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Disc-Like Mesogens: A Classification

C. Destrade^a, Nguyen Huu Tinh^a, H. Gasparoux^a, J. Malthete^b & A. M. Levelut^c

^a Centre de Recherche Paul Pascal, Domaine Universitaire, 33405, Talence, France

^b Laboratoire de Chimie Organique des Hormones, Collège de France, 75231, Paris Cedex, France

^c Laboratoire de Physique des Solides, Batiment, 510, 91405, Orsay, France

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Disc-Like Mesogens: A Classification

C. DESTRADE, NGUYEN HUU TINH and H. GASPAROUX

*Centre de Recherche Paul Pascal, Domaine Universitaire, 33405 Talence,
France*

and

J. MALTHETE

*Laboratoire de Chimie Organique des Hormones, Collège de France, 75231 Paris Cedex,
France*

and

A. M. LEVELUT

Laboratoire de Physique des Solides, Batiment 510, 91405 Orsay, France

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Five mesomorphic phases are described in several disc-like liquid-crystals. They are hexa-alkanoyloxy benzenes, triphenylenes or truxenes, hexa-alkoxy triphenylenes, hexa-benzoates of triphenylene. Several of these compounds exhibit a complex polymorphism. Informations on the structures of these phases have been obtained by means of X-Ray diffraction; except a N_D nematic phase all the others one are columnar phases. At the same time a systematic study has been performed by the well known method of isomorphy. All these results allow us to propose a general classification of these phases.

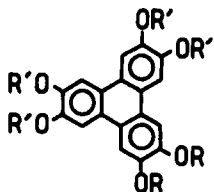
INTRODUCTION

Up to a recent date three different homologous series of disc-like mesogens were known:

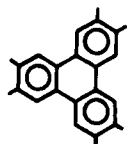
—hexa-*n*-alkanoyloxy benzenes (HAB),¹ hexa-*n*-alkoxy triphenylenes (HET)²⁻⁴ and hexa-*n*-alkanoyloxy triphenylenes (HAT).^{4,5} A fourth one has been recently described: hexa-*n*-alcoyl or alkoxybenzoates of triphenylene (HBT),⁶⁻⁸ at last a fifth one is in progress with a new central rigid core, these compounds are hexa-*n*-alcanoyloxy truxenes (HATX)^{9,10} (Figure 1).



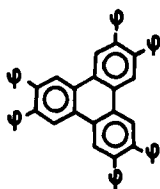
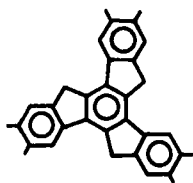
hexa - n - alcanoyloxybenzenes



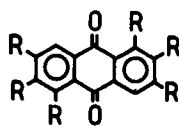
hexa - n - alkoxytriphenylenes

 $R = R'$ (symetric) $R \neq R'$ (dissymetric)

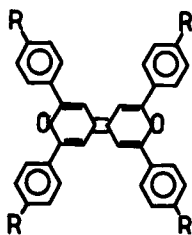
hexa - n - alcanoyloxytriphenylenes

 $R = OCOR$ hexa - n - alcyl or alkoxy
benzoates of triphenylene

hexa - n - alcanoyloxytruxenes



rufigallol hexa - n - octanoate



tetraarylpyrylidene pyranne

FIGURE 1 Chemical formula of the different disc-like mesogens

More punctual attempts have been also successful with some dissymmetric hexasubstituted triphenylenes,¹¹ two pyranes derivatives¹² (Figure 1), and one hexasubstituted rufigallol (RHO).¹³

Most of these compounds exhibit an hexagonal or trigonal symmetry,¹⁻¹⁰ but some of them a binary or tetragonal one.¹¹⁻¹³

The evidence of a complex polymorphism has been shown^{4,5} in several HAT derivatives we have studied, and in most cases the highly viscous and birefringent phases correspond to a tubular or better columnar arrangement of discs^{1,7} (we note these phases "D"). In some HBT derivatives⁶ we have shown the existence of a very fluid phase with typical nematic schlieren textures,⁶⁻⁸ the first demonstration of the existence of a nematic phase in disc-like mesogens.⁷ In fact this new phase corresponds to a structure in which the director vector is perpendicular to the average plane in which the flat molecules are aligned, we call this the nematic phase " N_D ".

All these results confirm definitively the real mesomorphic nature of these new kinds of liquid crystals, in fact a new mesomorphic state of matter.⁷

Up to now six different mesomorphic phases have been clearly identified.^{7,8}

The problem in the classification of classic liquid crystals (rod-like ones) in the last ten years is as suggested by Professor HELFRICH¹⁴ something is like opening a "Pandora's box"! One reason for the numerous controversies on this problem may be, at first, the lack of X-Ray diffraction information on the structures of these phases. In the case of disc-like mesogens such a problem has been avoided: in each series at least one compound has been studied by mean of X-Ray diffraction and the structure of the phases has been obtained. Then using optical textures observations and with the help of miscibility criteria we have identified all the phases of our compounds. Now we can propose a classification based on "objective" criterions such as structure and miscibility.

DISC-LIKE MESOGENS IN THE HOMOLOGOUS SERIES

Hexa-*n*-alcanoyloxy benzenes (HAB) and hexa-*n*-alkoxy triphenylenes (HET)^{1,3,4}

The transition temperatures of the HAB and HET derivatives are reported in Tables 1 and 2.

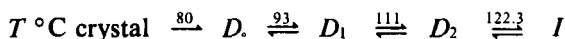
Only one phase is observed in both series. Optical textures are very different in any case but one can find large homeotropic domains revealing the uniaxial nature of these phases (Figure 2a, 2b). HAB derivatives exhibit typical "broken fan" textures¹ (Figure 2a), while with HET ones some typical linear defaults can be observed,³ furthermore, with these last compounds, on cooling, we have observed the appearance of little homeotropic hexagons revealing an hexagonal lattice.⁴ These two phases are highly viscous and the heat of transition mesophase-isotropic very big.^{1,4}

TABLE I
Transition temperatures of the hexa-*n*-alkanoyloxy benzenes (HAB)¹

R = OCO C _n H _{2n+1}						
<i>n</i>	<i>K</i> _{II}		<i>K</i> _I		M	I
6	•	75.7	•	94.5	—	•
7	—		•	81.2	•	87
8	—		•	79.8	•	83.4
9	—		•	75.6	•	85.4
10	•	50.5	•	85.5	—	•

Hexa-*n*-alkanoyloxy triphenylenes^{3,4}

It is within this series that we have found a mesomorphic polymorphism.^{4,5} For example, the hexa-*n*-dodecanoate of triphenylene exhibits three stable phases between 80 and 122°C; at first, we call them considering increasing temperature *D*, *D*₁, *D*₂:



This polymorphism is general with long alkyl chain HAT⁴ (*n* ≥ 10), in contrast with short alkyl chain derivatives in which only one phase is observed. Transition temperatures of this series are reported in Table 3. Some characteristic optical textures are given in Figure 3a, 3b and 3c. They correspond to broken fan, finger prints or focal conics.

