

PATENT SPECIFICATION

(11)

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- (21) Application No. 34063/76 (22) Filed 16 Aug. 1976
 (23) Complete Specification filed 24 Aug. 1977
 (44) Complete Specification published 19 Aug. 1981
 (51) INT CL³ C07C 69/773, 121/64/49/76, 63/04; C09K 3/34
 (52) Index at acceptance

C2C 220 226 227 22Y 234 238 240 266 26X 26Y 294 299 30Y
 313 316 31Y 326 339 34X 351 355 35Y 366 367 368
 37X 386 401 409 40Y 440 46Y 490 491 625 628 62Y
 665 666 668 66X 699 776 80Y 821 AA BQ BT MC
 UP

C4X 12

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(54) OPTICALLY ACTIVE LIQUID CRYSTAL MATERIALS AND LIQUID CRYSTAL DEVICES CONTAINING THEM

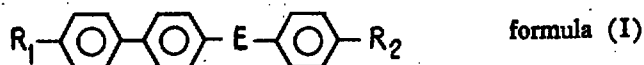
(71) I, SECRETARY OF STATE FOR DEFENCE, London, do hereby
 declare the invention, for which I pray that a patent may be granted to me, and the
 method by which it is to be performed, to be particularly described in and by the
 following statement:—

The present invention relates to optically active liquid crystal materials and liquid
 crystal devices containing them.

It is known to mix together compounds having liquid crystal properties or tenden-
 cies to produce mixtures with extended liquid crystal temperature ranges. If the
 resulting mixtures are optically active they may find use in electro-optical devices of
 the cholesteric-to-nematic phase change kind, or in applications where the colour of
 the mixture can change with temperature (thermochromic applications — e.g. surface
 thermography, temperature detection) or with the presence of an impurity (applications
 to detect unwanted particles, liquids or vapours, e.g. atmospheric pollutants).

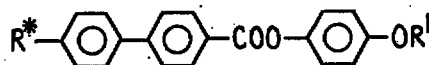
Copending United Kingdom Patent Application No. 36211/75 (UK Patent
 Specification No. 1,556,994) describes such mixtures formed by mixing 4' - alkyl - 4 -
 cyanobiphenyls with 4'' - alkyl - 4 - cyano - p - terphenyls.

In accordance with the present invention an optically active compound having
 liquid crystal properties or tendencies has a formula:



wherein E is an ester linkage group —O.CO— or —CO.O— wherein R₁ is alkyl or
 alkoxy when E is —CO.O— and R₁ is cyano when E is —O.CO— and wherein R₂
 is an alkyl, alkoxy or cyano group when E is —CO.O— and R₂ is an alkyl or alkoxy
 group when E is —O.CO—, at least one of the groups R₁ and R₂ including a chiral
 centre alkyl group, but excluding any compound as claimed in claim 1 of UK Patent
 Application No. 33860/76 Serial No. 1 592 161, and wherein R₁ and R₂ each contains
 up to 12 carbon atoms.

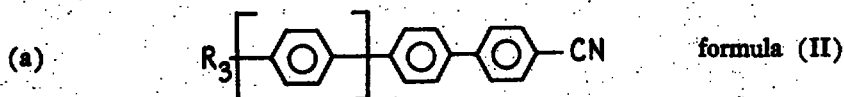
The compound of formula (I) may for example be of the form



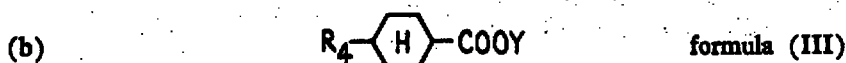
where R* is an optically active alkyl or alkoxy group and D is an n-alkyl or cyano
 group.

Preferably, the optically active alkyl group in the compound of formula (I) is CH_3 , CH_2 , $\text{CH}(\text{CH}_3)$, $(\text{CH}_2)_n$ where n is an integer in the inclusive range of 1 to 3.

