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Liquid Crystal Pyridine Derivatives of High Positive Dielectric Anisotropy

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Liquid Crystal Pyridine Derivatives of High Positive Dielectric Anisotropy

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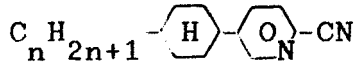
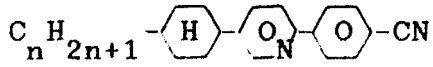
Pyridine derivatives of general formula I–YII have been synthesized. Phase transition temperatures and physico-chemical properties of the compounds are given.



I



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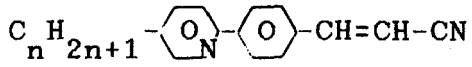
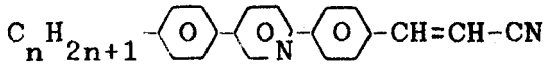
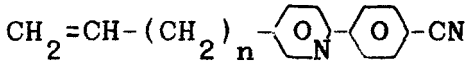


III

IY



Y



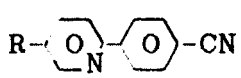
YI

YII

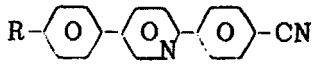
Keywords: pyridine derivatives, dielectric anisotropy, chemical synthesis

1. INTRODUCTION

5-Alkyl-2-(4-cyanophenyl)pyridines (YIII) and 5-(4-alkylphenyl)-2-(4-cyanophenyl)pyridines (IX) synthesized by us earlier are extremely useful components of liquid crystalline materials for displays based on the twist effect in view of their wide nematic phase intervals and high values of Δn and $\Delta\varepsilon$.[1-3]



YIII



IX

The way to further improve each of the above systems should involve development of compounds with high $\Delta\varepsilon$ and low viscosity to use them as basic components for LC materials or as dopants increasing positive dielectric anisotropy of the materials which possess other suitable parameters such as low viscosity, wide nematic phase range and so on. The major way to reduce viscosity and broaden the nematic range for systems YIII and IX while retaining positive dielectric anisotropy is introduction of a cyclohexane ring or —NCS-group (compounds I, II and YII), and increasing geometric anisotropy of molecules by inserting acrylonitrile group (compounds III–Y).

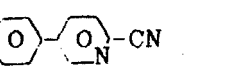
This paper reports phase transition temperatures for new LC pyridine derivatives and their physico-chemical properties. The synthetic procedures for these compounds are described in detail elsewhere.[4]

2. RESULTS AND DISCUSSION

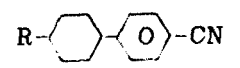
2.1. Phase Transition Points

Phase transition temperatures were measured in a polarizing microscope fitted with Mettler FP52. For some compounds, clearing points are obtained by extrapolation from the clearing points of mixtures with ZLI-1132.

Table I gives phase transition points for 2-cyano-5-(trans-4-alkylcyclohexyl)pyridines (I) and—for comparison—5-(4-alkylphenyl)-2-cyanopyridines (X)[5] and trans-4-alkyl-(4′-cyanophenyl)cyclohexanes (XI).[6]

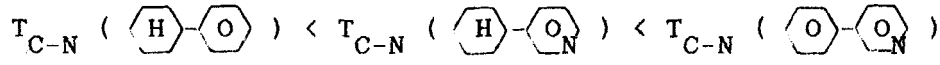


(X),



(XI)

From Table I it can be seen that for fifth, sixth and seventh homologs, dependence of phase transition points on the molecular structure of compounds I, X and XI can be represented as follows:



and

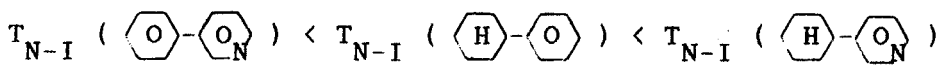
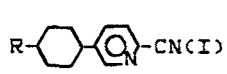
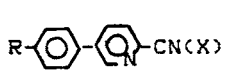
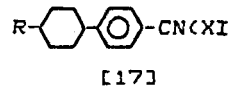


Table II gives phase transition points for 2-[4-(2-E-cyano-vinyl)phenyl]-5-alkylpyridines (III), 2-(4-isothiocyanophenyl)-5-alkylpyridines (YII) and for comparison 5-alkyl-2-(4-cyanophenyl)pyridines (YIII), 4,4′-alkylisothiocyanobiphenyls (XII).[7] Introduction of acrylonitrile group in a terminal position leads to growth in geometric molecular anisotropy and—as a result—to a significant increase in phase transition temperatures, especially clearing points for compounds III as compared to compounds YIII. Replacement of terminal CN-group with NCS results in a transition from a nematic to smectic phase and in a considerable growth of clearing points (YII and YIII, Table II). A similar effect was earlier observed for derivatives of biphenyl[7] and phenylpyrimidine[8] containing NCS-group.

