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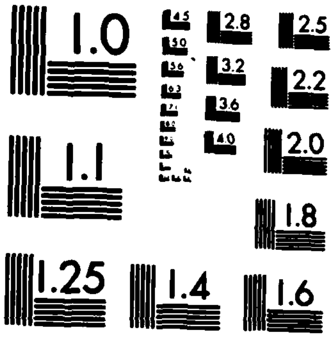

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EFFECTS OF CYCLOHEXYL GROUPS AND ORTHO-SUBSTITUENTS ON THE VISCOSITY OF ESTER LIQUID CRYSTAL COMPONENTS

by

J.D. Margerum, S.-M. Wong, J.E. Jensen,
C.I. van Ast, and A.M. Lackner

To Be Presented at the Tenth International Liquid Crystal Conference,
York, U.K., July 15-21, 1984

Hughes Research Laboratories
3011 Malibu Canyon Road
Malibu, CA 90265

June 1984

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Structural effects on the relative viscosity of liquid crystal ester components are studied by capillary flow viscosity measurements of each component as an additive (10 to 25%) in a standard nematic mixture of short length 4-alkoxyphenyl 4-alkylbenzoates. An approximate class viscosity at 25°C is calculated and assigned to each additive component, indicative of its viscosity contribution in the environment of the phenyl benzoate mixture. Studies are made

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on the viscosity effects of replacing benzene rings with cyclohexane rings, and of using various ortho-substituents on phenyl groups. All of the additive components are esters or thioesters, including several newly synthesized compounds. The effect of a cyclohexyl group is strongly dependent on its position in the structure. The class viscosity values show a large decrease when cyclohexanecarboxlate replaces benzoate, only a modest decrease when cyclohexylbenzoate replaces biphenylcarboxylate, and a slight increase when a cyclohexyl ester replaces a phenyl ester. Although o-groups can be used effectively to alter the dielectric anisotropy and modify the nematic range of a component, they increase the class viscosity values substantially, with the magnitude of the viscosity effect in the following approximate order from several o-groups: Br > CN > CH_3 > pyridyl > Cl.

EFFECTS OF CYCLOHEXYL GROUPS AND ORTHO-SUBSTITUENTS ON THE VISCOSITY OF ESTER LIQUID CRYSTAL COMPONENTS*

J. D. MARGERUM, S.-M. WONG, J. E. JENSEN,
C. I. VAN AST, AND A. M. LACKNER
Hughes Research Laboratories
3011 Malibu Canyon Road
Malibu, California 90265, USA

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1. INTRODUCTION

The effects of chemical structure on liquid crystal (LC) viscosity (η) are important considerations in the selection of components for nematic mixtures used in display applications. For example, it is desirable to be able to predict the viscosity of a new multicomponent LC mixture accurately enough to decide whether or not it is worthwhile to make up a calculated eutectic mixture for study. We have previously described the assignment of approximate class viscosity values (η_{class}) to eighteen different classes of ester LC structures[1], and we have shown that these η_{class} values are useful in predicting the viscosity of new mixtures.[1,2] We are particularly interested in developing additional new LC mixtures with low viscosity, negative dielectric anisotropy, and wide nematic temperature range for both dynamic scattering and field-effect display applications. In working toward these goals, we report here more detailed studies on the viscosity effect of structural changes in LC esters in which phenyl groups are replaced by cyclohexyl groups, or in which o-substituents are used on phenyl groups. Twenty additional ester structures are assigned η_{class} values, and systematic comparisons are made on the changes in class viscosities with structural variations.

2. EXPERIMENTAL

Flow viscosity studies are made with 29 different ester LC components. These are studied as additives in a standard mixture (HRL-2N42) of five 4-alkoxyphenyl 4-alkylbenzoate esters.[1,2,3] The capillary flow viscosity and density data are taken at 25°C with 10 to 25 mole percent of the components added to HRL-2N42, as previously described.[1] The code symbols used here for the individual compounds and their general classes are similar to those defined previously.[1-5] In our code a dash, -, is for the central phenyl benzoate structure, with alternate central structures represented by S for phenyl thiobenzoate, -[C] for phenyl cyclohexanecarboxylate, [C]- for cyclohexyl benzoate, [C]-[C] for cyclohexyl cyclohexanecarboxylate, and [Py]- for *o*-pyridyl benzoate. Additional rings in the structure are represented by P for phenyl and Y for cyclohexyl, while only *o*-substituents are shown in parenthesis, *e.g.* 1(Br)-Y3 stands for *o*-bromo-*p*-tolyl *p*-(4-propylcyclohexyl)benzoate. Twenty of the 29 LC compounds investigated are synthesized, and the other nine are commercial samples obtained from either E. Merck or from Atomergic Chemetals.

