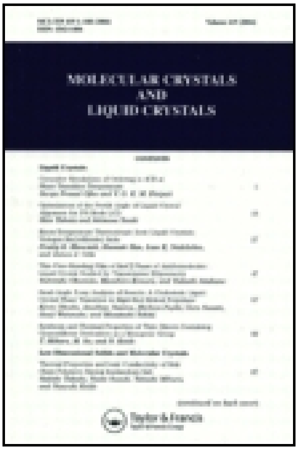

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Perfluoro(Alkoxy)Alkylation in One-Ring Liquid Crystalline Derivatives

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Perfluoro(Alkoxy)Alkylation in One-Ring Liquid Crystalline Derivatives

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The effect of introducing the perfluoro(alkoxy)alkyl groups into liquid crystalline one-ring derivatives on the appearance of the mesophases and their physico-chemical properties is discussed and compared with that of the corresponding (alkoxy)alkyl groups.

Keywords: perfluoro(alkoxy)alkyl groups; physico-chemical properties; liquid crystals

1. INTRODUCTION

Over the past two decades much interest has been shown in the design and synthesis of liquid crystalline perfluoro(alkoxy)alkyl substituted derivatives [1–29], especially one-ring systems. [1-9, 11–13, 17, 19-22,26,27] As a part of our systematic study of the structure-property relations existing in the liquid crystals (see, for example our previous publication [30]), we present here our results on the effect of introducing the perfluoro(alkoxy)alkyl groups into one-ring derivatives on the appearance of the mesophases and their physico-chemical properties comparing it with that of the corresponding (alkoxy)alkyl groups.

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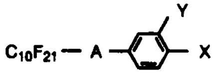
2. PHYSICO-CHEMICAL PROPERTIES

The phase transition temperatures of some one-ring perfluoro(alkoxy)alkyl substituted derivatives and the corresponding (alkoxy)alkyl-substituted reference compounds are summarized in tables I–VI, where Cr, SmA, SmB, SmC, N and I are the crystalline, smectic A, smectic B, smectic C, nematic and isotropic phases, respectively.

TABLE I Physico-chemical properties of liquid crystals:



No.	*A*	*X*	*Y*	*Phase transitions / °C*	d^a_{SmB}/Å	*d / L*	*Ref.*
1-1	C_2H_4OOC	CN	H	Cr 118 I			[1]
1-2	C_2H_4OOC	H	CN	Cr 77 SmB 90 I			[3]
1-3	C_2H_4OOC	NO_2	H	Cr 92 Sm (85) Sm (68) I			[2]
1-4	C_2H_4OOC	H	NO_2	Cr 62 SmB 92 I			[3]
1-5	C_2H_4OOC	F	H	Cr 74 I			[1]
1-6	C_2H_4OOC	H	F	Cr 63 SmB (60) I			[3]
1-7	C_2H_4OOC	CH_3	H	Cr 63 I			[3]
1-8	C_2H_4OOC	H	CH_3	Cr 60 SmB (54) I			[3]
1-9	C_2H_4OOC	OCH_3	H	Cr 66 I			[1]
1-10	C_2H_4OOC	H	OCH_3	Cr 63 I			[3]
1-11	C_2H_4OOC	$OC_{10}H_{21}$	H	Cr 77 I			[1]
1-12	C_2H_4OOC	$COOCH_3$	H	Cr 109 SmA (103) I			[5]
1-13	C_2H_4O	CN	H	Cr 83 SmB 104 SmA 112 I	44.1	1.92	[9]
1-14	C_2H_4O	H	CN	Cr 79 SmB 87 I	44.6	2.0	[9]
1-15	C_2H_4O	NO_2	H	Cr 82 SmB 86 SmA 109 I	42.9	1.92	[9]
1-16	C_2H_4O	H	NO_2	Cr 69 SmB 85 I	43.7	1.95	[9]

a. $T_{meas} = T_{SmB - SmA} - 10°C$.

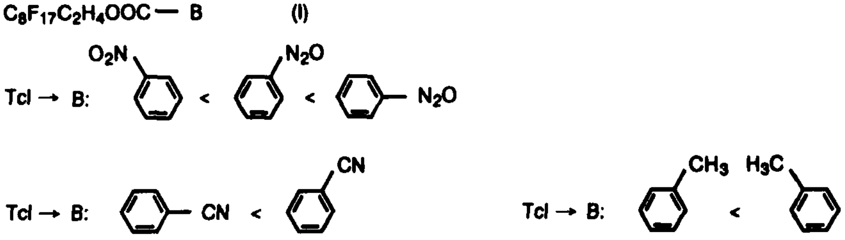
As can be seen from table I, the introduction of perfluorodecyl group into one-ring benzene derivatives has an effect on their mesomorphic properties which depends on the structure and positions of other substituents.