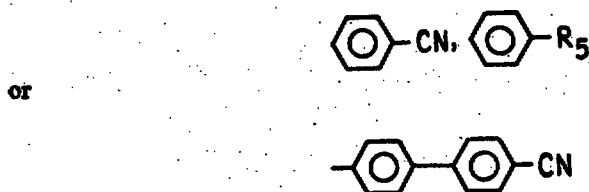
The compounds of formula (I) may be used to form thermochromic liquid crystal mixtures with one or more compounds in one or more of the categories characterised by the following formulae (I) to (V) in which the groups R_3 , R_4 , R_5 , R_6 and R_7 contain up to 12 carbon atoms:



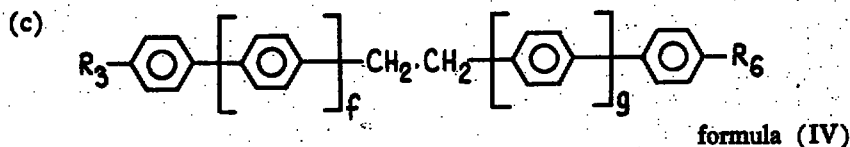
where R_3 is an alkyl group and b is 0 or 1, or R_3 is an alkoxy when b is 0;



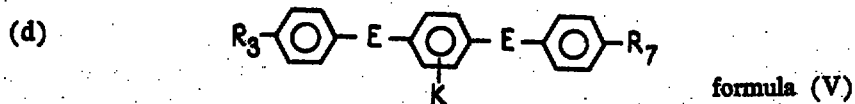
where R_4 is an alkyl group and Y is H ,



where R_5 is an alkyl or alkoxy group;



where R_3 is alkyl, R_6 is an alkyl, alkoxy or cyano group and f and g are 0 or 1 but $f+g=1$;



where R_6 is alkyl or alkoxy, R_7 is an alkyl, alkoxy or cyano group, E is an ester linkage group $-\text{COO}-$ or $-\text{OOC}-$ and K represents hydrogen or a lateral methyl or halogen substituent.

Such thermochromic mixtures may be used in known applications for optically active liquid crystal materials, the actual application depending on the specific properties of the mixture, e.g. the helical pitch of its molecular arrangement. As is well understood in the liquid crystal art the thermochromic effect is dependent upon the pitch of the helical structure adopted by the liquid crystal material being of the order of the wavelength of visible light and also being temperature dependent. As is also well understood, on admixture of a non-chiral liquid crystal material with a chiral liquid crystal material the pitch tends to increase and accordingly the pitch of chiral liquid crystal material may be adjusted by admixture with non-chiral materials in appropriate proportions, which may be determined by trial and error. The compounds

having formulae (II) to (V) may accordingly be chiral or non-chiral. Preferably the mixtures are eutectics.

According to the invention in another aspect a liquid crystal electro-optic device or thermochromic device includes as a liquid crystal material a mixture including at least one compound having formula (I) with at least one having formula (II), (III), (IV) or (V).

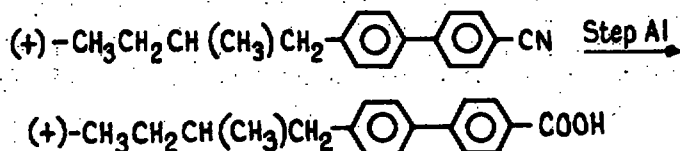
Compounds having the formulae (II) and (V) are known and their methods of preparation are also known. Certain of the compounds having formulae (III), (IV) and their preparations are described in Application No. 8 012 466 Serial No. 1 596 014 and No. 8 010 594 Serial No. 1 596 013 respectively divided from this Application.

Examples of compounds having formula (I) and methods of preparing them, which include methods of preparing intermediates used in their production, will now be given.

The optically active compounds produced as products in the following description have a positive optical rotation angle denoted by the symbol (+). Analogous compounds with negative optical rotation angles (−) may be prepared by analogous routes using the appropriate 'negative' reactants in the preparation routes.

EXAMPLE 1.

The production of (+) - 4 - (2'' - methylbutyl)biphenyl - 4' - carboxylic acid by the following route:



(+) - 4 - (2'' - methylbutyl - 4' - cyanobiphenyl is known and its production is described in Copending United Kingdom Patent Application No. 36211/75 (UK Patent Specification No. 1 556 994) and elsewhere.