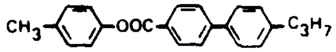
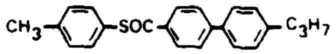
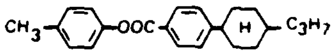
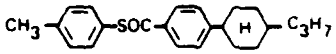
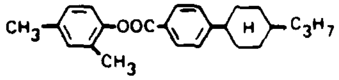
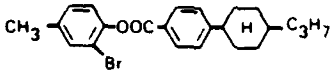
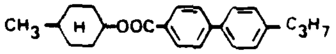
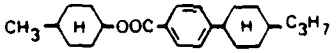
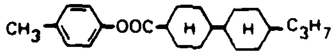
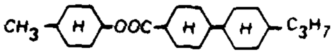
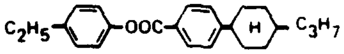
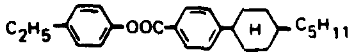
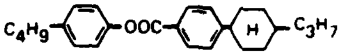
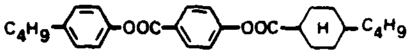
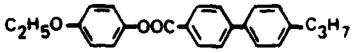
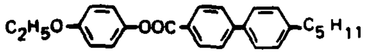
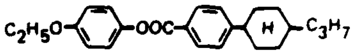
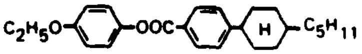
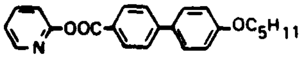
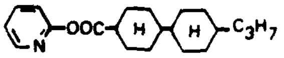
Nineteen of the synthesized esters are prepared by reacting purified samples of commercially available (Aldrich) phenols or thiophenols with acid chlorides, whose acid intermediates are obtained by hydrolysis of the corresponding nitrile LC compounds (E. Merck). The other diester (4-OOCY4) is made using intermediates prepared as described before, namely the monoester 4-OH phenol[1] and the trans-4-butylcyclohexanecarbonyl chloride.[4] Thermal analysis data on the melting point (mp), clearpoint (clpt), and molar heat of fusion (ΔH_f) of the new compounds are obtained by differential scanning calorimetry.[3] The purity of all of the LCs is checked by thin layer chromatography and by liquid chromatography.[1-3]

3. RESULT AND DISCUSSION

3.1 LC Compounds Synthesized

The abbreviation code, structure, melting point, clearpoint, and molar heat of fusion of the 20 LC compounds synthesized are given in Table I. The first 10 of these are all variations of similar three ring structures with the same alkyl end groups (e.g. 1-P3, 1SP3, 1-Y3, etc) and essentially the same molecular length. These structural variations are chosen not only to observe the viscosity effects of thioesters,

Table I. New LC Components Synthesized

CODE	STRUCTURE	mp (°C)	Clpt. (°C)	ΔH_f (kcal/mole)
1-P3		126	199	7.26
1 S P3		126	235	6.12
1-Y3		121	188	5.84
1 S Y3		120	224	4.72
1(CH$_3$)-Y3		79	118	5.18
1(Br)-Y3		97	119	7.12
1(C)-P3		94	126	4.89
1(C)-Y3		91	129	6.35
1-(C)Y3		53(96)[a]	204	5.93
1(C)-(C)Y3		67(129)[a]	153	7.72
2-Y3		104	175	5.13
2-Y5		92	172	6.32
4-Y3		92	174	5.33
4-OOCY4		62(65)[a]	185	7.27
2O-P3		128	235	7.88
2O-P5		126	220	6.08
2O-Y3		127	223	6.39
2O-Y5		106	215	6.39
[P$_y$]-PO5		78(86)[a]	114	3.65
[P$_y$]-(C)Y3		108	118	8.51

