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Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:

http://www.tandfonline.com/loi/gmcl19

Dependence of Mesomorphic Properties of 3,5-Disubstituted 1,2,4-Oxadiazoles on Geometric and Electronic Factors

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To cite this article: Liudmila A. Karamysheva, Sofia I. Torgova, Irina F. Agafonova & Nkolai M. Shtikov (1995): Dependence of Mesomorphic Properties of 3,5-Disubstituted 1,2,4-Oxadiazoles on Geometric and Electronic Factors, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 260:1, 217-225

To link to this article: http://dx.doi.org/10.1080/10587259508038693

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^a Organic Intermediates and Dyes Institute, Moscow, Russia Version of record first published: 23 Sep 2006.

DEPENDENCE OF MESOMORPHIC PROPERTIES OF 3,5-DI-SUBSTITUTED 1,2,4-OXADIAZOLES ON GEOMETRIC AND ELECTRONIC FACTORS

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Abstract New three and four rings containing liquid crystalline 3,5-disubstituted 1,2,4-oxadiazoles with 4-substituted phenyl, cyclohexyl, biphenyl and phenylcyclohexyl substituents have been synthesized. It was shown that structure and nature of the cyclic fragments, position of the strong polar (NO₂) or weak polar (Alk, AlkO, Hal) groups in respect to the heterocyclic unit significantly effect on the temperature and dielectric properties of the new mesogens.

INTRODUCTION

For the first time the possibility of manifestation of the mesomorphic properties in 3,5-disubstituted 1,2,4-oxadiazoles was demonstrated in our preceding work¹. 1,2,4-Oxadiazole is asymmetrical polarized five-membered heterocycle ²

$$R_1$$
 R_1
 R_2
 R_1

It was shown that the temperature and dielectric properties of the new mesogens are strongly depend on the nature of the substituents and on theirs position in the heterocycle. In the present work such investigations were continued in the series of three and four rings containing 3,5-disubstituted 1,2,4-oxadiazoles, with phenyl, cyclohexyl, biphenyl, phenylcyclohexyl substituents and terminal weak polar groups (Alk, AlkO, F,Br) and strong polar NO₂-group.

RESULTS AND DISCUSSION

The synthesis of the new 3,5-disubstituted 1,2,4-oxadiazoles was described in our previous work 1. The transition temperatures were measured using Mettler-FP-5 apparatus with polarizing microscope.

The dielectric constants for 1,2,4-oxadiazoles, containing NO₂-group were calculated by extrapolation from measurements on 5% by weight solutions in a mixture LCM-1289 ($T_{NI} = 62$ °C, $\epsilon_{\perp} = 5.6$, $\epsilon_{H} = 16.4$) at 20 °C. Measurements were carried out at a frequency of 1 kHz using a E 7-8 capacitance bridge. The dielectric constants were measured at the parallel plate capacitors (thickness 50 µm) with oriented layers of LC.

The transition temperatures of 1,2,4-oxadiazoles with weak polar substituents are presented in the Tables I and II, respectively. As a rule three rings containing 1,2,4-oxadiazoles (Table I) are nonmesomorphic or exhibit monotropic nematic properties. The biphenyl substituent in position 3 (compounds 1,2) causes an enantiotropic smectic phase. Symmetrization of a molecule (compare compounds 1,2 and 3) results in the disappearance of a mesophase. There are no clear correlation in the temperature properties and position of a weak polar groups (Alk, AlkO, Br, F) with respect to the heterocycle (compare reverse structures: 3,4 and 5, 6 and 9, 16 and 18, 17 and 19). In comparison with electronic factors the role of geometric factors in this series is more essential. For example the prolongation of terminal Alk radicals traditionally leads to monotropic nematic (compounds 3,4) or enantiotropic nematic phases (the pairs 6 and 7, 8 and 9, 10 and 11). Replacing the benzene ring with cyclohexane one in oxazolic part of molecule causes in occurrence of mesomorphic behavior (compounds 3 and 6, 15 and 16). In contrary such a change in the diazolic part of a molecule results in a disappearance of mesomorphic properties (compounds 5 and 8).