4-Subsituted [2-(n-perfluorodecyl)ethyl] benzoates are not mesomorphic (compounds **1–1, 1–5, 1–7**) nor do they exhibit lower smectic thermostability (compound **1–3**) in comparison with those of the corresponding 3-substituted benzoates (compounds **1–2, 1–4, 1–6, 1–8**). Neither corresponding 4- nor

3-methoxy substituted derivatives are mesomorphic (compounds **1–9** and **1–10**). Furthermore, 4-decyloxy-substituted benzoate **1–11** is also non-mesomorphic, revealing that increasing the alkoxy group length does not always lead to the enhancement of the mesomorphic stability (compounds **1–9** and **1–11**, see also reference 3), while the introduction of the COO group between the methyl group and the benzene ring creates the monotropic smectic A phase with high melting-point (compounds **1–7**and **1–12**).

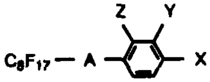
Similar results have been found for 2-(n-perfluorooctyl)ethyl 4-and 3-cyano substituted benzoates **2–7** and **2–8** (table II), respectively, with non-mesomorphic behavior again observed for the 4-substituted compound.

However, the replacement of the C_2H_4OOC group as a connector between the perfluorodecyl group and benzene ring in compounds **1–1, 1–2** and **1–3, 1–4** by the ethoxy group to obtain compounds **1–13, 1–14** and **1–15, 1–16**, respectively changes the dependence of their mesomorphic properties on the positions of the cyano and nitro groups. Higher smectic thermostabilities and a lower ratio of d / L (where d is the layer spacing in the bilayer smectic B phase and L is the molecular length) are recorded for 4-(2-perfluorodecylethoxy)-benzonitrile and -nitrobenzene compared to those of the corresponding 3-(2-perfluorodecylethoxy)-benzonitrile and 3-(2-perfluorodecylethoxy)-nitrobenzene (compounds **1–13, 1–14** and **1–15, 1–16**, table I). The thermal data collated in tables I–III reveal that decreasing the length of the perfluoroalkyl group attached via connector to the benzene ring results in decreasing the clearing and melting (excepting compound **2–2**) temperatures (compounds **1–1** and **2–7, 1–2**and **2–8, 1–3** and **2–1, 1–4** and **2–2, 1–12** and **3–1, 1–13** and **2–10, 1–14** and **2–11**), and results in changing the dependence of the mesomorphic properties of perfluoroalkylated one-ring derivatives on the position of their nitro substituents, with higher monotropic smectic thermostability observed for 4-nitro-substituted benzoate **2–1** in comparison with that of the corresponding 3-nitro-substituted benzoate **2–2** (see the opposite behavior observed for compounds **1–3** and **1–4**, table I). The corresponding 2-nitro-substituted derivative **2–3** exhibits a bilayered smectic B phase and a partially bilayered smectic A phase with the lowest smectic thermostability among compounds **2–1** – **2–3**. These results, the phase transition temperatures of compounds **2–12** and **2–13** having 3-methyl and 2-methyl substituents, respectively, as well as previously discussed mesomorphic behaviour of the cyano substituted derivatives **2–7** and **2–8** can be expressed by the following orders of increasing the clearing temperatures Tcl (smectic-isotropic phase transition temperatures) in dependence on the positions of the nitro, cyano and methyl groups introduced into the benzene ring (table II): .



SYSTEM 1

TABLE II Physico-chemical properties of liquid crystals:



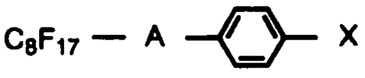
No.	A	X	Y	Z	Phase transitions / °C	d^a_{SmA} / Å	d / L	Ref.
2-1	C_2H_4OOC	NO_2	H	H	Cr 80 Sm (60) I			[2]
2-2	C_2H_4OOC	H	NO_2	H	Cr 65 SmB (44) SmA (53) I			[3]
2-3	C_2H_4OOC	H	H	NO_2	Cr 26 SmB 26 SmA 39 I	34.5[b]	1.82	[3]
						40.5[c]	2.13	
2-4	C_4H_8O	NO_2	H	H	Cr 73 SmA (69) I			[6]
2-5	C_2H_4O	NO_2	H	H	Cr 49 SmA 78 I	34.0	1.68	[9]
2-6	C_2H_4O	H	NO_2	H	Cr 55 SmA (41) I	23.5[d]	1.18	[9]
2-7	C_2H_4OOC	CN	H	H	Cr 93 I			[3]
2-8	C_2H_4OOC	H	CN	H	Cr 70 SmB (41) SmA (48) I			[3]
2-9	C_4H_8O	CN	H	H	Cr 72 SmA 73 I			[6]
2-10	C_2H_4O	CN	H	H	Cr 56 SmB (42) SmA 81 I	35.6	1.75	[9]
2-11	C_2H_4O	H	CN	H	Cr 65 SmA (42) I			[9]
2-12	C_2H_4OOC	H	CH_3	H	Cr 35 SmB (-7) I			[3]
2-13	C_2H_4OOC	H	H	CH_3	Cr 29 SmB (17) I			[3]
2-14	C_4H_8O	$COOCH_3$	H	H	Cr 69 SmA 81 I	33.1[e]	1.35	[6]

a. $T_{meas} = T_{SmA\text{-}I} - 10$ °C
b. T_{meas} = 39°C.
c. T_{meas} = 28°C.
d. T_{meas} = 20°C.
e. T_{meas} = 75°C.

These orderings reveal that the mesomorphic behavior of 2-(n-perfluorooctyl)ethyl-substituted benzoates strongly depend on the structure and positions of their substituents.

Interestingly, for 2-perfluoroalkylethoxy-substituted benzenes, in contrast to the corresponding 2-(n-perfluoroalkyl)ethyl-substituted benzoates, decreasing the length of the perfluoroalkyl group does not change the dependence of their mesomorphic properties on the positions of the nitro and cyano groups, with the higher clearing points observed for 4-(2-perfluoroalkylethoxy)-substituted benzenes compared to those of the corresponding 3-substituted derivatives (compounds **1–13, 1–14** and **2–10, 2–11; 1–15, 1–16** and **2–5, 2–6**; tables I, II). The only changes observed are more pronounced partially bilayered smectic A behavior with decreased clearing points recorded for perfluorooctyl derivatives in comparison with those of the corresponding perfluorodecyl derivatives (compounds **1–15** and **2–5, 1–16** and **2–6**, tables I, II) and the higher value of ratio d / L (where d is the layer spacing in the smectic A phase) observed for 4-nitro-substituted derivative **2–5** in comparison with that of the corresponding 3-nitro-substituted derivative **2–6**.

As can be seen from tables I–III, the mesomorphic properties of 4-perfluoroalkyl-substituted benzenes depend on the structure of the connectors, with higher smectic thermostabilities recorded for the C_2H_4O group in comparison with that of the corresponding C_4H_8O and C_2H_4OOC groups (compounds **1–1** and **1–13; 1–3** and **1–15; 2–1, 2–4** and **2–5; 2–7, 2–9** and **2–10; 2–14** and **3–1**):



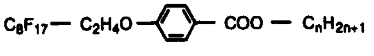
(II)

$X = NO_2, CN;\ Tcl \rightarrow A: C_2H_4OOC < C_4H_8O < C_2H_4O$

$X = COOCH_3;\ Tcl \rightarrow A: C_4H_8O < C_2H_4O$

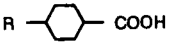
SYSTEM 2

TABLE III Physico-chemical properties of liquid crystals:



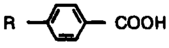
No.	*n*	*Phase transitions / °C*	*d/Å*	*d/L*	T_{meas}/°C	*Ref.*
3–1	1	Cr 86 SmA (84) I	30.0	1.36	78	[5]
3–2	2	Cr 80 SmA (70) I	28.7	1.22	67	[5]
3–3	3	Cr 83 SmA (66) I	28.5	1.12	63	[5]
3–4	4	Cr 70 SmA (52) I	27.9	1.08	35	[5]
3–5	5	Cr 66 SmA (45) I				[5]
3–6	6	Cr 65 I				[5]
3–7	7	Cr 77 I				[5]
3–8	8	Cr 69 I				[5]
3–9	9	Cr 71 I				[5]
3–10	10	Cr 73 I				[5]
3–11	11	Cr 72 SmA (56) I				[5]
3–12	12	Cr 61 SmA (58) I	60.5	1.72	56	[5]