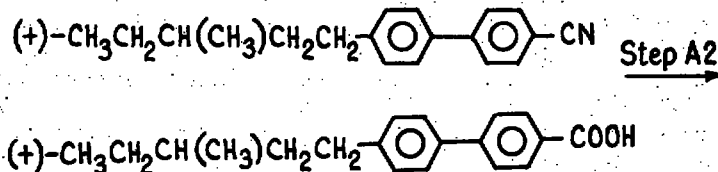
Step A1: The production of (+)-4-(2''-methylbutyl)biphenyl-4'-carboxylic acid.

One example of a way of carrying out this step is as follows:

(+) - 4 - (2'' - Methylbutyl) - 4' - cyanobiphenyl (0.015 mole) is dissolved in methanol (50ml) and is added to a mixture of potassium hydroxide (0.54 mole) and sodium hydroxide (0.75 mole) in methanol (40ml) and water (20ml). The solution is heated under reflux until evolution of ammonia ceases (approximately 72 hr). The mixture is diluted with water (1000ml) and acidified with concentrated hydrochloric acid. The precipitated product is filtered off and crystallised twice from ethanol. The product has a crystal — cholesteric liquid crystal transition temperature of 224°C; and a cholesteric liquid crystal — isotropic liquid transition temperature of 247°C.

EXAMPLE 2.

The preparation of (+) - 4 - (3'' - methylpentyl)biphenyl - 4' - carboxylic acid by the following route:



(+) - 4 - (3'' - methylpentyl - 4' - cyanobiphenyl is a known compound and its preparation is described in Copending United Kingdom Patent Application No. 36211/75 and elsewhere.

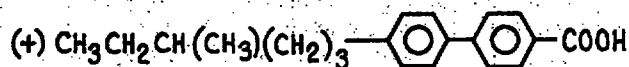
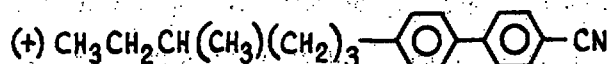
Step A2: The production of (+)-4-(3''-methylpentyl)biphenyl-4'-carboxylic acid.

One example of a way of carrying out this step is as follows:

The acid is prepared and purified by a method analogous to that in step A1 of Example 1. The colourless crystals which are produced have a crystal — cholesteric liquid crystal transition temperature of 219°C; and a cholesteric liquid crystal — isotropic liquid transition temperature of 242°C.

EXAMPLE 3.

The preparation of (+) - 4 - (4'' - methylhexyl)biphenyl - 4' - carboxylic acid by the following route:



(+) - 4 - (4'' - methylhexyl) - 4' - cyanobiphenyl is known and its preparation is described in Copending United Kingdom Patent Application No. 36211/75.

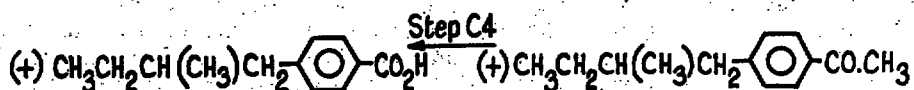
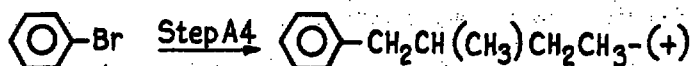
Step A3: The production of (+)-4-(4''-methylhexyl)biphenyl-4'-carboxylic acid.

One example of a way of carrying out this step is as follows:

The acid is prepared and purified by a method analogous to that in step A1 of Example 1 and has the following physical properties $C-S_0$, 175.4°C; S_0-Ch , 219.1°C; $Ch-I$, 235.8°C.

EXAMPLE 4.

The preparation of (+) - 4 - (2' - methylbutyl)benzoic acid by the following route.



Step A4: The production of (+)-2-methylbutylbenzene.

One example of a way of carrying out this step is as follows:

A solution of bromobenzene (0.51 mole) in sodium dried ether (200ml) is added in drops to magnesium turnings (0.51 mole) in sodium dried ether (50ml). A single crystal of iodine is added to initiate the reaction. When addition is complete the solution is heated under reflux for 1 hour.

The Grignard reagent is then cooled in an ice bath and ferric chloride (0.0025 mole) in ether (2ml) is added. A solution of (+)-2-methylbutyl bromide (0.54 mole) (the preparation of which is known and described in United Kingdom Patent Application No. 36211/75) in sodium dried ether (100ml) is then added over 30 mins.