TABLE I

Phase transition points (°C)

					 [17]	
R	T_{c-n}, °C	T_{n-i}, °C	T_{c-n}, °C	T_{n-i}, °C	T_{c-n}, °C	T_{n-i}, °C
C_3H_7	68,0	(25)	—	—	43,0	45,0
C_4H_9	60,7	(37)	64,0	—	41,0	41,0
C_5H_{11}	45,4	55,4	73,0	31*	30,0	55,0
C_6H_{13}	35,3	49,4	58,0	(44)	41,0	49,0
C_7H_{15}	50,1	60,2	66,0	(56)	30,0	57,0
C_8H_{17}	49,0	57,9	61,0	(58)		

( ), Monotropic transition.
* Extrapolated from mixture with ZLI-1132.

TABLE II

Phase transitions points (°C)

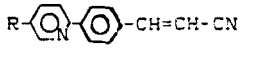
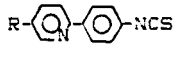
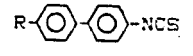
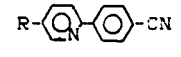
	 (III)			 (VII)		 (XII)[8]		 (VIII)	
R	T_{c-s}, °C	T_{s-n}, °C	T_{n-i}, °C	T_{c-s}, °C	T_{s-i}, °C	T_{c-s}, °C	T_{s-i}, °C	T_{c-n}, °C	T_{n-i}, °C
C_3H_7	84,2	110,0	162,0	64,0	99,0			43,4	43,8
C_4H_9	73,5	—	150,2	37,6	100,0			32,3	(26,5)
C_5H_{11}	65,7	—	155,1	34,0	98,5	53,0	74,5	33,6	43,5
C_6H_{13}	32,1	123,1	146,2	27,0	99,0			29,0	32,5
C_7H_{15}	48,0	133,0	143,0	26,1	99,2	56,0	73,0	30,9	47,0

Table III shows phase transition points for 2-(4-cyanophenyl)-5-alkenylpyridines (YI) and 2-(4-bromophenyl)-5-alkenylpyridines (XIII).

From Tables II and III it is seen that alkenyl cyano derivatives of pyridine have higher melting and clearing points than those for alkyl derivatives, besides of 5 homolog.

Table IV shows phase transition points for 2-(4-cyanophenyl)-5-(4-trans-alkyl-cyclohexyl)pyridines (II), 2-[4-(2-E-cyanovinyl)-phenyl]-5-(4-alkylphenyl)pyridines (Y) and for comparison 5-(4-alkylphenyl)-2-(4-cyanophenyl)pyridines (IX). Replacement of the phenyl ring with cyclohexane (II and IX), Table IV leads to a significant growth of melting points, slight decrease in clearing temperatures and disappearance of the smectic mesophase. Introduction of acrylonitrile group in the terminal position—in the same way as in the case with two-ring compounds (III), Table II—results in growth of melting and clearing temperatures (II and IY, IX and Y, Table IV).

2.2. Dielectric, Optic, Viscoelastic and Electro-Optic Properties

The procedures for measuring dielectric, optic, viscoelastic and electro-optic characteristics of liquid crystal compounds and their mixtures are described in detail in Reference 1. Parameters for some compounds were calculated from the corresponding parameters of mixtures with ZLI-1132.

In Table V dielectric properties of 2-cyano-5-(trans-4-alkyl-cyclohexyl)pyridines (I) are given in comparison with other known pyridine derivativers containing cyano group. From the table it can be seen that introduction of a pyridine ring in the LC compound structure results in a large diversity of dielectric properties, because the position of the nitrogen atom and the related dipole moment of the pyridine ring considerably affects the magnitude and direction of the total dipole moment: when the longitudinal component of the dipole moment for the pyridine ring coincides with that for the CN-group, $\Delta\varepsilon$ grows (I and YIII, Table V), in the other case $\Delta\varepsilon$ decreases (XIII and XIY, Table V). Introduction of a trans-cyclohexane fragment in the structure of two-ring pyridine cyano derivatives—as in the case with cyano derivatives of biphenyls and phenylcyclohexanes[6,9] leads to reduction in the dielectric anisotropy value.