Let us point out that the *D*₂ phase is uniaxial while the *D*₁ one seems to be a biaxial one. Heats of transition of mesophase-isotropic are lower in HET or HAB derivatives and some are practically second order transitions (observe *D* - *D*₁ or *D*₁ - *D*₂⁴).

TABLE II
Transition temperatures of the hexa-*n*-alkoy triphenylenes (HET)⁴

<i>n</i>	R = O C _n H _{2n+1}			
	<i>K</i>		<i>D</i>	<i>I</i>
1	•	317	—	•
2	•	247	—	•
3	•	177	—	•
4	•	88.6	•	145.6
5	•	69	•	122
6	•	68	•	97
7	•	68.6	•	93
8	•	66.8	•	85.6
9	•	57	•	77.6
10	•	58	•	69
11	•	54	•	66
12	•		•	
13	•		•	49

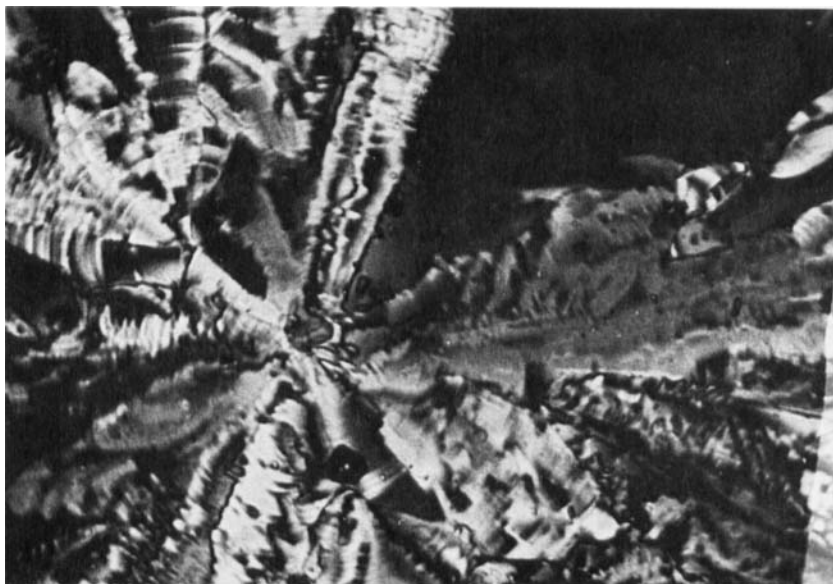


FIGURE 2 Optical texture observed with:
2a: HAB C_7 derivative at 81°C
2b: HET C_5 derivative at 82°C

TABLE III
Transition temperatures of hexa-*n*-alcanoyloxy triphenylenes (HAT)^{4,5}

<i>n</i>	<i>K</i>	<i>R</i> = OCO C _{<i>n</i>} H _{2<i>n</i>+1}				<i>I</i>
		<i>D</i> ₀		<i>D</i> ₁	<i>D</i> ₂	
1	• 296	—		—	—	—
3	• 230	—		—	—	•
4	• 193	—		—	—	•
5	• 146	—		—	—	•
6	• 108	—		• 120	—	•
7	• 64	—		• 130	—	•
8	• 62	—		• 125	—	•
9	• 75	—		• 125.5	—	•
10	• 67	• [56]		• 108	• 121.5	•
11	• 80	• 93		• 111	• 122.3	•
12	• 83	• [81]		• 99.2	• 118	•
13	• 86.5	—		• 96	• 111	•

Hexa-*n*-alcoyl or alkoxy benzoates of triphenylene^{6–8}

In this series, we have recently showed the existence of a new kind of mesophase characterized by a high fluidity and optical textures very similar to nematic ones (Figure 4a). In fact typical nematic schlieren textures are observed; for example, desclination lines with $s = \pm 1/2$ and ± 1 are observed and one can see also thermal fluctuations as with “classic” nematic phases but the optical sign is negative in this new phase; we have called it N_D .^{6*}

We have proposed a molecular organisation in which the different flat molecules present a total orientational and rotational freedom but with their plane, on an average, parallel to a reference plane.⁶ Furthermore, at lower temperatures, several highly viscous phases are observed with optical textures in general similar to previous ones (Figure 4b), but with the hexyloxy benzoate of triphenylene (C₆OHBT) we have observed a typical mosaic texture.⁷ Transition temperatures within this series⁸ are summarised in Table 4.

Dissymmetrical hexa-*n*-alkoxy triphenylenes¹¹

These compounds (Figure 1, Table 5) exhibit only one phase characterized by relatively low transition temperature, crystal-mesophase. Optical textures are in any point similar to those of the corresponding symmetrical alkoxy triphenylenes.

*The nematic nature of this phase has been confirmed by some X-Ray and magnetic measurements.¹⁵