The reaction mixture is then left for 48 hours (with stirring) at 25°C. After this time the mixture is poured into an ice-cold 20% hydrochloric acid solution and stirred for 30 mins. The product is extracted into ether and the combined extract is washed with water and dried (Na_2SO_4). The ether is evaporated off and the oily residue distilled. The fraction boiling at 120°C at 15 mm Hg pressure is collected.

Step B4: The production of (+)-4-(2'-methylbutyl)acetophenone.

One example of a way of carrying out this step is as follows:

Crushed anhydrous aluminium trichloride (0.295 mole) is suspended in carbon disulphide (80ml). Acetyl chloride (0.25 mole) and 2-methylbutylbenzene, prepared in Step A4, are dissolved in carbon disulphide (80ml) and added to the aluminium trichloride suspension under anhydrous conditions; the mixture is left to stand overnight (with stirring).

The solvent is distilled from the reaction mixture and the viscous residue poured onto crushed ice and stirred for 30 min. The product is extracted into ether, washed with water and dried (Na_2SO_4). The ether is removed by rotary evaporation and the oily residue is distilled. The product boils at 95°C at a pressure of 0.1 mm Hg.

Step C4: The production of (+)-4-(2'-methylbutyl)benzoic acid.

One example of a way of carrying out this step is as follows:

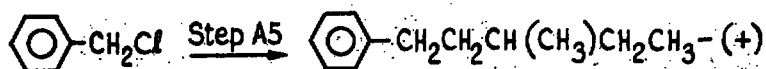
A solution of sodium hypobromite, prepared by dissolving bromine (156g) in a solution of sodium hydroxide (3.5 mole) in water (700ml) at 0°C , is added to a well stirred solution of (+)-4-(2'-methylbutyl)acetophenone (0.2 mole) prepared in step B4, in dioxan (500ml). Throughout the addition, and for 15 minutes after, the reaction temperature is maintained at $35-40^\circ\text{C}$.

Excess of hypobromite is destroyed by adding a solution of sodium metabisulphite. Water (3.5 litre) and bromoform are distilled from the reaction mixture.

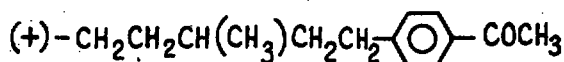
On cooling, the solution is acidified with concentrated hydrochloric acid and the benzoic acid precipitated is filtered off and washed with water. The product is crystallised from ethanol/water. The melting point of the product is 130°C .

EXAMPLE 5.

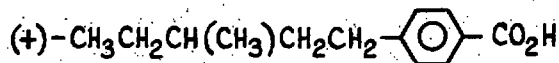
The preparation of (+)-4-(3'-methylpentyl)benzoic acid by the following route:



↓ Step B5



↓ Step C5



Step A5: The production of (+)-3-methylpentylbenzene.

One example of a way of carrying out this step is as follows:

The Grignard reagent of commercially available benzyl chloride is prepared and alkylated in an analogous manner to step A4 of Example 4.

Step B5: The production of (+)-4-(3'-methylpentyl)acetophenone.

One example of a way of carrying out this step is as follows:

The alkylbenzene prepared in step A5 is acylated and purified in an analogous manner to step B4 of Example 4.

Step C5: The production of (+)-4-(3'-methylpentyl)benzoic acid.

One example of a way of carrying out this step is as follows:

The acetophenone prepared in step B5 is oxidised to the acid product (melting point 121°C) by an analogous manner to step C4 of Example 4.

EXAMPLE 6.

The preparation of (+)-trans-4-alkylcyclohexane-1-carboxylic acids by the following route:

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Step A6: The production of (+)-trans-4-alkylcyclohexane-1-carboxylic acids.