In Table VI dielectric characteristics of 5-alkyl-2-(2-E-cyanovinylphenyl)-pyridines (III) are given.

For the homologous series presented in Tables V and VI, as for other homologous

TABLE III

Phase transition points (°C)

	R-(pyridine)-Br (XIII)		R-(pyridine)-CN (VI)	
R	T_{c-s} °C	T_{s-i} °C	T_{c-n} °C	T_{n-i} °C
CH_2=CH—CH_2	53,0	64,0	55,3	—
CH_2=CH—$(CH_2)_2$	70,4	—	50,0	(49,0)
CH_2=CH—$(CH_2)_3$	71,0	(55,0)	<0	

TABLE IV

Phase transition points (°C)

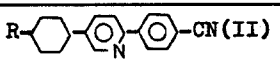
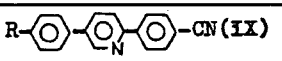
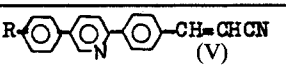
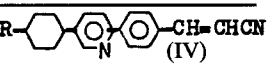
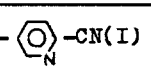
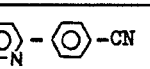
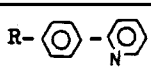
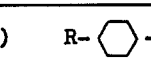
	 (II)		 (IX)			 (V)			 (IV)	
R	T_{c-n} °C	T_{n-i} °C	T_{c-n} ($c-s$) °C	T_{s-n} °C	T_{n-i} °C	T_{c-n} ($c-s$) °C	T_{s-n} °C	T_{n-i} °C	T_{c-n} °C	T_{n-i} °C
C_3H_7	123,0	237,0	—	—	—	—	—	—	133,0	318,4
C_4H_9	124,0	228,0	95,0		233,0	—	—	—	—	—
C_5H_{11}	113,0	228,0	76,0	—	232,1	136,1	168,1	310,0	—	—
C_6H_{13}	114,0	216,0	72,0	166,0	221,0	—	—	—		

TABLE V

Dielectric properties of some compounds

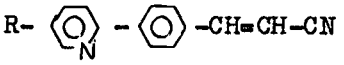
	R-  -CN(I)		R-  -CN (VIII)		R-  -CN(XIII)		R-  -CN(XIV)	
R	$\varepsilon_\perp$	$\Delta\varepsilon$	$\varepsilon_\perp$	$\Delta\varepsilon$	$\varepsilon_\perp$	$\Delta\varepsilon$	$\varepsilon_\perp$	$\Delta\varepsilon$
C_3H_7	—	—	9,7*	25,3*	8,9	9,4	7,4	4,3
C_4H_9	—	—	13,1*	21,4*	—	—	—	—
C_5H_{11}	10,8	12,1	10,9	17,8	8,0	6,5	6,5	3,7
C_6H_{13}	11,0	11,5	12,0	15,2	7,8	4,8	6,9	2,9
C_7H_{15}	9,5	10,3	9,2	15,8	7,7	3,1	6,8	2,3
C_8H_{17}	8,7	9,6	—	—	—	—	—	—

$\tau = T_{meas}/T_{n-i}$, *K*.

* Extr. from mixtures with 4,4′-pentylcyanobiphenyl.

TABLE VI

Dielectric properties of 5-alkyl-2-(2-E-cyano-vinylphenyl)pyridines

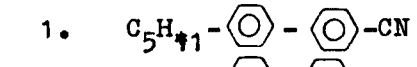
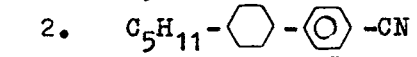
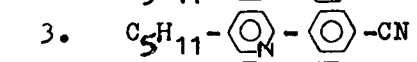
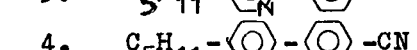
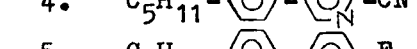
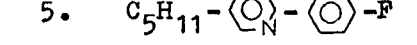
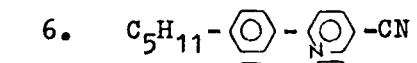
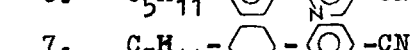
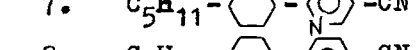
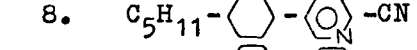


R	$\varepsilon_{\perp}$ $T = T_{n-i}$ − 75°C	$\Delta\varepsilon$ $T = T_{n-i}$ − 75°C
C_3H_7	8,4*	16,3*
C_4H_9	7,3	16,8
C_5H_{11}	7,6	14,7

* $T = T_{n-i}$ − 45°C.