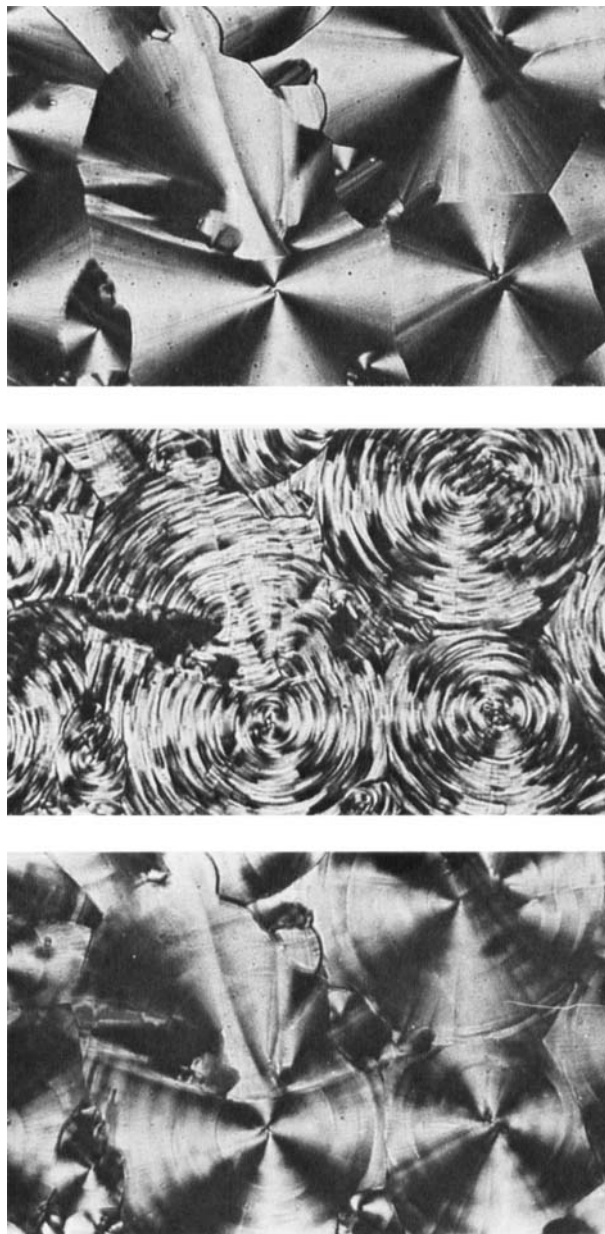


FIGURE 3 Optical textures observed with the C_{11} HAT derivative:
3a: D_2 phase at 112°C
3b: D_1 phase at 180°C
3c: D_0 phase at 92°C

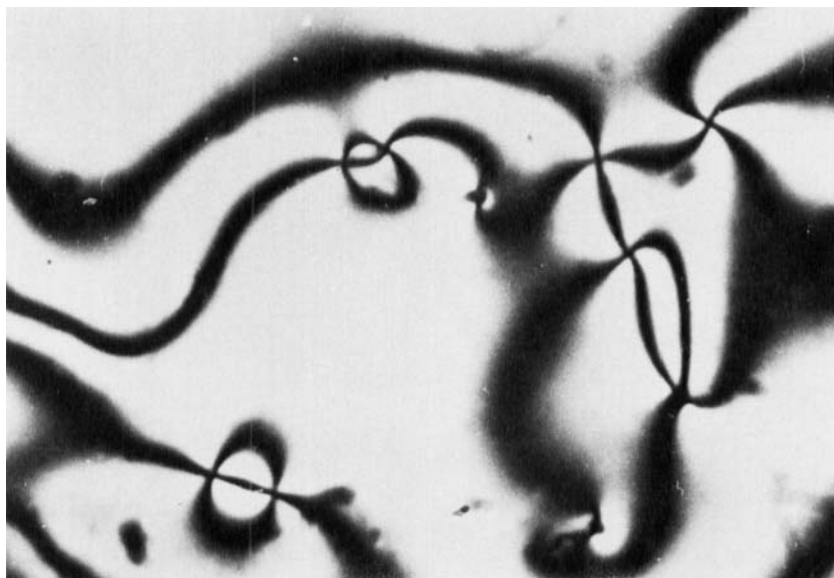


FIGURE 4 Optical texture observed with the C_8O HBT derivative:
4a: N_D nematic phase at $200^\circ C$
4b: Columnar phase at $160^\circ C$

TABLE IV
Transition temperatures of hexa-*n*-benzoates of triphenylene (HBT)^{6,8}

<i>n</i>	<i>K</i>	<i>D_i</i>	$R' = C_nH_{2n+1}$	<i>D_i</i>	<i>N_D</i>	<i>I</i>
7	• 210	—		—	—	•
8	• 208	—		—	• 210	•
9	• 175	—		• 183	• 192	•
10	• 185	—		• 189	—	•
$R' = OC_nH_{2n+1}$						
4	• 257	—		—	• 300	•
5	• 224	—		—	• 298	•
6	• 186	• 193		—	• 274	•
7	• 168	—		—	• 253	•
8	• 152	—		• 168	• 244	•
9	• 154	—		• 183	• 227	•
10	• 142	—		• 191	• 212	•
11	• 145	—		• 179	• 185	•
12	• 146	—		• 174	—	•

Hexa-*n*-alkanoyloxy truxenes (HATX)⁹ (Table 6)

In HBT derivatives a complex polymorphism has been shown,⁶⁻⁸ to correspond to the “normal” sequence:

crystal $\longrightarrow D \rightleftharpoons N_D \rightleftharpoons I$ (D = columnar viscous phase,
 N_D = nematic phase in disc-like mesogens, I = isotropic).

With truxene derivatives we have found a new phenomenon⁹: an inverted nematic-columnar sequence:

crystal $\longrightarrow N_D \rightleftharpoons D \rightleftharpoons D' \rightleftharpoons I$

perhaps the first example of a reentrant nematic phase in disc-like mesogens.

TABLE V
Transition temperatures of dissymmetric hexa-*n*-alkoxy triphenylenes¹¹

<i>n</i>	<i>m</i>	$R = OC_nH_{2n+1}$ $R' = OC_mH_{2m+1}$	<i>K</i>	<i>D</i>	<i>I</i>
6	8	•	47	• 84	•
8	6	•	47	• 84	•
5	9	•	53	• 71	•
9	5	•	54	• 74	•
5	10	•	61	• 63	•
10	5	•	57	• (56)	•
11	5	•	61	—	•

TABLE VI
Transition temperature of hexa-*n*-alkanoyloxy truxenes (HATX)^{9,10}

R = OCO C _n H _{2n+1}							
<i>n</i>	<i>K</i>	<i>N_D</i>	<i>D_r</i>	<i>D_h</i>	<i>I</i>		
6	• 112	• (96)	• 138	• 280†	•		
7	• 98	• (85)	• 140	• 280†	•		
8	• 88	• (87)	• 141	• 280†	•		
9	• 68	• 85	• 138	• 280†	•		
10	• 62	• 89	• 118	• 250†	•		
11	• 64	• 83.5	• 130	• 250†	•		
12	• 57	• 84	• 107	• 249†	•		
13	• 58	• 83	—	• 235†	•		

† with some decomposition of the sample

Furthermore, from an optical point of view, one of these *D* phases, the highest temperature one, is very similar to the one of HET derivative and the lower one *D* to the HAT derivative phase *D*₁.