Ways of carrying out this step are exemplified by the preparation of (+) - trans-4 - (2' - methylbutyl)cyclohexane - 1 - carboxylic acid as follows:

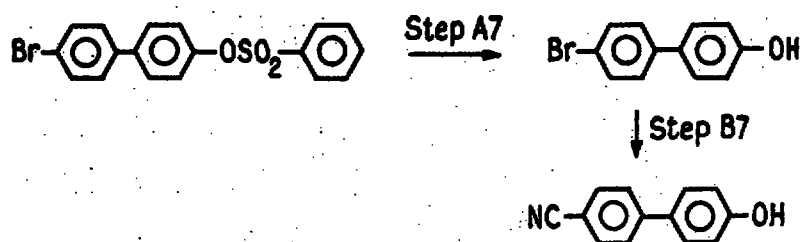
A solution of (+) - 4 - (2' - methylbutyl)benzoic acid (0.2 mole) in sodium hydroxide (6.205 mole) dissolved in water (160ml) is hydrogenated in the presence of Raney Nickel catalyst (10g) in an autoclave (1 litre at 195°C and a pressure of 170 atm of hydrogen for 30 hr.

On cooling, the catalyst is filtered off and the filtrate washed with ether. The aqueous layer is acidified and the precipitated acids are extracted into ether, washed with water and dried (Na_2SO_4). The ether is distilled off and the acids are taken up in methanol (200ml); the solution is treated successively with 40g and 30g of thiourea. After each treatment with thiourea, the crystallisate is filtered from the methanol. The combined crystallisates are dissolved in a 5% potassium hydroxide solution (800ml) which is then acidified and the (+) - trans - 4 - (2' - methylbutyl)cyclohexane - 1 - carboxylic acid precipitated is extracted into ether; the extract is washed with water and dried (Na_2SO_4).

The ether is evaporated off and the acid recrystallised from acetone. The melting point of the product is 50.3°C.

EXAMPLE 7.

Preparation of 4' - cyano - 4 - hydroxybiphenyl by the following route:



Step A7: The production of 4'-bromo-4-hydroxybiphenyl.

One example of a way of carrying out this step is by a standard method using sodium hydroxide to hydrolyse commercially available 4' - bromo - 4 - benzenesulphonyloxybiphenyl in a mixture of water and dioxan as solvent. The colourless crystals obtained by crystallisation of the product from ethanol having a melting point of 166°C.

Step B7: The production of 4'-cyano-4-hydroxybiphenyl.

One example of a way of carrying out this step is as follows:

A mixture of 4' - bromo - 4 - hydroxybiphenyl (6g), N - methyl - 2 - pyrrolidone (23ml) and copper (I) cyanide (2.5g) is heated under reflux and vigorously stirred for 2 hr. The mixture is protected from atmospheric moisture using CaCl_2 guard tubes. After cooling, the mixture is poured into a warm (60°C) solution of iron (III) chloride in water (200ml) and concentrated hydrochloric acid (8ml) and stirred (20 min). The cooled mixture is shaken twice with ether ($2 \times 100\text{ml}$) and the combined organic extracts are water washed. Rotary evaporation of the solvent gives a pale brown solid which is crystallised from a mixture of water and ethanol. This yields pale tan needles of melting point 198°C.

EXAMPLE 8.

The preparation of 4-alkylphenols — where the alkyl group may or may not be chiral.

These are prepared by standard methods such as those described by Van der Veen, de Jeu, Grobben and Boven (*Mol. Cryst. Liq. Cryst.*, 1972, 17, 291) for the

preparation of 4-alkylanilines, followed by diazotisation of the amines and hydrolysis of the diazonium sulphates.

EXAMPLE 9.

Preparation of 4-alkoxyphenols — where the alkyl chain may or may not be chiral.

These are prepared by the mono-alkylation of *p*-quinol using the method of Neubert, Carlino, D'Sidocky and Fishel (Liquid Crystals and Ordered Fluids, Vol. 2 (Edited by J. F. Johnson and R. S. Porter). Plenus Press, N.Y., 1973 p. 303). The colourless products are purified by column chromatography and crystallisation.

EXAMPLE 10.

4-cyanophenol is a commercially available material.

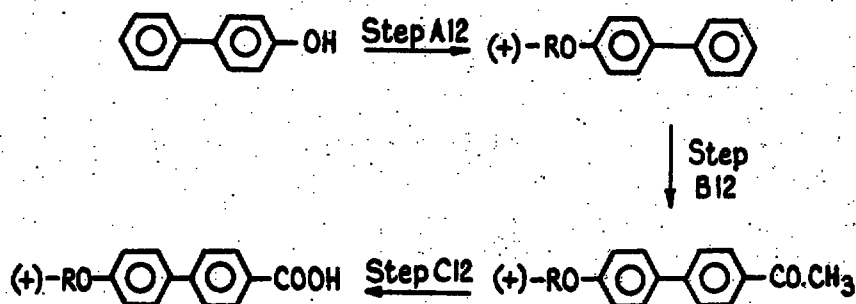
EXAMPLE 11.