TABLE VII

Permittivity results for some polar compounds

N	Compounds	ε_{iso}, $T = T_{cl}$
1.		11,2
2.		9,4
3.		17,4
4.		18,6
5.		8,5
6.		10,3
7.		8,6
8.		15,5
9.		6,3
10.		9,2

series of known chemical structures, the normal alteration of dielectric properties is observed.

Table VII gives dielectric constant values measured for the isotropic phase at $T = T_{cl}$ for the series of the fifth homologs of polar derivatives belonging to various chemical structures.

$$\varepsilon_{isotr.} \approx \varepsilon_{avr.} = \frac{2\varepsilon_{\perp} + \varepsilon_{\parallel}}{3}.$$

TABLE VIII

Dielectric constants of some compounds

N	Formula	$\varepsilon_\parallel$	$\varepsilon_\perp$	$\Delta\varepsilon$	τ
1.	C_5H_{11}-Ph-Ph-Ph-CN	16,0	3,0	13,5	0,75
2.	C_5H_{11}-Ph-Py-Ph-CN	22,8	6,1	16,7	0,70
3.	C_5H_{11}-Cy-Py-Ph-CN	17,2	3,2	14,0	0,75
4.	C_4H_9-Ph-Pyr-Ph-CN	27,0	4,0	23,0	0,70

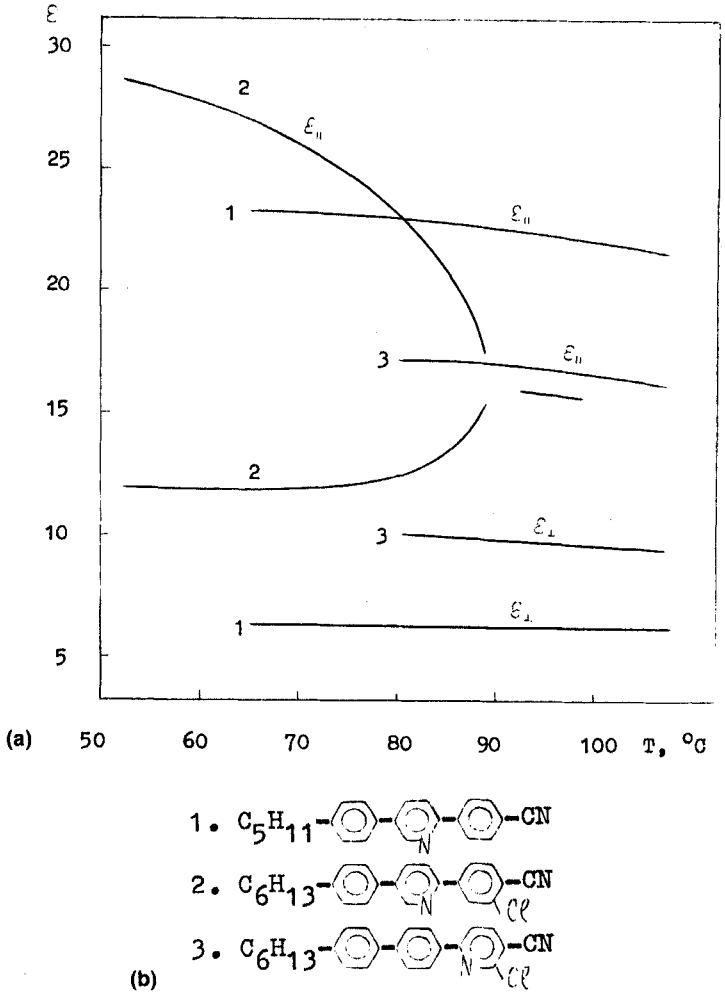
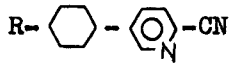


FIGURE 1 Dielectric properties of pyridine derivatives.