ISOMORPHY

The isomorphy method is widely used for the identification of “classical” mesophases.¹⁶ Using this method we have performed a systematic study of the previously described polymorphism at one and the same time in each series and between different series. From this study we can emphasize several facts:

—total miscibility is always observed in a given series but of course if there is only one phase in it, that means isomorphy (Figure 5). But if the alkyl chain length is too different the isotropic phase appears just after the eutectic (Figure 6). For example, C₆HET and C₁₀HET are not miscible, but C₆HET and C₈HET on one hand and C₈HET and C₁₀HET on the other hand are miscible so we can conclude that in this series all the phases of the different compounds are similar.

—no example of miscibility has been found between two different series. Furthermore, in the binary thermic diagrams between HAT and HET one can observe a spectacular stabilisation of the last derivative phase and that the HAT *D*₁ phase is more stable, with respect to temperature, than the HET one (Figure 7).

—the unic phase of short alkyl chain HAT is the *D*₁ phase of long alkyl chain derivatives (Figure 8). It is not very surprising to obtain a new phase *D*₀ while increasing the alkyl chain length, but it is not so classic to observe at the same time another new phase (*D*₂) at a higher temperature. The *D*₀ phase is stable only for the dodecanoate derivative (*n* = 11) while for *n* = 10 and 12 derivatives it is metastable. Several examples of binary diagrams are given in

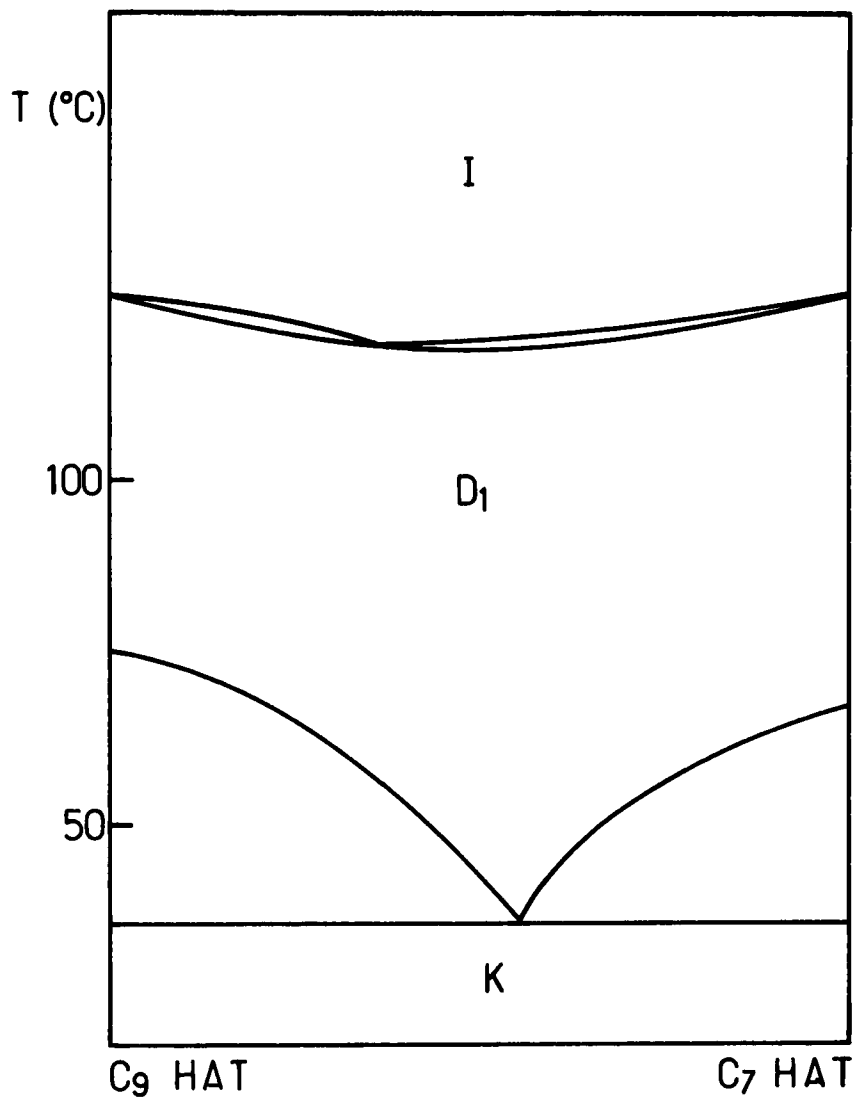


FIGURE 5 to 10 Isobar phases diagram of different binary mixtures of HET and HAT derivatives: the compounds are figured by the number of carbon atoms of the substituents.