Additional introduction in a molecule of the second polarizable group such as AlkO or double bond leads to the expected significant increase of the phase transition temperatures (compare compounds 3, 4 and 10, 11; 13 and 15 and also 12, 20, 21) and what's more in the last compounds there are enantiotropic smectic and nematic phases.

In four rings containing oxadiazoles (Table II) a trans-phenylcyclohexane fragment in the diazolic part of the molecules (compounds 22-25) has a nematogenic character. The presence of this fragment in the oxazolic part (compounds 26, 27) results in smectic properties. 4-Alkylbiphenyl substituent in oxazolic part (position 5) is characterized by the smectogenic activity too (compounds 28, 29). But the temperature range of mesophase in cyclohexane containing LCs is greater than in aromatic analogous. In contrast to three rings containing molecules biphenyl substituent in position 3 of four rings containing oxadiazoles has nematogenic effect. The last one is more significant when the Alk

TABLE 1 Three rings containing 1,2,4-oxadiazoles.

1	otem-	Transition of peratures,		R ₂	R ₁		NN
3 $C_7H_{15}O-O-$ 4 $C_7H_{15}O-O-$ 5 $C_5H_{11}-O-$ 6 $C_7H_{15}O-O-$ 7 $C_7H_{15}O-O-$ 8 C_4H_9-O- 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 12 $C_7H_{15}O-O-$ 13 $F-O-$ 14 $C_7H_{15}O-O-$ 15 $C_7H_{15}O-O-$ 16 $C_7H_{15}O-O-$ 17 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 19 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 12 $C_7H_{15}O-O-$ 13 $C_7H_{15}O-O-$ 14 $C_7H_{15}O-O-$ 15 $C_7H_{15}O-O-$ 16 $C_7H_{15}O-O-$ 17 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 19 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 12 $C_7H_{15}O-O-$ 13 $C_7H_{15}O-O-$ 14 $C_7H_{15}O-O-$ 15 $C_7H_{15}O-O-$ 16 $C_7H_{15}O-O-$ 17 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 19 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 12 $C_7H_{15}O-O-$ 13 $C_7H_{15}O-O-$ 14 $C_7H_{15}O-O-$ 15 $C_7H_{15}O-O-$ 16 $C_7H_{15}O-O-$ 17 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 19 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 12 $C_7H_{15}O-O-$ 13 $C_7H_{15}O-O-$ 14 $C_7H_{15}O-O-$ 15 $C_7H_{15}O-O-$ 16 $C_7H_{15}O-O-$ 17 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 19 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 12 $C_7H_{15}O-O-$ 13 $C_7H_{15}O-O-$ 14 $C_7H_{15}O-O-$ 15 $C_7H_{15}O-O-$ 16 $C_7H_{15}O-O-$ 17 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 19 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 10 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 11 $C_7H_{15}O-O-$ 12 $C_7H_{15}O-O-$ 13 $C_7H_{15}O-O-$ 14 $C_7H_{15}O-O-$ 15 $C_7H_{15}O-O-$ 16 $C_7H_{15}O-O-$ 17 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 18 $C_7H_{15}O-O-$ 19 $C_7H_{15}O-O-$ 10 C_7H_{15}	I	100 s 107 3	C	-C ₅ H ₁₁	<u>,</u> o-O-O	C7H15	1