TABLE IX

Optic properties of 2-cyano-5-(trans-4-alkylcyclohexyl)pyridines



R	$n_{\parallel}$	$n_{\perp}$	Δn, λ = 589 nm	τ
C_5H_{11}	1,5700	1,4860	0,0840	0,99
C_6H_{13}	1,5680	1,4843	0,0837	0,98
C_7H_{15}	1,5650	1,4820	0,0830	0,98
C_8H_{17}	1,5630	1,4810	0,0820	0,98

Analysis of the data given in Table VII allows to define the effect of the rigid molecular core type, in particular terminal substituents, on the dielectric properties of the compounds studied. Measurements of dielectric properties in the isotropic phase also allow to investigate dielectric characteristics of nonmesomorphic compounds (4 and 5 in Table VII), as well as of those compounds which have only smectic phase (9 and 10, Table VII). As follows from the table, replacement of the phenyl ring with pyridine leads to growth in $\varepsilon_{isotr.}$ when the directions of dielectric moments for the pyridine ring and terminal polar group coincide (compounds 1, 3, 4; 2 and 8; 9 and 10) or to drop in $\varepsilon_{isotr.}$ in the other case (compounds 1 and 6; 2 and 7). Replacement of the terminal CN-group with isothiocyano and F results in decreasing $\varepsilon_{isotr.}$ in the same order in which the values of the dipole moments for CN (4.05 D), NCS (3.59 D) and F (1.47 D) decrease.[10]

Dielectric properties of three-ring cyano derivatives of various chemical structures are presented in Table VIII and Figure 1. As for the two-ring compounds (see Tables V and VI), introduction of a pyridine ring or lateral substituent (CI) in the molecular structure of a three-ring compound leads to a considerable change in the value of $\Delta\varepsilon$ defined by mutual direction of dipole moments of the CN-group, pyridine ring and the CI-atom-substituted phenyl ring.

Optical properties of 2-cyano-5-(trans-4-alkylcyclohexyl)-pyridines are presented in Table IX.

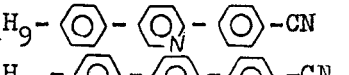
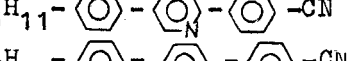
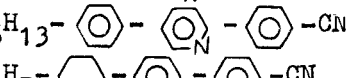
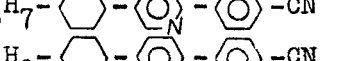
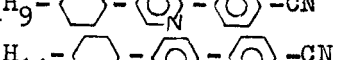
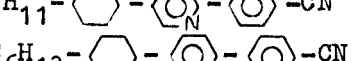
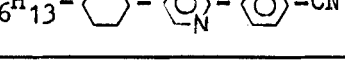
Here again decrease in optical anisotropy with increasing alkyl chain length is observed. Introduction of rings with saturated bonds in the molecular structure of pyridine cyano derivatives results in reduction of refractive indices (compare with Reference 6).

Table X gives values of rotational viscosity γ_1 for the series of 5-(4-alkylphenyl)-2-(4-cyanophenyl)pyridines (X), 5-(trans-4-alkylcyclohexyl)-2-(4-cyanophenyl)pyridines (II).

As can be seen from Table X, γ_1 exhibits even-odd alteration and grows for even and odd homologs with alkyl chain length. Introduction of a cyclohexane ring instead of the phenyl one results in diminishing rotational viscosity. Similar effects were observed for other known cyano derivatives as well.[11,12]

TABLE X

Values for some pyridine derivatives

N	Formula	*γ_1 Poise, 25°C
1.	C_4H_9- 	41
2.	C_5H_{11}- 	11,8
3.	C_6H_{13}- 	58,8
4.	C_3H_7- 	4,8
5.	C_4H_9- 	17,3
6.	C_5H_{11}- 	6,2
7.	C_6H_{13}- 	30,6

* Extrapolated from mixture with ZLI-1132.

2.3. Comparative Characteristics of LCM

Selection of the best components for liquid crystal materials and prediction of new chemical structures need comprehensive comparative investigations of physico-chemical and electro-optic characteristics of liquid crystal compounds.

Tables XI and XII illustrate phase transition points and calculated from mixtures with ZLI-1132 values of dielectric, optic and viscous properties for the fifth homologs of new pyridine derivatives in comparison with those for other chemical structures.