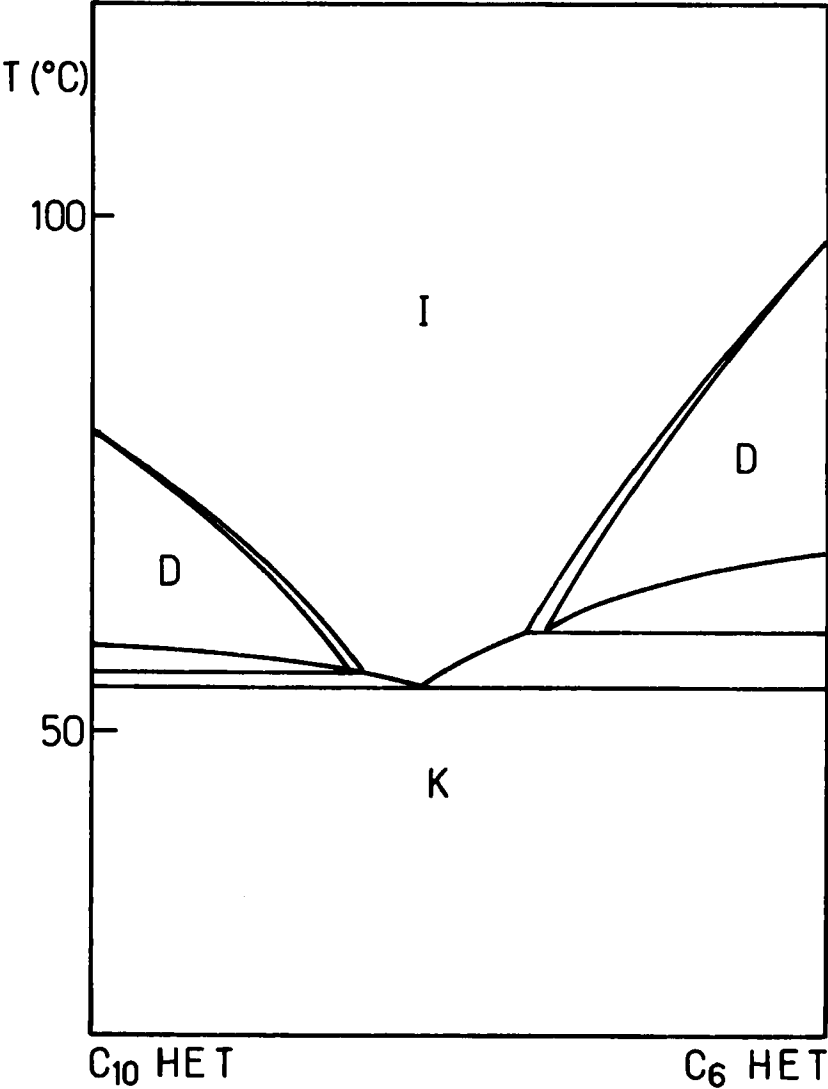


FIGURE 6

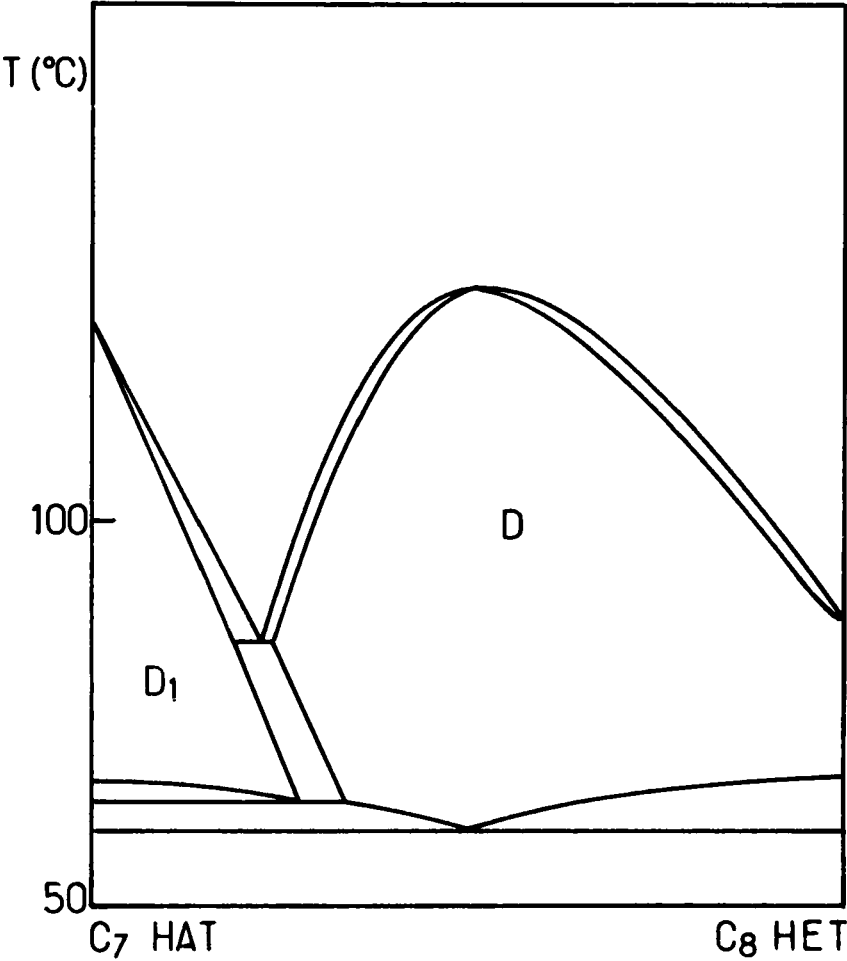


FIGURE 7

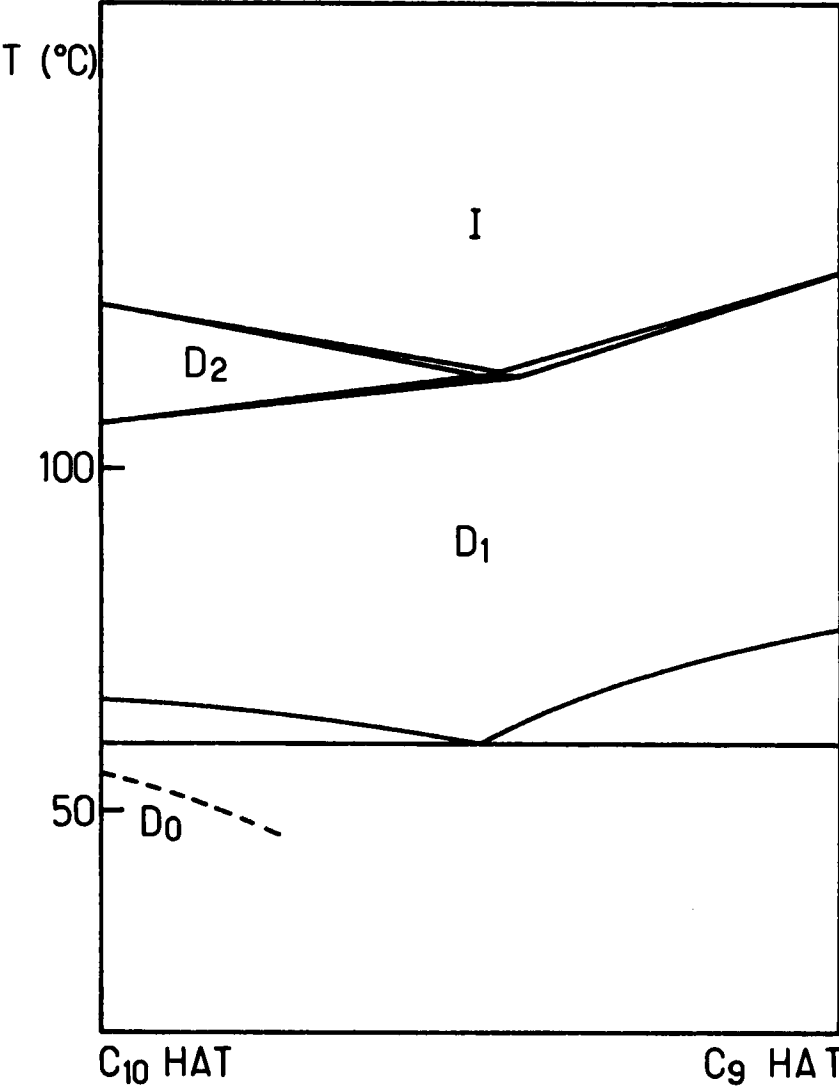


FIGURE 8

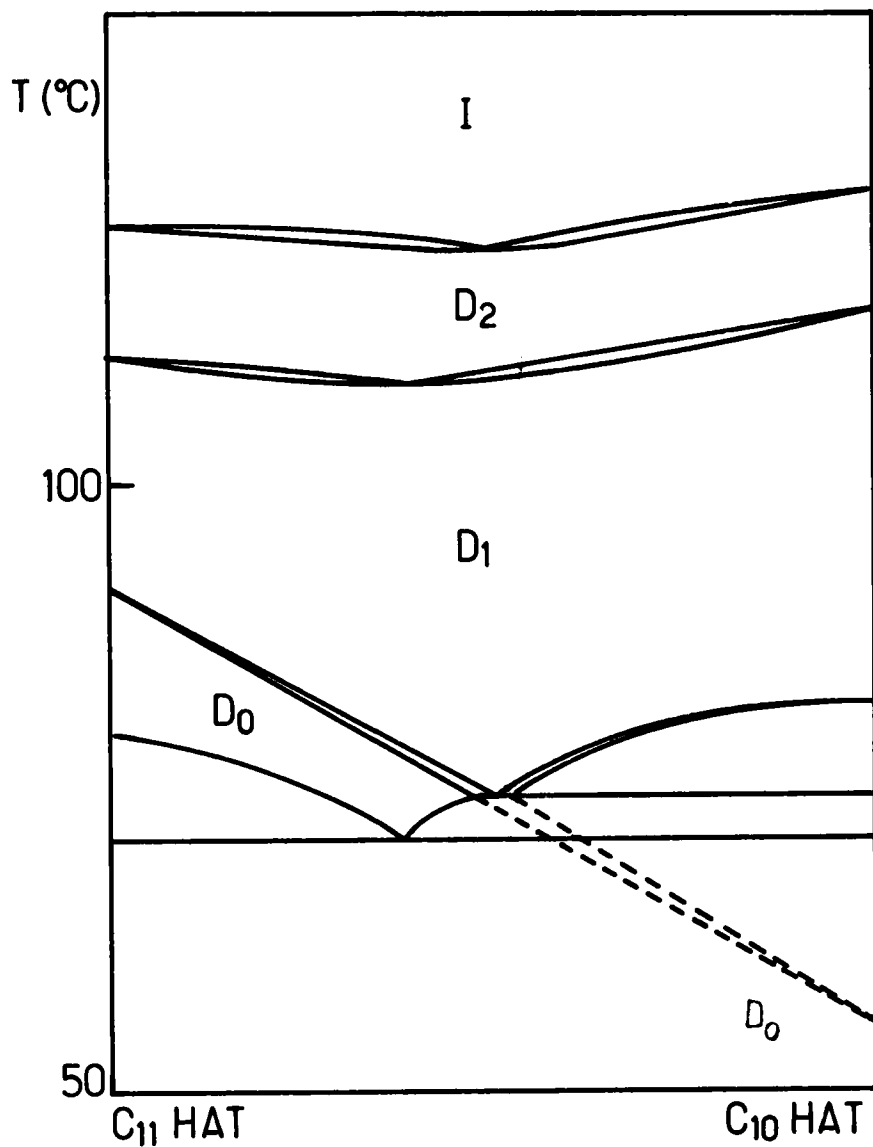