[a] SMECTIC TO NEMATIC TRANSITION

cyclohexane groups, and o-substituents, but also to note these structural effects on the nematic temperature range, the birefringence (Δn), and the dielectric anisotropy ($\Delta\varepsilon$). Studies on Δn and $\Delta\varepsilon$ are reported for several of these components in a recent paper.[2] The phenyl thiobenzoate derivatives, 1SP3 and 1SY3, have substantially higher clearpoint and about the same melting point as their corresponding phenyl benzoates, 1-P3 and 1-Y3. These thiobenzoates also have higher Δn and more positive $\Delta\varepsilon$ values than the benzoates.[2] The two o-substituted phenyl benzoates of this group, 1(CH_3)-Y3 and 1(Br)-Y3, each have a lower melting point and clearpoint than either 1-P3 or 1-Y3. The o-substituents also have a large effect on $\Delta\varepsilon$, with 1(CH_3)-Y3 more positive and 1(Br)-Y3 much more negative in $\Delta\varepsilon$ than the unsubstituted R-YR' structures.[2] The cyclohexyl benzoate derivatives 1[C]-P3 and 1[C]-Y3 each have a lower melting point and clearpoint than 1-P3 and 1-Y3. In general, the replacement of a biphenylcarboxylate by a cyclohexylbenzoate has little effect on the nematic range, e.g. each of the following pairs have similar nematic ranges: 1-P3 and 1-Y3, 2O-P3 and 2O-Y3, 1[C]-P3 and 1[C]-Y3. However, the bicyclohexylcarboxylate structures, 1-[C]Y3 and 1[C]-[C]Y3, do have substantially lower melting points than these other structures.

3.2 Viscosity Measurements and Calculations

The flow viscosity measurements and calculated results are summarized in Table II. The LC components are all studied at 25°C as additives to the RO-R' mixture HRL-2N42, which is a five-component eutectic mixture[1,2,3] of 10-1, 20-3, 20-5, 40-1, and 40-3. The observed viscosity of this RO-R' mixture with x mole fraction of the additive(s) is used to estimate the approximate viscosity contribution of the additive (η_{calc}) by applying equation 1 at the average molecular length ($\bar{L}$) of the overall mixture:

$$\eta_{obs} = \eta_{calc}\,(x) + \eta_{RO-R'}\,(1-x) \qquad (1)$$

The host viscosity value $\eta_{RO-R'}$ is assumed to vary linearily with the $\bar{L}$ of the overall mixture, as noted previously[1], and the values are taken from equation 2.

$$\eta_{RO-R'} = 32.5 + 1.54\,(\bar{L}-20) \qquad (2)$$

This provides a small correction for differences in the molecular lengths of the additive. The class value of viscosity for each type of structure is assigned from the η_{calc} values obtained from components of each class.

The η_{calc} values are extrapolations which are assumed to correspond to the relative

Table II. Viscosity Measurements and Calculated Component Viscosity[a]

Code	Source[b]	Length (Å)	Mixed In HRL-2N42 %	Mixed In HRL-2N42 $\bar{l}$ (Å)	Mixed In HRL-2N42 η_{25}, (cP)	η_{calc} (cP)
1-P3	I	21.45	10	20.50	[illegible]	81
1SP3	I	21.80	10	20.74	38.20	79
1-Y3	I	22.68	10	20.62	[illegible]	69
1SY3	I	22.53	10	20.60	36.27	62
1(CH_3)-Y3	I	22.68	25	20.96	61.56	144
1(Br)-Y3	I	22.68	25	20.96	76.04	202
1[C]-P3	I	21.60	10	20.51	39.00	90
1[C]-Y3	I	21.30	10	20.48	37.62	77
1-[C]Y3	I	22.20	10	20.57	31.61	16
1[C]-[C]Y3	I	22.50	10	20.60	31.36	13
2-Y3	I	24.28	25	21.40	41.99[c]	54
2-Y5	I	25.50	25	21.48	42.72	67
4-Y3	I	25.52				
4-OOCY4	I	28.74	25	22.48	50.01	91
2O-P3	I	23.60	10	20.86	40.64[d]	102
2O-P5	I	26.10				
2O-Y3	I	23.70	25	21.60	49.68[e]	94
2O-Y5	I	26.18				
[Py]-PO5	I	23.65	10	20.72	49.72	195
[Py]-[C]Y3	I	20.50	10	20.40	36.83	37
3[C]-Y2	M	23.45	25	21.47	47.57[f]	86
3[C]-Y4	M	25.75				
3[C]-[C]Y4	M	24.90	10	20.84	32.54	21
3Y-[C]4	M	25.65	10	20.92	33.75	32
4(CN)-Y3	M	25.52	10	20.90	48.19	177
5-[C]5	A	25.00	10	20.85	29.48	-9
2OS[C]5	A	22.80	25	20.99	30.63	20
5OS[C]4	A	25.80	25	21.74	35.18	35
NC-[C]5	A	21.85	25	20.76	38.69	54