For the corresponding 3-perfluoroalkyl-substituted benzenes, the opposite situation has been found, again revealing the influence on their mesomorphic properties of the positions of the substituents (compounds **1–2** and **1–14**; **1–4** and **1–16**; **2–2** and **2–6**; **2–8** and **2–11**, tables I, II). There have been reported similar results showing the influence of the structure of the connector on the mesomorphic properties of one-ring benzene derivatives [6], and two-ring biphenyl derivatives.[23-25,28,29] The thermal data presented in table III show the mesomorphic behavior of a homologous series of alkyl 4-(2-perfluorooctyl)ethoxybenzoates exhibiting decreasing dependence of their monotropic smectic A thermostabilities with increasing the alkyl chain length C_nH_{2n+1} for methyl – pentyl derivatives (compounds **3–1** – **3–5**), disappearance of the mesophase at n = 6 – 10 (compounds **3–6** - **3–10**), and increasing dependence of their monotropic smectic A thermostabilities from n = 11 (compounds **3–11, 3–12**). This behavior corresponds to the decreasing dependence of the ratio d / L (d is the layer spacing in the partially bilayered smectic A phase) with increasing the alkyl chain length for methyl – butyl derivatives **3–1** – **3–4**, and sufficiently increased the value of the ratio d / L observed for compound **3–12** (n = 12). Similar trends have been observed in the corresponding perfluorodecyl derivatives[5] and can be explained in terms of the influence of fluorophilic interactions around the perfluoroalkyl group on the stability of the smectic phase which is dependent on the relative constitution of the perfluoroalkyl group to the alkyl one within these molecules.[4,5] From comparison of the values of d and ratio d / L for compounds **2–14** and **3–1** (tables II, III), it follows that decreasing the length of the connector from the butoxy group to the ethoxy group results in decreasing the layer spacing (as expected) and molecular overlap in formation of the bilayered smectic A phase. The formation of the hydrogen-bonded cyclic dimers, their dissociation and the appearance of "open associates" and monomers [8,19–22] may influence the mesophase stability in some trans-4-perfluoroalkylcyclohexanecarboxylic acids presented in table IV. So far non-mesomorphic perfluoropropyl-substituted derivative **4–1** exhibits a higher melting temperature compared to that of the corresponding non-mesomorphic propyl-substituted derivative **4–2**. But perfluorobutyl-, perfluoropentyl- and perfluoroheptyl-substituted derivatives show higher melting and clearing points of their nematic and smectic phases, respectively, in comparison with those of the corresponding butyl-, pentyl- and heptyl-substituted derivatives exhibiting the nematic, and nematic and smectic C phases, respectively (compounds **4–3** and **4–5**, **4–6** and **4–7**, **4–8** and **4–9**; table IV).

TABLE IV Mesomorphic properties of liquid crystals: 

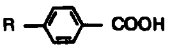
No.	*R*	*Phase transitions / °C*	*Reference*
4–1	C_3F_7	Cr 131 I	[8, 22]
4–2	C_3H_7	Cr 99 I	[36]
4–3	C_4F_9	Cr 117 N 131 I	[8]
4–4	$CF_3C_3H_6$	Cr_1 47 Cr_2 51 N 58 I	[22]
4–5	C_4H_9	Cr 41 N 94 I	[36]
4–6	C_5F_{11}	Cr 95 Sm 147 I	[8, 22]
4–7	C_5H_{11}	Cr 54 N 105 I	[36]
4–8	C_7F_{15}	Cr 105 Sm 177 I	[8, 22]
4–9	C_7H_{15}	Cr 39 SmC 75 N 101 I	[36]