4 $C_7H_{15}O-O-O-O-C_7H_{15}$ C 63 (N 60.7) 5 $C_5H_{11}-O-O-O-O-C_4H_9$ C 91 (N 86) I 6 $C_7H_{15}O-O-O-O-C_4H_9$ C 29 (N 43.5) I 7 $C_7H_{15}O-O-O-O-C_5H_{11}$ C 29 (N 43.5) 8 $C_4H_9-O-O-O-C_4H_9$ C 80 I 9 $C_4H_9-O-O-O-C_4H_9$ C 93 (N 84) I 10 $C_7H_{15}O-O-O-O-O-C_4H_9$ C 93 (N 84) I 11 $C_6H_{13}O-O-O-O-O-C_7H_{15}$ C 54 S 91 N 10 12 $C_7H_{15}O-O-O-O-C_7H_{15}$ C 54 S 91 N 10 13 $F-O-O-O-C_7H_{15}$ C 87 I 14 $Br-O-O-O-C_5H_{11}$ C 60 I 15 $F-O-O-C_5H_{11}$ C 60 I 16 $F-O-O-C_5H_{11}$ C 53 (N 42) I 17 $Br-O-O-C_5H_{11}$ C 74 I 18 $C_4H_9-O-O-F$ C 84 I	I	93 S 117 I	C	-c ₂ H ₅	;o-O-O	C7H15	2
5 $C_{5}H_{11}-O$ $-O$ $-O$ $C_{4}H_{9}$ C 91 (N 86) I 6 $C_{7}H_{15}O$ $-O$ $-C_{4}H_{9}$ C 38 (N 33) I 7 $C_{7}H_{15}O$ $-O$ $-C_{5}H_{11}$ C 29 (N 43.5) 8 $C_{4}H_{9}-O$ $-O$ $-O$ $-O$ $-O$ $-O$ $-O$ $-O$		39 I	C	-(0)-c ₂ H ₅	50 -	C7H15	3
6 $C_7H_{15}O-O C_4H_9$ C_5H_{11} C_29 (N 43.5) 8 $C_4H_9-O C_5H_{11}$ $C_7H_{15}O-O C_5H_{11}$ $C_7H_{15}O-O C_7H_{15}$ $C_7H_{15}O-O C_7H_{15}O-O C_7H_{15}O$	7) I	63 (N 60.7)	C	-©-c ₇ H ₁₅	50 - (0)	C7H15	4
7 $C_7H_{15}O-O-O-O-O_{2}H_{15}$ C 29 (N 43.5) 8 $C_4H_9-O-O-O_{2}H_{15}$ C 30 N 34.5 I 10 $C_7H_{15}O-O-O-O-O-O_{2}H_{15}$ C 30 N 34.5 I 11 $C_6H_{13}O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-$	I	9 1 (n 86) 1	C	-(0)-∞ ₄ H ₉	ı - ⊙	C5H11	5
8 $C_{4}H_{9}$ — — — — — — — — — — — — — — — — — — —	I	; 38 (N 33)]	C	$-C_4H_9$;0 - 0}	C7H15	6
9 $C_{4}H_{9}$ $ 0$ $ 0C_{7}H_{15}$ $ 0$ 0 0 0 0 0 0 0 0 0	5) I	3 29 (N 43.5)	C	-O-0 ₅ H ₁₁	50-(0) - -	C7H15	7
10 $C_7H_{15}O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-O-$		3 80 I	C	-(0)-oc ₄ H ₉	- -	C4H9-	8
11° $C_{6}H_{13}O-O$ — $O-OC_{9}H_{19}$ C 93 N 109 I 12 $C_{7}H_{15}O-O$ — $-CH=CH-O$ — $C_{7}H_{15}$ C 54 S 91 N 10 13 $F-O$ — $O-OC_{7}H_{15}$ C 87 I 14 $Br-O$ — $O-OC_{4}H_{9}$ C 125 (N 100) 15° $F-O$ — $O-C_{5}H_{11}$ C 60 I 16° $F-O$ — $O-C_{5}H_{11}$ C 74 I 18° $C_{4}H_{9}-O$ — $O-F$ C 80 I 19 $C_{4}H_{9}-O$ — $O-Br$ C 84 I	I	30 N 34.5	C	-©-∞ ₇ H ₁₅	- -	C4H9-	9
12 $C_7H_{15}O-O$ — $CH=CH-O$ — C_7H_{15} C 54 S 91 N 10 13 $F-O$ — $O-OC_7H_{15}$ C 87 I 14 $Br-O$ — $O-OC_4H_9$ C 125 (N 100) 15* $F-O$ — $O-C_5H_{11}$ C 60 I 16* $F-O$ — C_5H_{11} C 74 I 18* C_4H_9-O — C_5H_{11} C 80 I 19 C_4H_9-O — C_9 —	I	93 (N 84) I	C	- (0)-∞ ₄ H ₉	;o- (0)	C7H15	10
13 F- \bigcirc -	I	93 N 109 I	C	-(0)-00 ₉ H ₁₉	,o- (0)	C6H13	11*
14 Br- \bigcirc -	101 I	54 S 91 N 1	H ₁₅ C	-CH=CH-(0)-C	50 -(0)	C7H15	12
15* F- \bigcirc -		87 I	C	-00-007H ₁₅)- -	F- (0)	13
16* F- \bigcirc - \bigcirc -C ₅ H ₁₁ C 53 (N 42) I 17 Br- \bigcirc - \bigcirc -C ₅ H ₁₁ C 74 I 18* C ₄ H ₉ - \bigcirc - \bigcirc -F C 80 I 19 C ₄ H ₉ - \bigcirc - \bigcirc -Br C 84 I	O) I	; 125 (N 100)	C	-(0)-00 ₄ H ₉	<u>)</u>	Br-O	14
17 Br- \bigcirc - \bigcirc -C ₅ H ₁₁ C 74 I 18* C ₄ H ₉ - \bigcirc - \bigcirc -F C 80 I 19 C ₄ H ₉ - \bigcirc - \bigcirc -Br C 84 I		60 I	C	-©-c ₅ H ₁₁)	F-O	15*
18* C ₄ H ₉ -\(-\) -\(-\) -\(\) -\(\) -\(\) -\(\) C 80 I 19 C ₄ H ₉ -\(-\) -\(\) -\(\) -\(\) -\(\) C 84 I	I	53 (N 42)]	C	-C ₅ H ₁₁)	F-O	16*
19 C ₄ H ₉ -O- C 84 I		74 I	C	-C ₅ H ₁₁	<u>-</u>	Br-(0	17
7 4 9 0		80 I	C	-(0)-F	○- -	C ₄ H ₉ -⟨	18*
20 F-0H-0H-0H-0H-0 R0 R 92 N 10		84 I	C	-(0)-Br	○	С ₄ н ₉ -(19
20 F-(0)CH=CH-(0)-C ₇ H ₁₅ C 80 S 92 N 10	105 I) 80 s 92 n 1	H ₁₅ C	-CH=CH-(O)-C	-	F-(0)	20
21 Br-{0}CH=CH-{0}-C ₇ H ₁₅ C 107 S 117N12	M122I	107 S 117N	,H ₁₅ С	-CH=CH-(O)-C	<u>-</u>	Br-O	21