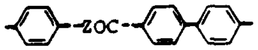
[a] Extrapolated viscosity from effect of component when added to the RO-R' mixture HRL-2N42. [b] I= Table I in this paper, M= E. Merck, A= Atomergic Chemetals. [c] First mixture with 11.2% 2-Y3 and 13.8% 4-Y3; second mixture with 7.8% 2-Y3, 7.9% 2-Y5, and 9.4% 4-Y3. [d] Mixture with 3.9% 2O-P3 and 6.1% 2O-P5. [e] Mixture with 9.8% 2O-Y3 and 15.2% 2O-Y5. [f] Mixture with 11.2% 3[C]-Y2 and 13.8% 3[C]-Y4.

viscosity values of the additives at 25°C in the environment of the RO-R' host. Our comparisons are made assuming that each additive is accommodated by (or disrupts) the LC host similarly. Of course this is not strictly true, as is obvious, for example, in the case of the additive 5-[C]5 which gives a negative η_{calc} value. Nevertheless, we find that the η_{class} values assigned from these η_{calc} values (and from prior studies) give fairly accurate predictions for the η_{25^oC} of many multicomponent ester mixtures.[1,2] Thus, the η_{class} values are suitable for relative comparisons of LC structural effects on the flow viscosity.

3.3 Effects of Cyclohexane Rings

Table III shows the effects on the extrapolated component viscosity of replacing from one to three of the phenyl groups in 1-P3 with cyclohexyl groups, and of replacing one phenyl group in 1SP3 witn a cyclohexyl ring. Each component in Table III has three rings in the same arrangement and has the same alkyl end groups, so that the only structural variation for each ester or thioester is the number and the position of the cyclohexyl groups. The clearpoint of these ester components vary between 126 and 204°C, but since only 10% of each is used in the HRL-2N42 the clearpoints of the mixtures which are used for the viscosity measurements are not significantly different. The structural effects on viscosity in this series are apparent. The replacement of phenyl by cyclohexyl on the "alcohol" side of the ester linkage increases the viscosity: *e.g.* 1[C]-P3 > 1-P3, 1[C]-Y3 > 1-Y3, and 1[C]-[C]Y3 > 1-[C]Y3. The substitution of a cyclohexyl ring for the outside phenyl ring on the biphenylcarboxylate side decreases the viscosity: *e.g.* 1-Y3 < 1-P3, and 1[C]-Y3 < 1[C]-P3. The thioester structure shows a similar effect, *e.g.* the viscosity of 1SY3 < 1SP3. However, these effects are relatively small compared to the large decrease in η_{calc} resulting by replacement of both phenyls on the biphenylcarboxylate side with cyclohexyl rings: *e.g.* 1-[C]Y3 << 1-P3(and 1-Y3), and 1[C]-[C]Y3 << 1[C]-P3 (and 1[C]-Y3).

Table III. Effect on Calculated Component Viscosity of Substituting Cyclohexyl For Phenyl Groups in 1-P3 and 1SP3.

Compound Code	Number of Cyclohexyls	Replacement Position of Cyclohexyl Groups 			$\eta_{calc.}$ (cP, 25°C)
1-P3	0	---	---	---	81
1SP3	0	---	---	---	79
1[C]-P3	1	X	---	---	90
1-Y3	1	---	---	X	69
1SY3	1	---	---	X	62
1[C]-Y3	2	X	---	X	77
1-[C]Y3	2	---	X	X	16
1[C]-[C]Y3	3	X	X	X	19

VISCOSITY OF ESTER LIQUID CRYSTAL COMPONENTS

The comparisons in Figure 1 confirm that there is in general a substantial decrease in class viscosity when a phenyl ring adjacent to ester carbonyl ("acid" side) is replaced by a cyclohexyl ring, e.g. when a benzoate group is