Interestingly, decreasing the fluorination content of the perfluorobutyl group sufficiently lowers the melting point and nematic thermostability (compounds **4–3** and **4–4**, table IV), leading even to a lower clearing temperature compared to that of the corresponding butyl substituted derivative **4–5**. As can be seen from table V, the complete fluorination of the alkyl groups of 4-alkylbenzoic acids sufficiently increases their melting temperatures and results in the disappearance of the mesophases in comparison with those of the corresponding 4-alkylbenzoic acids (compounds **5–4** and **5–6**, **5–7** and **5–8**, **5–9** and **5–10**, **5–11** and **5–12**, **5–13** and **5–14**; table V). The introduction of these perfluoroalkyl groups into benzoic acid does not change its non-mesomorphic behavior, leading to increasing (compounds **5–1** and **5–2, 5–9, 5–11, 5–13**) and decreasing (compounds **5–1** and **5–3, 5–4, 5–7**) the melting points. In contrast to previously discussed mesomorphic properties of compounds **4–3** and **4–4** (table IV), decreasing the fluorination content of the perfluorobutyl group of non-mesomorphic 4-perfluorobutylbenzoic acid (compound **5–4**) results in the appearance of the monotropic nematic phase (compound **5–5**, table V), while the melting temperatures of these compounds show the similar behavior compared to that of the corresponding compounds **4–3** and **4–4**, with the lower values recorded for the partially fluorinated derivatives **5–5** and **4–4**, respectively. Non-mesomorphic behavior of 4-perfluoroalkylbenzoic acids (compounds **5–2** – **5–4, 5–7, 5–9, 5–11, 5–13**) was correlated with the absence of open associates and monomers in their crystals at temperatures close to the corresponding melting points.[19–22] Open associates and monomers were found in these compounds only in the isotropic state, in contrast to their appearance observed before the corresponding meltings into the mesophases in compound **5–5** and 4-(alkoxy)alkylbenzoic acids, [19,20,22,31,32] and in the nematic phase of the trans-4-alkylcyclohexanecarboxylic acids. [33] These results and the phase transition temperatures shown in tables IV and V reveal that the effect on their mesomorphic properties of introducing the perfluoroalkyl groups into one-ring acids depends on their structures.

TABLE V Mesomorphic properties of liquid crystals: 

No.	*R*	*Phase transitions / °C*	*Reference*
5–1	H	Cr 187 I	[22]
5–2	CF_3	Cr 223 I	[22]
5–3	C_3F_7	Cr 166 I	[19, 20, 22]
5–4	C_4F_9	Cr 166 I	[19, 20, 22]
5–5	$CF_3C_3H_6$	Cr_1 110 Cr_2 116 N (104) I	[22]
5–6	C_4H_9	Cr 99.5 N 113 I	[37]
5–7	C_5F_{11}	Cr 186 I	[19, 20, 22]
5–8	C_5H_{11}	Cr 88 N 126.5 I	[37]
5–9	C_6F_{13}	Cr 192 I	[19, 20, 22]
5–10	C_6H_{13}	Cr 97.5 N 114.5 I	[37]
5–11	C_7F_{15}	Cr 200 I	[19, 20, 22]
5–12	C_7H_{15}	Cr 101.5 N 120 I	[37]
5–13	C_9F_{19}	Cr 207 I	[19, 20, 22]
5–14	C_9H_{19}	Cr 98.5 N 115 I	[37]

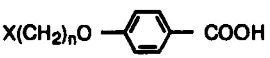
The thermal data collated in table VI show that the effect of perfluoroalkoxylation of some benzoic acids on their mesomorphic properties depends on the structures of the perfluoroalkoxy groups and their fluorination content. As it was found for 4-perfluoroalkylbenzoic acids presented in table V, the lower perfluoroalkoxy substituted derivatives, even completely fluorinated, exhibit non-mesomorphic behavior with the higher melting temperatures in comparison with those of the corresponding 4-alkoxybenzoic acids (compounds **6–1, 6–2** and **6–3**; **6–4, 6–5** and **6–6**; **6–7, 6–8** and **6–9**; table VI). As it was observed for the acids presented in tables IV and V, increasing the fluorination content of the perfluoroalkoxy groups further increases the melting temperatures of the lower acids shown in table VI (compounds **6–1** and **6–2, 6–4** and **6–5, 6–7** and **6–8**).