Tables XIII and XIV show properties of mixtures containing each a pentyl and heptyl component in molar proportions 40%: 60% for 2-cyano-5-(trans-4-alkylcyclohexyl)pyridines and LCs of other chemical structures. Because of structural similarity it is reasonable to compare properties of the first three classes of pyridines (1Py–3Py) containing along with the pyridine ring also a phenyl one, with 4,4'-cyanobiphenyls (CB) and 5-alkyl-2-(4-cyanophenyl)pyrimidines (Pyr) and accordingly, properties of pyridines with cyclohexane ring (4Py and 5Py) with 4-(trans-4-alkylcyclohexyl)benzonitriles (PCH).

On the whole, the first group of pyridine derivatives in terms of properties is intermediate between CB and Pyr. Both have similar Δn values, higher clearing points T_{cl} than those for CB, their dielectric properties depend on the position of the nitrogen atom in the pyridine ring: for 3Py $\Delta\varepsilon$ is comparable with that for CB; $\Delta\varepsilon$ for 1Py is higher and for 2Py lower than for CB; and for all of them $\Delta\varepsilon$ is lower than for Pyr. The ratio K_3/K_1—an important parameter defining LCM multiplexing capabilities—is lower for 1Py than for CB but does not attain the value of K_3/K_1 for Pyr which is the lowest for the compounds studied. Viscosities of 1Py and 2Py are close to that for Pyr while viscosity for 2Py is anomalously high. Activation energy of rotational viscosity for this group of compounds is high, though slightly

TABLE XI

Physico-chemical properties of some compounds

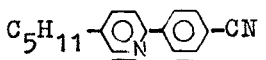
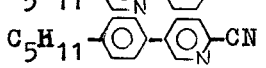
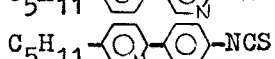
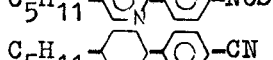
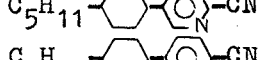
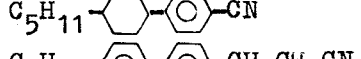
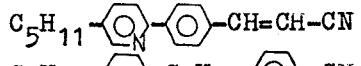
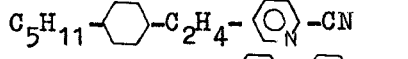
N	Formula	T_{c-s} ($c-n$) °C	T_{s-n} ($s-i$) °C	T_{n-i} °C	Δε, 20°C	Δn, 20°C	γ, mm²/s 20°C
1.		32,9	-	43,5	18,9	0,214	50
2.		73,0	-	(31,0)	17,9	0,204	
3.		34,0	98,5	-	15,5	0,220	18
4.		45,4	-	55,4	17,3	0,120	41
5.		30,0	-	55,0	13.3	0,130	23 /17/
6.		67,5	-	155,1	17,0	0,323	
7.		86,5	-	(53,0)	16,2	0,115	
8.					28,3	0,233	

TABLE XII

Physico-chemical properties of some compounds

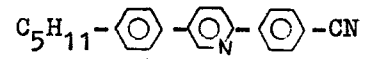
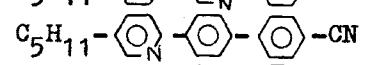
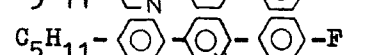
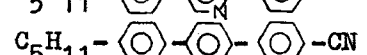
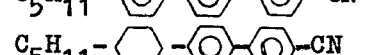
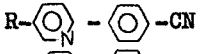
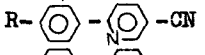
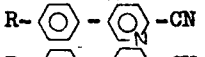
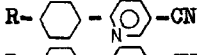
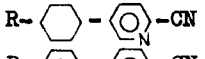
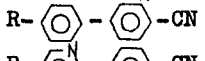
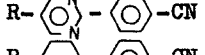
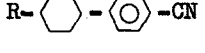
N	Compounds	T_{c-s} ($s-n$) °C	T_{s-n} ($s-i$) °C	T_{n-i} °C	Δε, 20°C	Δn, 20°C	γ, mm²/s 20°C
1.		76,0	-	232,0	19,6	0,344	115
2.		90,0	162,0	254,0	21,4	0,344	-
3.		150,0	(126,0)	-	8,3	0,179	-
4.		131,0	-	239,0	13,0	0,190	90/16?
5.		96,0	-	222,0	12,0	0,190	90/17/
6.		124,0	-	228,0	17,6	0,232	
7.		136,0	168,5	310,0	17,6	0,404	