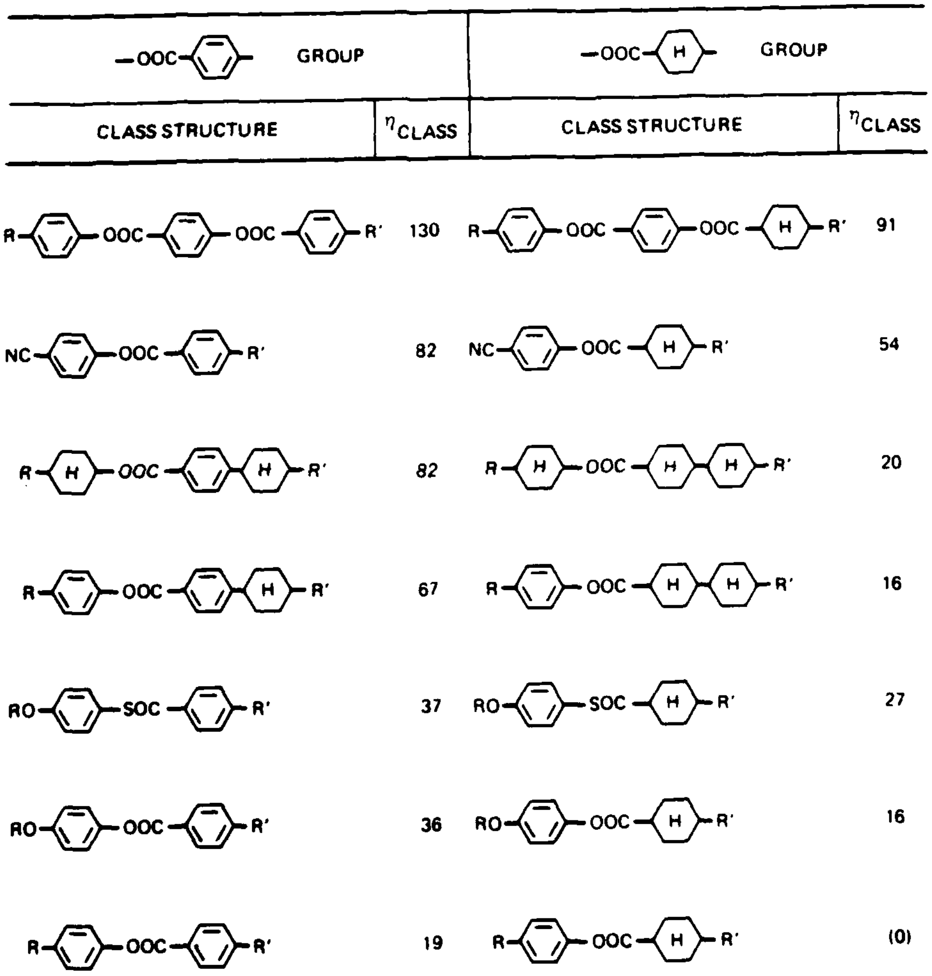


FIGURE 1 Effect on class viscosity of replacing a benzoate group with a cyclohexanecarboxylate group.

replaced by a cyclohexanecarboxylate group. A similar, but smaller decrease in η_{class} occurs in thio-ester structures: *e.g.* ROS[C]R' < ROSR'. Even when only one benzoate group is replaced in a diester structure, a large viscosity decrease is shown: *e.g.* R-OOCYR' < R-OOCPR'. Quite low η_{class} values are observed for the R-[C]YR', RO-[C]R', and R-[C]R' cyclohexanecarboxylate structures, each of whose LC compounds also have the advantage of having higher clearpoints than the corresponding benzoate compounds.

The effects on η_{class} of replacing a phenyl ester with a cyclohexyl ester are summarized in Figure 2. The η_{class} increases by an average of

