H, 5·43; N, 3·28 per cent; Found: C, 61·84; H, 5·42; N, 3·31 per cent. IR (KBr), 1254 (C-O-C), 1318 (B-O), 1737 (C=O); 2108 cm^{-1} (N=C=S). PRM: ^1H NMR $\delta = 0·90$ (3 H, t, CH_3), 1·32 (8 H, m, $(CH_2)_4$), 2·11 (1 H, m, CH–CH_2—O), 3·76, 3·82 (2 H, dd, $J = 11$ and 4 Hz, CH-CH_2-O, ax), 4·17, 4·82 (2 H, dd, $J = 4$ and 4 Hz, CH–CH_2-O, eq), 7·90 (2 H, d, $J = 8$ Hz, H_D), 8·11 (2 H, d, $J = 8$ Hz, H_E), 7·15 (3 H, complex multiplet belongs to H_C, H_A and H_B).

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