FIGURE 9

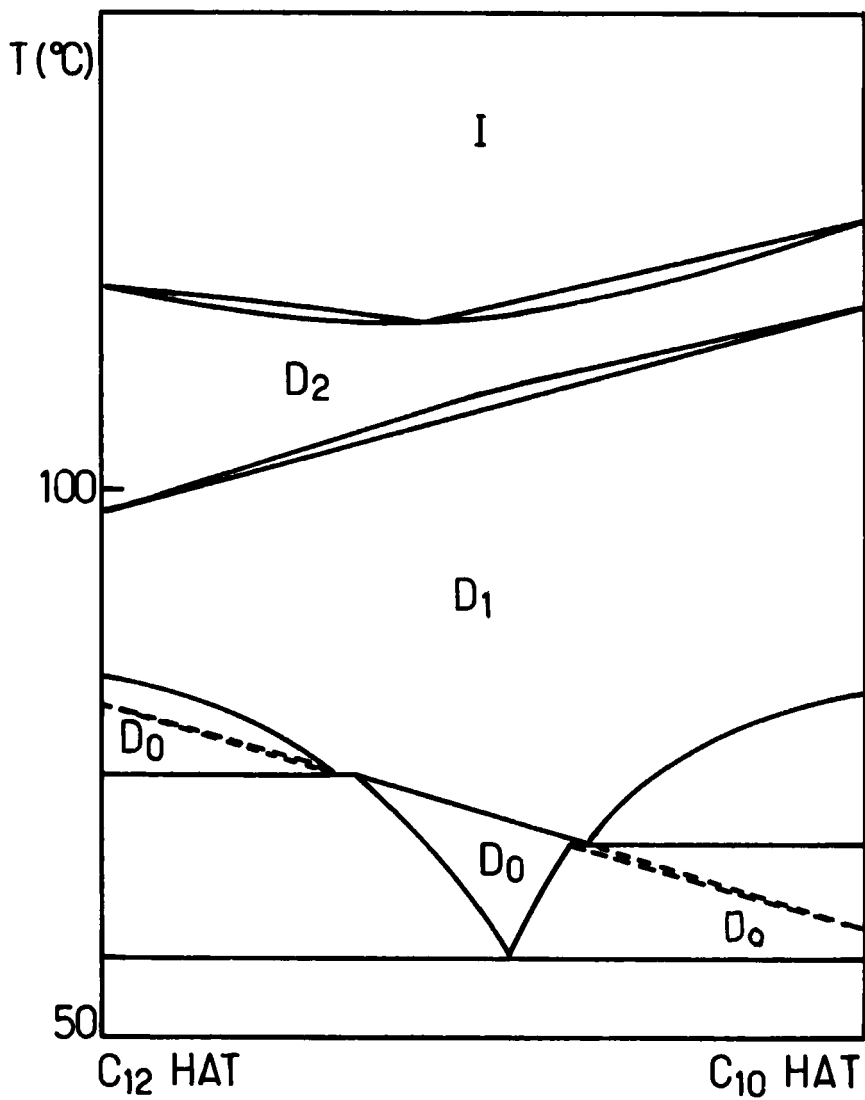


FIGURE 10

Figures 9 and 10. One can see in Figure 10 an example of stable phase obtained from two D_0 metastable ones.