^{*} Compounds have been synthesized in 1.

TABLE II Four rings containing 1,2,4-oxadiazoles.

NN	F.	R2	Transition temperatures, O _C	A Trem'	ATnem, AT,	٠° م
55	05H11	-(0)-c5H11	C 62 N 190 I	1	128	128
23	0,5H10	-(0)-00,H15	C 65 S 68 N 182 I	K	114	117
24	0,FH10	-∞-∞ ₄ H ₉	C 75 N 195 I	1	120	120
25	Control	O-05H11	C 63 S 66 N 164 I	W	98	5
5 6	6,E11-0	O C 5H1	C 68 S 163 N 194 I	<u>ام</u>	31	132
22	C7H150-	(0 C 5 H11	C 41 S 169.5 N 178.5 I	128.5	0	137.5
28	C5H1-Q-	-0-0-5H1	G 82 S 180 N 197 I	98	17	115
29	29 C7H150-{O}-	() -() -() -() -() -() -() -() -() -() -	C 69 S 180 N 188 I	111	ω	119
30	05H1	-C5H1	G 53 S 110 N 155 I	23	45	2 8
23	0,H,50-(0)-	-C ₅ H₁1	C 101 S 103.5 N 187 I	2.5	83.5	88
35	C/H2000-00-	-0-c ₇ H ₁₅	C 98 S 101 N 182 I	М	ळ	\$
33	05H1-(0)-(0)-	Ochi,	c 67 s 79 n 159 i	12	80	95