Preparation of (+)-4-alkoxybenzoic acids.

One way of preparing these materials is by a standard method of alkylating hydroxy aromatic carboxylic acids (Gray and Brynmor Jones, *J. Chem. Soc.*, 1954, 678), in this case 4-hydroxybenzoic acid.

EXAMPLE 12.

Preparation of (+) - 4 - alkoxybiphenyl - 4' - carboxylic acids by the following route:



Step A12: The production (+)-4-alkoxybiphenyl.

One example of a way of carrying out this step is described by Coates and Gray, *J. Chem. Soc., Perkin II*, 1976, 863.

Step B12: The production (+)-4-alkoxy-4'-acetyl biphenyl.

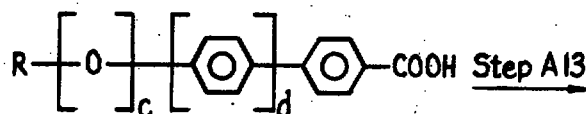
One example of a way of carrying out this step is by a Friedel-Crafts acylation as described in step A15 of Example 15 below.

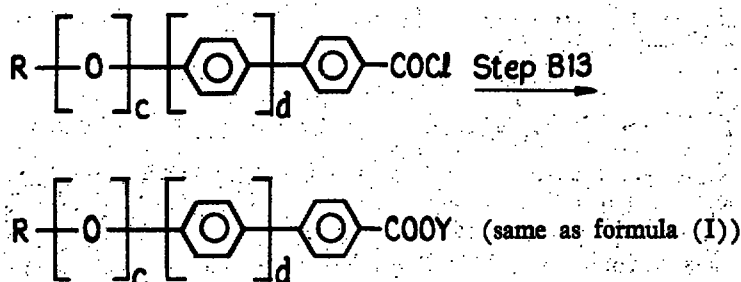
Step C12: The production of (+)-4-alkoxybiphenyl-4'-carboxylic acids.

One example of a way of carrying out this step is by a haloform oxidation reaction analogous to that described in step C4 of Example 4 above.

EXAMPLE 13.

The preparation of ester compounds of formula (I) defined above by the following route:





Step A13: The production of the optically active acid chloride.

The carrying out of this step is exemplified by the preparation of (+) - 4 - (2''-methylbutyl)biphenyl - 14' - carboxylic acid chloride as follows:

(+) - 4 - (2'' - Methylbutyl)biphenyl - 4' - carboxylic acid (0.002 mole) prepared in step A1 of Example 1, is heated with thionyl chloride (20ml) under anhydrous conditions for 1.5 hr. The excess of thionyl chloride is removed by distillation under reduced pressure. The acid chloride residue is used in the next step without further purification.

Step B13: The production of the ester.

The carrying out of this step is exemplified by the production of 4'' - n - pentylphenyl (+) - 4 - (2'' - methylbutyl)biphenyl - 4' - carboxylate.

(+) - 4 - (2'' - Methylbutyl)biphenyl - 4' - carboxylic acid chloride (0.002 mole), prepared in step A13, is dissolved in dry 'AnalaR' (Trade Mark) pyridine (10ml) and cooled in an ice bath. 4 - n - Pentylphenol (0.002 mole), prepared as in Example 8, is added to the stirred solution and the mixture is left, with stirring, for 18 hr to rise to room temperature. It is then heated at 100°C for 1 hr. The pyridine is removed by rotary evaporation and the residue column chromatographed on silica gel, eluting with chloroform. The combined fractions of ester are crystallised from hexane.

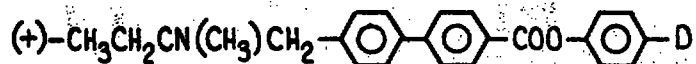
Analogous products prepared by steps A13 and B13 involve the use of the analogous intermediate products produced by the preceding Examples.