TABLE XIII

Physico-chemical properties of some binary mixtures

Signature	Formula	$T_{cl.}$ °C	γ_1, 25°C Poise	E eV	$K_1 \times 10^{12}$ N, 25°C	$K_1 \times 10^{12}$ N, $\tau = 0{,}95$	K_3/K_1 $\tau = 0.95$	$\varepsilon_\perp$ $\tau = 0{,}95$	$\Delta\varepsilon$ $\tau = 0{,}95$	$\Delta\varepsilon/\varepsilon_\perp$ $\tau = 0{,}95$	Δn $\tau = 0{,}95$
1Py	R-  -CN	44,2	1,84	0,540	7,50	6,80	1,25	10,7	16,0	1,5	0,175
2Py	R-  -CN	62,7	3,50	0,505	14,0	8,66	1,11	8,6	7,3	0,85	0,172
3Py	R-  -CN	49,2	2,20	0,495	8,25	7,25	1,31	11,25	11,3	1,0	0,175
4Py	R-  -CN	57,3	1,40	0,485	8,80	6,00	1,37	7,5	3,3	0,44	0,104
5Py	R-  -CN	58,0	1,78	0,480	7,46	5,88	1,53	9,95	12,4	1,25	0.093
CB	R-  -CN	39,0	1,1	0,546	7,65	8,01	1,36	6,0	11,7	0.95	0,184
Pyr	R-  -CN	51,2	1,9	0,565	10,2	8,03	1,06	8,7	19,6	2,86	0,174
PCH	R-  -CN	56,4	1,0	0,41	11,5	7,6	1,68	5,4	9,2	1,71	0,100

$\tau = T_{meas}/T_{cl.K}$ K-reduced temperature.

TABLE XIV

Electrooptical properties of some binary mixtures

L.C.	U_{90}, V	U_{10}, V	$U_{meas.}$, V	τ^1 on ms	τ^2 rise ms	τ off ms
1Py	1,08	1,54	3/2,5	75/130	20/40	105/95
2Py	1,90	2,63	5	130	30	60
3Py	1,53	2,16	3	130	30	105
4Py	2,50	3,45	5	130	20	43
5Py	1,25	1,82	3/2,5	114/164	30/45	95/80
CB	1,30	1,78	3/2,5	85/117	21/32	69/64
Pyr	1,07	1,48	3/2,5	70/110	20/35	90/80
PCH	1,67	2,32	3	133	33	46

lower than for CB and Pyr (but significantly higher than for PCH).[12] The second group of pyridine cyano derivatives have their T_{cl} and Δn values close to those for PCH while dielectric anisotropy varies from +12.4 for 5Py to +3.3 for 4Py. Here again the values are associated with the position of the nitrogen atom in the pyridine ring. Viscosity—in spite of the presence of the cyclohexane ring—is rather high as compared to γ_1 for PCH. Comparison of viscosity for 4Py and 2Py, as well as for 5Py and 3Py shows a common for such pairs tendency: replacement of the phenyl ring bound with an alkyl group with cyclohexane in cyano derivatives leads to reduction in viscosity and its activation energy.[14] K_3/K_1 also grows[15] but these values for 4Py and 5Py are significantly lower than for PCH.

Table XIII shows that by chosing a specified structure of pyridine cyano derivatives or their specific blend in a mixture it becomes possible to vary their physicochemical properties in a wide range and achieve a set of desired parameters. Thus dielectric anisotropy for the given five structures varies from +16.0 to 3.3, $\Delta\varepsilon/\varepsilon_\perp$ from 1.5 to 0.44 (values $\Delta\varepsilon/\varepsilon_\perp < 1$ are typical only for pyridines), K_3/K_1 from 1.25 to 1.53, Δn from 0.093 to 0.175. The possibility to vary parameters $\Delta\varepsilon/\varepsilon_\perp$ and K_3/K_1 combined with selecting Δn in a wide range makes the pyridine cyano derivatives very suitable for development of high multiplexing LCM (in particular, for super-twist effects).

Analysis of data given in Tables V–XIII shows that the revealed trends for changes in properties of LC compounds (Tables XI and XII) extrapolated via mixtures with ZLI-1132 are confirmed by investigation of properties of individual LC components and double mixtures for these classes of compounds in those cases when direct measurements were possible (Tables V–X and XIII).

CONCLUSION

The obtained results provide good grounds to consider the discussed pyridine derivatives very suitable components for LC materials to applied in electro-optic devices.

Acknowledgment

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