TABLE II (continued)

≴	C7H150-(0)-(0)-	O-05H1	C 72 S 113 N 159 I	4	9#	87
35	C5F1-0	-CH2-(0)-C5H11	c 70 I	ı	i	ı
36	65H100	-Co-Co-H15	C 67 N 175.5 I	1	108.5	108.5 108.5
37	0,5H10	-CH=CH-(O)-C ₂ H ₁₅	C 68 S 71 N 215 I	W	4	147
38	C5H1-0-0-0-	-CH=CH-(0)-0,H15	C 97 S 152 N 203 I	55	72	106
33	C7H150-00-0-	-00-00- -00-4H ₉	C 105 N 215 I	1	110	110
40	C7H2000-00-	-(⊙)-Br	C 127 S 212 N 234 I	85	22	107
4	C7H150-(0)-(0)-	†	G 103 S 185 N 220 I	88	35	117
45	-\(\)	~0~0~0°412	c 104 N 214 I	i	110	110

substituent is replacing by the AlkO group (compare the compounds 28 and 30, 30 and 31, 32). Changing the aromatic cycle in position 5 for the saturated one causes a considerable increase of smectic properties (compare pairs 30 and 33, 31 and 34). This experimental fact confirms our conclusion about the smectogenic influence of cyclohexane substituent in position 5 both in individual form and when it is a part of phenylcyclohexane system. Compounds 36-42 demonstrate the influence of additional polar and/or polarizable groups on the mesomorphism of new LCs..

The present experimental investigation of new liquid crystalline heterocycles with weakly aromatic 1,2,4-oxadiazole fragment testifies about predominance of geometric factors in their mesomorphic behavior. The investigated five-membered heterocycle disturbs the linearity of derived compounds. Therefore the essential depression of all transition temperatures of new LCs comparing with the corresponding carbocyclic analogues³. At the same time the self-polarization of this heterocycle correspond to its derivatives an own dipole moment. The arrangement of two polar substituents in Hal containing compound 42 is in agreement with the self-polarization, in the reversed structure 41 it is oppositive. This is reflected on their mesomorphic behaviour.

The introduction of strong polar groups into LCs of oxadiazolic series has to produce compounds with a great value of $\Delta\epsilon$ depending on the position of the polar substituent in respect to the heterocycle. In order to investigate the influence of strong polar substituents on the mesomorphic properties of 3,5-disubstituted 1,2,4-oxadiazoles, the compounds containing NO₂ substituent have been synthesized.

The transition temperatures and dielectric anisotropy of the new mesogens are presented in the Table III. As seen from Table III all compounds have relatively high melting points due to strong intermolecular interactions between heteroatoms. When nitro-group is in the diazole part of the heterocycle (conjugation with diazole link) the melting points are higher than for reversed structure. With the exception of the compounds 51, 52 (with lateral polar substituent) and for 61, 62 when nitrogroup is in meta-position (lateral) of benzene ring. The compounds with four rings (53 - 60) have wide mesophase and high thermal stability. The introduction of the nitro-group in the meta-position (compare 59 and 61,60 and 62) leads to narrowing of the smectic phase and decreasing of the thermal stability, due to increasing of the intermolecular distance.

The dielectric permittivity anisotropy is much greater when nitro-group is in the diazole part of the heterocycle. This fact can be explained by the coordinated influence of the acceptor nitro-group and the heterocycle self-polarization, together with the elongation of the conjugation chain. The optic anisotropy has the same tendency: Δn for compounds 43, 44, 45, 46 is 0.156, 0.161, 0.255, 0.299 respectively.