—at last we must note that one of the phases of a ruffigallol derivative is totally miscible with the D_1 phase of HAT derivatives.¹³

X-RAY MEASUREMENTS

In this paper, we only report conclusions obtained from the X-Ray work. The detailed description and interpretation of the X-Ray patterns obtained on HBT and HAT have been published elsewhere.¹⁷

X-Ray measurements for all of these compounds have not been made. Nevertheless, we have studied at least one compound of each series, except for the truxene one and for the HAB studied by S. Chandrasekhar *et al.*¹

In some cases, we have obtained single domains patterns from sample held in a capillary glass tube or between two slides of mica; complementary results have been obtained by means of powder patterns obtained on a high resolution Guinier camera.

We shall say nothing about the fluid phase of the HBT (nematic disc-like phase N_D) except the fact that the feature of the X-Ray pattern are fully "liquid type; for more details see Reference 15.

The other phases are all built of columns of molecules; the columns form regular two dimensional periodic array and the different phases can differ either by the symmetry group of the lattice of the columns or by the stacking of molecules in each column. The columns are always free to slide in a direction parallel to their axis.

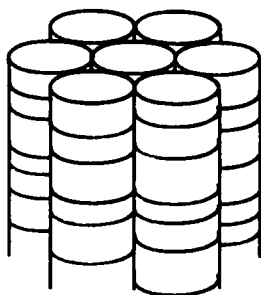
HET derivatives

The molecules are regularly stacked in the columns¹⁷ with a distance of 3.6 Å between two molecules for all the compounds studied. The lattice of the columns is hexagonal and Table 8 and Figure 12 give the lattice parameters for three examples.

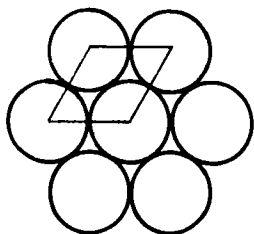
TABLE VII
Transition temperature of ruffigallol hexa-*n*-octanoate¹³

K	$R = \text{OCO C}_7\text{H}_{15}$ D_2	D_1	I
• 107.5	• [95]	• 127.5	•

hexa-*n*-alcanoyloxy benzenes



desordered discs in the
columns



hexagonal lattice

FIGURE 11 Columnar mesophase structure of HAB and HAT D_2 phase.

HAB derivatives

The same hexagonal lattice is observed for the columns¹ but the molecules are not regularly spaced in each columns.

HAT derivatives¹⁷

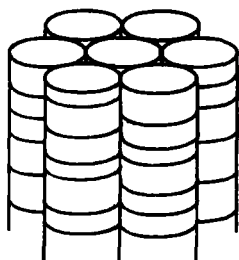
D_2 PHASE: The structure of this phase is in any point similar to the one proposed for HAB derivatives.

D_1 PHASE: The stacking of the molecules is of the same type as in D_2 phase but the hexagonal lattice is slightly distorted and we obtain a rectangular

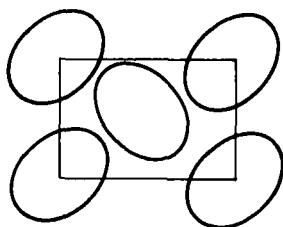
TABLE VIII
Molecular spacing in the different phases

Compounds	HAB ¹ C ₇ H ₁₅	C ₈ H ₁₁	HET C ₇ H ₁₅	C ₈ H ₁₇	C ₇ H ₁₅	HAT D ₁	C ₁₁ H ₂₃ D ₂	C ₈ H ₁₃	HBT C ₁₁ H ₂₃
Ordering of the discs inside a column	disordered		ordered			disordered			disordered
Intermolecular spacing along the column	4.6 Å		3.6 Å			4 Å			
Two dimensional lattice of the columns	hexagonal		hexagonal		rectangular pgg		hexagonal		rectangular pgg
Parameters of the two dimensional lattice	17.4 Å	19 Å	22.2 Å	23. Å	37.8 22.2 Å Å	44.9 26.4 Å Å	26.6 Å		51.8 32.6 Å Å
Inter columns distance	17.4 Å	19 Å	22.2 Å	23.4 Å	22.2-21.9 Å Å	26.4-26 Å Å	26.6 Å		

hexa-*n*-alcanoyloxy triphenylenes



desordered discs in the
columns



rectangular lattice

FIGURE 12 Columnar mesophase structure of HET derivatives.

lattice of P_{gg} symmetry with two columns per lattice unit (Figure 13). As far as the two dimensional lattice is concerned the transformation $D_1 - D_2$ is very similar to the well known transformation $S_B - S_E$ observed in liquid crystals formed of elongated molecules.

D_0 PHASE: We have not carried out an X-Ray pattern of this phase.

HBT derivatives

In the columnar phase of the hexyloxy derivative (Table 4) the molecules are irregularly stacked in each column and appear to further be tilted *versus* the column axis ($\sim 55^\circ$). From structural studies, we note the D_t phase (t = tilted). The lattice of the column is a centered rectangular lattice (Figure 14).

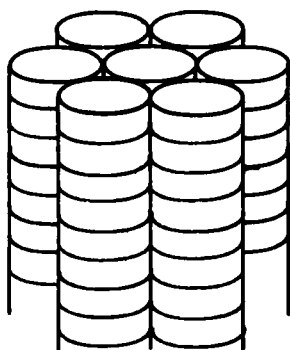
The powder pattern of the mesophase of the undecyl derivative is similar to

one of the D_1 phases of HAT derivatives. Therefore, this phase is likely constituted by columns of molecules in a rectangular two dimensional lattice (Table 8).

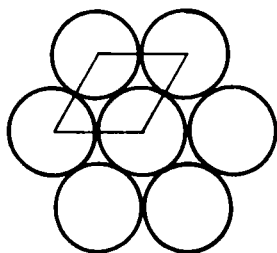
Classification

Taking into account all these results a classification can be proposed. All these phases (except the N_D one) are built up with columns of discs, we call them " D " (D for disc-like mesogens). According to the symmetry of the packing of columns we affect D with the index h (hexagonal) or r (rectangular) and eventually of another index relating the order in the column: o = ordered, d = disordered. The result of this classification is given in Table 9.

hexa- n -alkoxy triphenylenes



ordered discs in the
columns



hexagonal lattice

FIGURE 13 Columnar mesophase structure of HAT D_1 phase.