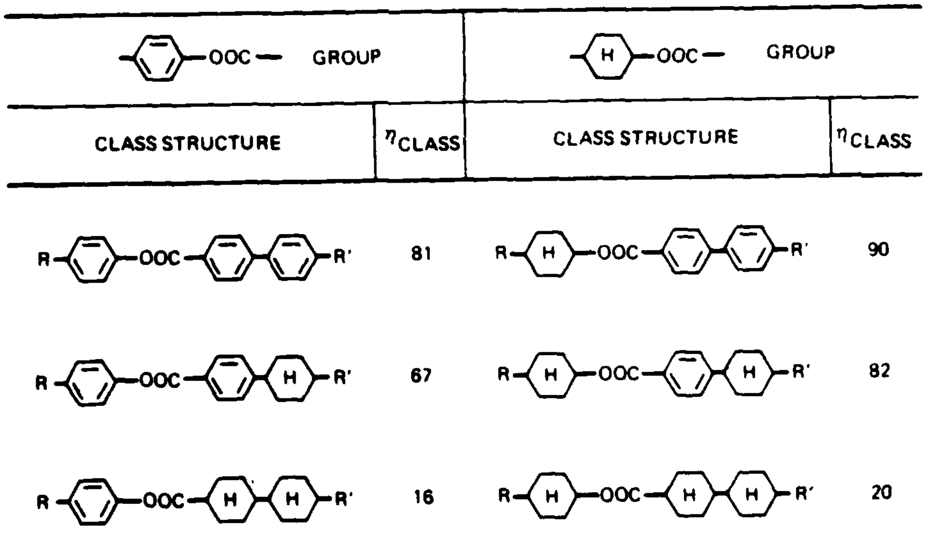


FIGURE 2 Effect on class viscosity of replacing phenyl ester with a cyclohexyl ester.

about 20% when cyclohexyl replaces phenyl on the "alcohol" side of these structures.

The effects of replacing a biphenylcarboxylate group with a cyclohexylbenzoate group are summarized by the η_{class} comparisons in Figure 3. On the average this structural change decreases η_{class} by about 13%.

3.4 Effects of Ortho-Groups

The use of <u>ortho</u>-groups in LC structures is of

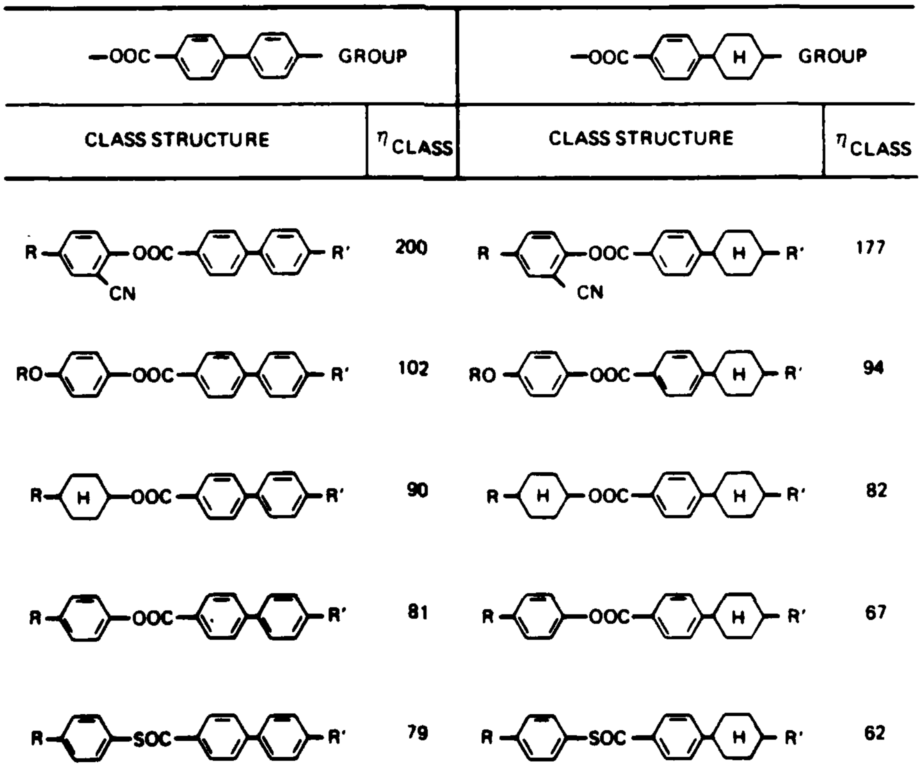


FIGURE 3 Effect on class viscosity of replacing a biphenylcarboxylate with a cyclohexylbenzoate group.

interest mainly as a means of altering the Δε and lowering the mp compared to the structure without the o-group. Among the o-groups whose viscosity effects are studied here, the o-Br, o-pyridyl, and o-CN groups give an increasingly strong negative Δε effect to their parent phenyl ester LC structures, while the o-CH_3 group increases the positive Δε of its parent structure.[2] However, one price paid for the lower mp and the altered Δε is that of a substantial increase in η_{class} of the o-group structures, as shown in Figure 4. The addition of an o-CN or o-Br group greatly increases the viscosity of a structure, generally more than doubling the η_{class}, while the presence of an o-pyridyl group causes a smaller viscosity increase. The addition of an o-CH_3 group strongly increases η_{class}, while an o-Cl group causes a smaller increase. In general the results shown in Figure 4 indicate that the increase of η_{class} due to the presence of o-groups is in approximately the following order: Br > CN > CH_3 > pyridyl > Cl.