TABLE VI Mesomorphic properties of liquid crystals:  *

No.	*R*	*Phase transitions / °C*	*Reference*
6–1	$C_3H_7CF_2O$	Cr 155–156 I	[12]
6–2	$C_3F_7CH_2O$	Cr 182 I	[13]
6–3	C_4H_9O	Cr 147 N 160 I	[37]
6–4	$HC_5F_{10}O$	Cr 147–148 I	[12]
6–5	$C_5F_{11}O$	Cr 165–166 I	[12]
6–6	$C_5H_{11}O$	Cr 124 N 151 I	[37]
6–7	$HC_7F_{14}O$	Cr 162–164 I	[12]
6–8	$C_7F_{15}O$	Cr 185–186 I	[12]
6–9	$C_7H_{15}O$	Cr 92 SmC 98 N 146 I	[37]
6–10	$FC_{10}H_{20}O$	Cr 102 SmA 125 N 134 I	[7]
6–11	$C_6F_{13}C_4H_8O$	Cr 162.5 SmC 183.8 I^a	[7]
6–12	$C_{10}H_{21}O$	Cr 97 SmC 122 N 142 I	[7]
6–13	$FC_{11}H_{22}O$	Cr 82 SmA 125 N 128 I	[7]
6–14	$C_7F_{15}C_4H_8O$	Cr 166.3 SmC 187.5 I^a	[7]
6–15	$C_{11}H_{23}O$	Cr 84 SmC 128 N 139 I	[7]
6–16	$FC_{12}H_{24}O$	Cr 86 SmC 102 SmA 128 N 129 I	[7]
6–17	$C_8F_{17}C_4H_8O$	Cr 181.3 SmC 195 I^a	[7]
		Cr$_1$ 165 Cr$_2$ 178 SmC 190 SmA 193 I	[6]
6–18	$C_{12}H_{25}O$	Cr 95 SmC 129 N 137 I	[7]
6–19	$C_8F_{17}C_2H_4O$	X 209 I	[13]

* Estimated from the figure [7]. x is the unknown mesophase [13].

The replacement of one hydrogen atom by the fluorine atom in the alkoxy groups of higher acids **6–12, 6–15, 6–18** to obtain compounds **6–10, 6–13, 6–16,** repectively, results in decreasing their nematic thermostabilities, introduction of the smectic A phase, disappearance of the smectic C phase in compounds **6–10** and **6–13**, and decreasing the melting points in derivatives **6–13** and **6–16** in comparison with those of the corresponding 4-alkoxybenzoic acids. Increasing the quantity of the hydrogen atoms replaced by the fluorine ones in the alkoxy groups leads to the disappearance of the nematic phases and creates mesophases with pronounced smectic C behavior exhibiting higher clearing and melting temperatures observed for 4-perfluoroalkoxybenzoic acids compared to those of the corresponding 4-alkoxybenzoic acids (compounds **6–11** and **6–12, 6–14** and **6–15**, **6–17**and **6–18**, table VI). These findings are consistent with the following orders of thermal efficiency:

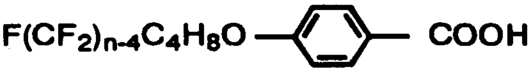


(III)

X = F $T_{cl} \rightarrow n: n = 11 < n = 12 < n = 10$ $\Delta T \rightarrow n: n = 12 < n = 11 < n = 10$

X = H $T_{cl} \rightarrow n: n = 12 < n = 11 < n = 10$ $\Delta T \rightarrow n: n = 11 < n = 12 < n = 10$

SYSTEM 3



(IV)

$T_{cl} \rightarrow n: n = 10 < n = 11 < n = 12$

SYSTEM 4

These results reveal that the replacement of the hydrogen atoms by the fluorine atoms in the terminal alkoxy groups of 4-alkoxybenzoic acids affects the terminal group's efficiency in different degrees depending on the quantity and positions of introduced fluorine atoms. This effect is also observed from comparison of the phase transition temperatures of compounds **6–11** and **6–19**, and as in the case of 4-substituted derivatives considered above, we can see the increased thermal efficiency of the ethoxy connector in comparison with that of the corresponding butoxy group (compounds **6–17** and **6–19**).

Similar effects on their physico-chemical properties of introducing the perfluoro(alkoxy)alkyl groups into other liquid crystalline one-ring derivatives have been reported in the following references: 3–9, 26, 27.

Keeping in mind that the perfluoroalkyl group, which is more rigid and linear than the corresponding alkyl one,[1,10,11,17,18] promotes microphase segregation,[6,18,24,34,35] and strong electrostatic interactions, such as fluorophobic [1,3–6,11,17,18,24,34,35] and fluorophilic [5] interactions around the perfluoroalkyl groups, can be responsible for the pronounced layer arrangements of molecules and phase formation. We can point out that, according to the results of this study, the effect on their physico-chemical properties of introducing the perfluoro(alkoxy)alkyl groups into one-ring derivatives strongly depends on their structures.

3. CONCLUSION

Systematic studies on the effect of introducing the perfluoro(alkoxy)alkyl groups into one-ring derivatives on the creation of the mesophases and their physico-chemical properties have been performed, with attempts to correlate the molecular-level parameters with the observed properties. The information here presented may lead to a better understanding of the nature of liquid crystals.

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