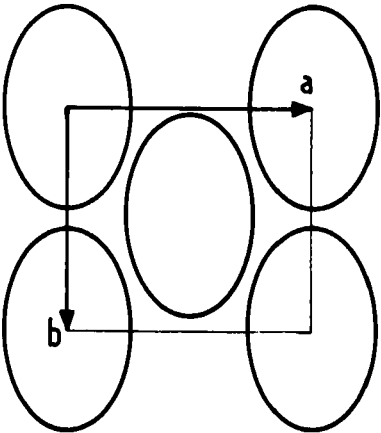
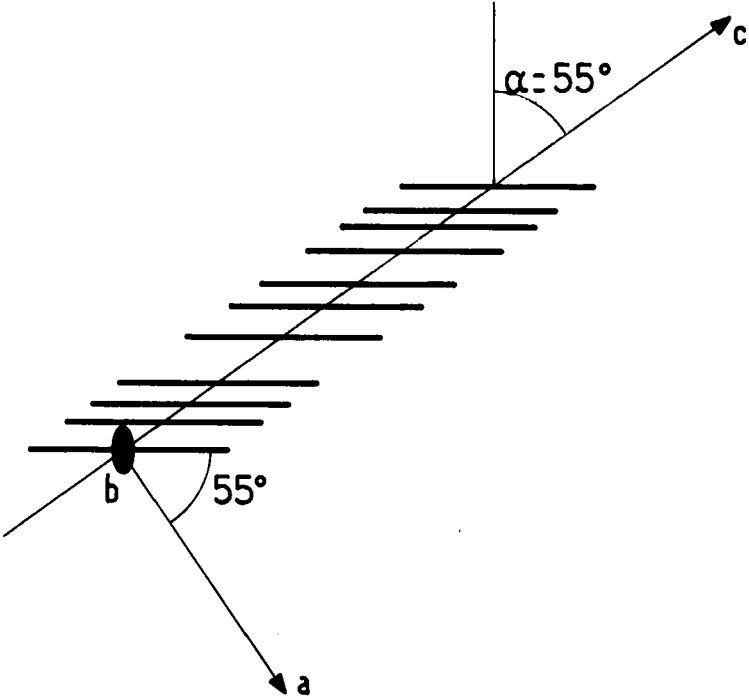


FIGURE 14 Columnar mesophase structure of HBT *D_i* phase.

TABLE IX

HAB	HET	HAT	HBT	RHO	HATX
D_{hd}	D_{ho}	D_{hd} D_{rd}	N_D D_t D_{rd}	D_{rd}	N_D D_h D_r

Remarks: the transformation $D_h - D_r$ implies a slight anisotropy of the columns in the D_r phase (Figure 13), this slight anisotropy can be obtained by an anisotropic distribution of the chain around the core of the columns; this anisotropy is not visible at all on X-Ray patterns. At first, before structural information on the phases, had been obtained, several designations were used for these phases: disc-like mesophases or "discotic" phases.³ Taking into account the existence of several columnar phases and of a nematic phase these terms may lead to confusion (*the molecules* are disc-like or discotic but not *the phases*). It is in our opinion, it is better to use the term disc-like mesogens for the molecules and N_D nematic phase or D columnar phases for the different phases (this last designation has been proposed by Hellfrich).¹⁴

Now let us consider disc-like mesogen phases in a more general point of view. For example, we can separate in the same classification columnar phases and the N_D nematic phase as smectic and nematic phases in liquid crystals built up with rod-like molecules and use for the columnar phases a presentation very similar to that for smectics. We have made such an attempt in Table 10 using the general frame of Hellfrich classification¹⁴ for "classic" liquid crystals.

TABLE X
Classification of disc-like mesogens phases with respect to temperature

	Solid Crystals	Columnar liquid crystals			Nematic disc-like liquid crystals	isotropic fluid
		disordered rectangular	ordered hexagonal	disordered		
Upright (orthogonal)	K	D_{rd}	D_{ho}	D_{hd}	$N_D \uparrow$	I
Tilted	K	D_t				
Chiral (twisted)		D_t^* (?)			$N_D^{* \uparrow \uparrow}$	

\uparrow In truxene derivative the inverse sequence is observed: K - N_D - D_r - D_h - I^{9,10}

$\uparrow \uparrow$ This last phase has been discovered very recently

DISCUSSION

All our experimental data are consistent with this classification, with yet one exception: two phases are classified D_{hd} the HAB one and the higher temperature phase of HAT derivatives and yet one cannot find any miscibility between them. It is possible to explain these facts by simple geometric considerations, see for instance in Table 8 that the columns diameter are very different in these two series (note also that C_6 HET and C_{10} HET are also not miscible), furthermore the temperature ranges are quite different (Tables 1 and 3).

The HET derivatives exhibit a mesomorphic molecular arrangement characterized by the existence of "quasi crystalline" columns, while in all the other compounds the columns are "liquid". The steric hindrance of the carboxylate group is by evidence responsible for this disorder, these groups are probably, on an average, perpendicular to the flat central triphenylene core. The crystal structure of one non-mesogenic HAT derivative has been performed ($R = C_3H_7$).¹⁸ Of course it is hazardous to transpose these results to mesogenic derivatives. Anyway interesting features have been observed: the molecular arrangement in the crystal shows an hexagonal columnar arrangement of discs; more precisely a columnar arrangement of pairs of molecules (distance between aromatic cores in a pair = 3.43 Å, distance between two pairs in a column = 17.13 Å).

CONCLUSION

Our classification is mainly based on structure consideration and our notations for the phases give immediately the molecular arrangement. One can wonder why we do not use letters like for smectics. In fact the first discovered columnar phase (D_{hd} in our notation) should be designated D_A ; the second one of the HAT derivatives should be designated D_B but of course everybody knows that a S_B phase is an hexagonal ordered arrangement of molecule while in HAT derivatives it is a rectangular disordered arrangement!

One can expect that this columnar arrangement of molecule may lead to a more complex polymorphism than smectics and therefore require a larger classification. In fact in two years about fifty compounds have been prepared and five different columnar phases founded!

We must also point out the great analogy between these phases and some lyotropic cylindric phases (inverse middle phase M_2 ¹⁹) for instance, and that such columnar phases have been described in some neat soaps at high temperature.²⁰

The existence of these new pure substances with a thermotropic meso-

morphism very close to complex lyotropic systems may induce a new desire for fundamental studies in this domain.

Lastly, recent evidence of the existence of a nematic,⁶ inverse (or reentrant⁹) and of a cholesteric phase²¹ in disc-like mesogens opens new fields of reflection (and applications?) in this new state of matter.

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