4. CONCLUSIONS

1. The replacement of phenyl rings by cyclohexyl rings in ester LC compounds alters their relative value of class viscosity, with the magnitude and the direction of the effect strongly

VISCOSITY OF ESTER LIQUID CRYSTAL COMPONENTS

WITHOUT *o*-GROUP		WITH *o*-GROUP	
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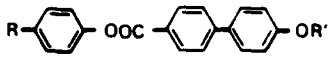
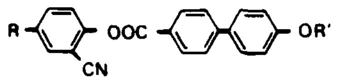
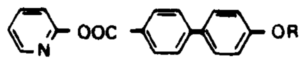
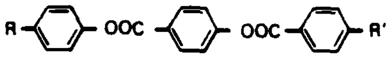
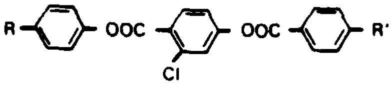
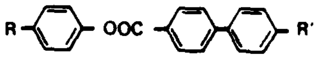
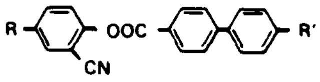
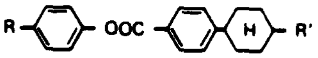
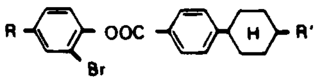
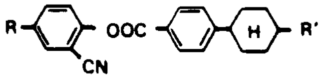
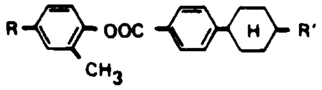
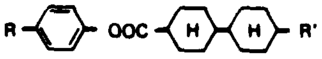
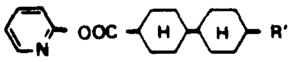
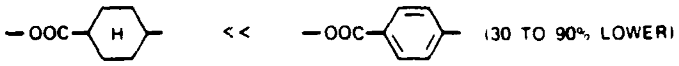
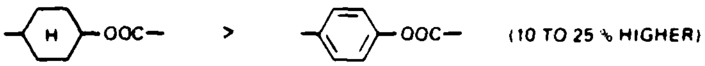
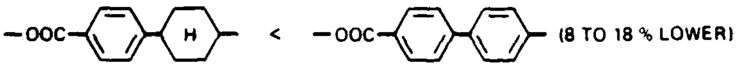
	160		310
			195
	130		153
	81		200
	67		202
			177
			144
	16		37

FIGURE 4 Effect on class viscosity of *ortho*-groups.

dependent upon the position of the cyclohexyl groups in the structure. When the class viscosities are extrapolated from viscosity measurements of the LC compounds as additives in a standard host mixture of alkoxyphenyl alkylbenzoates, then for otherwise comparable structures the effects on η_{class} of replacing a phenyl ring with a cyclohexyl ring are typically as follows:







Thus, substitution of a cyclohexyl for a phenyl ring adjacent to the carbonyl group is by far the most effective way of using cyclohexyl rings to reduce the viscosity of ester LC structure.

2. Although *o*-groups can be used effectively to control the dielectric anisotropy contribution of component structures, and also to lower the melting point, this advantage is offset by a substantial increase in the class viscosity of a structure with an *o*-group as compared to the structure without it. In the present studies the presence of an *o*-group in a component increases the η_{class} value in the range of 20 to 200%. For these structures the magnitude of the *o*-group effect on η_{class} increases as follows:
Cl < pyridyl < CH_3 < CN < Br.

3. The assignment of class viscosities to LC ester structures is useful in comparing structural

effects of cyclohexyl rings and o-substituents on the relative viscosity contributions of ester and thioester LC compounds. The twenty η_{class} values assigned here along with the other eighteen assigned earlier,[1] can be used effectively to predict the flow viscosity of new multicomponent LC nematic mixtures.[2,6]

5. ACKNOWLEDGMENTS

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