The Table IV shows the influence of the polarity of the substituents on the mesomorphic properties.

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Mesomorphic properties of NO₂ containing 1,2,4-oxadiazoles with "reversed" structure. H TABLE

N	ਬ	. R.	Phase transition temperatu- AT, res, C	0°0	△€
54	°4 ^H 9	-{O}-NO ₂	c 91 I	1	3.7
77	O ₂ N ₂ O	Q449	C 77 (N 70) I	ı	6.7
45	05H11-(O)-	-(0)-NO ₂	C 73 S 106 N 119 I	9†	11.5
46	O2N-O-	~ C>~ C5H11	C 100 S 114 N 117 I	17	17.6
47	-(O)-0 ⁶ H ⁷ O		C 106 N 116 I	10	
48	O ₂ N-O	-∞ ₄ H ₉	C 140 S 142 N 150 I	10	
64	C7H150-(0)-	-(0)-NO ₂	C 81 S 117 I	36	
20	O ₂ N ₂ O	-(0)-cc,Ft,5	C 87 S 115 I	28	
2	C8H17g-{O}-	$-\langle 0 \rangle$	c 93 N 120 I	27	6•9
52	°2N-(O)-	$\langle 0 \rangle_{\mathbb{F}}^{-\infty_8 \mathbb{H}_{17}}$	C 71 S 126 I	55	16.7
23	0,5H10	-(0)-No ₂	C 138 S 236 I (decomp.)	98	6.1

TABLE III (continued)

Ż	O2N-O	O O SHI	C 140 S 230 N 244 I	5	13.1
	°3H7 ○○	CO-NO ₂	c 115 s 140 n 204 I	89	2.6
	-\O\NZO	O Contraction	c 122 s 125 n 258 I	136	12.5
	C, Hoo	ON-NO ₂	C 126 S 148 N 222 I	96	
	O2N-O		c 129 s 153 n 244 I	115	
	05H1-0-0-	CO-NO ₂	0 119 S 169 N 256 I(de	c.) 137	10.7
	OZN-O	O C Potent	C 121 S 176 N 247 I 126	126	
	a5H11		C 134 S 137 N 172 I	38	4.5
	-ONZO	-0-0-0-11	C 97 S 101 N 160 I	63	
	Br O	-(O)-NO ₂	c 175 I	1	5.0
	O2M-©-	-R-	C 180 I	ı	
	F-0-	<0>-No ₂	C 178 I	ţ	8•4
	02M-(O)-	4-0	c 198 I	1	

TABLE IV The influence of the substituents polarity on the mesomorphic properties.

R ₁	R ₂	Phase transiti on temperature OC	
C4H9-		C 14 N 29 I	0.03
с ₄ н ₉ -О-	(0)- F	C 80 I	2.1
C4H9	- 0 -80 ₂	C 91 I	3•7
с ₄ н ₉	- О-си	c 100 (N 99)I	5.8
F- O-	-С ₄ н ₉	C 50 (N 42) I	4.8
B r- (0)-	- С -с ₄ н ₉	C 68 I	5.0
02N-0	-С ₄ н ₉	C 71 (N 70) I	6.7
MC-O-	-С ₄ Н ₉	C 114(N 93) I	10.42

CONCLUSION

The present work is a continuation of investigations in the sphere of new liquid crystalline heterocycles -1,2,4-oxadiazoles. Listed examples confirm the weakly aromatic behavior of this five-membered heterocycle and the predominance of geometric factors in mesomorphic properties of derivatives with nonpolar or weak polar terminal substituents. The introduction of strong polar NO₂-group increases the role of electronic factors. Finally the value of $\Delta \varepsilon$ in LCs with NO₂-substituent depends on the sum of two dipole moments: the own dipole moment of heterocycle and the own dipole moment of polar NO₂-group.

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