The constants measured for some Examples of the esters of Group II are given in the Tables 1—8 below, in which the following symbols have the stated meaning: C=crystal; S_A=Smectic A; Ch=cholesteric; I=isotropic liquid; →=transition temperature; ΔH=enthalpy of fusion in K cal mol⁻¹ and the temperatures are in °C; for monotropic transitions, the temperatures are in parenthesis.

It should be noted that the chiral S_C and Ch phases of any single compound always have the same sense (right or left-handed) of pitch. However, compounds having the value of n odd in the grouping CH₂CH₂CH(CH₃)(CH₂)_n— with an absolute configuration which is S, have right handed helical pitch senses whereas those with even values of n have left-handed helical pitch senses for the S_C and Ch phases.

(17)

TABLE 1



D = alkyl or cyano

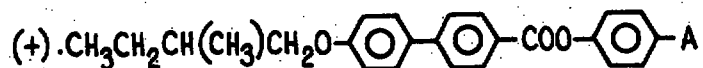
D	C → Ch (°C)	Ch → I (°C)	ΔH
-CN	99.5	195.9	7.1
-C ₂ H ₅	81.3	141.1	5.7
n-C ₃ H ₇	81.5	151.3	5.6
n-C ₄ H ₉	65.6	127.5	4.4
n-C ₈ H ₁₁	63.6	138.2	4.1
n-C ₈ H ₁₃	60.3	132.3	6.5

TABLE 2



n	C → ch °C	Ch → I °C	ΔH k cal mol ⁻¹
1	96.5	210	5.2
2	99.2	205	4.5

TABLE 3



A	C - S _C °C	S _B - S _C °C	S _C - S _A °C	S _A - Ch °C	Ch - I °C	Pitch μm
C ₆ H ₁₃ -n	72	(67.8)	85.5	145	158	1.2
OC ₈ H ₁₇ -n	95	—	136.2	152.5	170	1.2

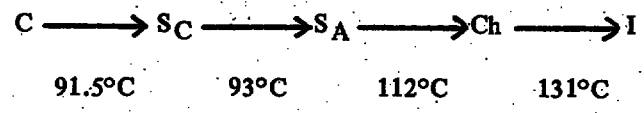
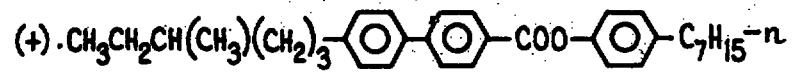
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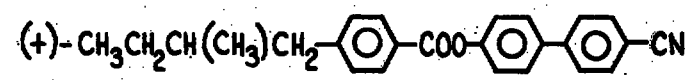
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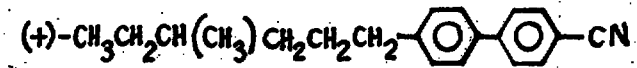
TABLE 4



Mixture I
20% by weight



5 and
80% by weight

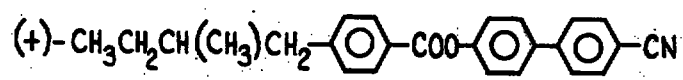


This mixture has a smectic A to cholesteric transition at -10°C and a cholesteric to isotropic liquid transition at $+19^\circ\text{C}$.

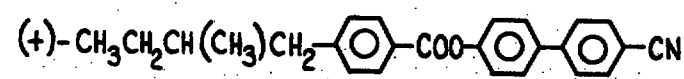
Mixture I is thermochromic giving the following selective colour reflections (in the Grandjean plane texture).

Selective Reflection	Temperature ($^\circ\text{C}$)
Red	-10.0
Green	-9.0
Turquoise	-8.0
Blue	-7.0

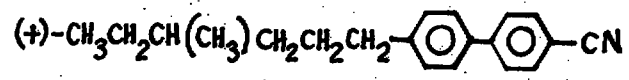
Mixture II
12% by weight



20 8% by weight



80% by weight



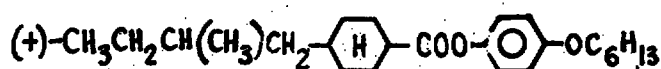
This mixture has a Smetic A to cholesteric transition at -10°C and a cholesteric to isotropic liquid transition at $+19^\circ\text{C}$.

Mixture II is thermochromic and the selective colour reflected by this mixture in the Grandjean plane texture is as follows:

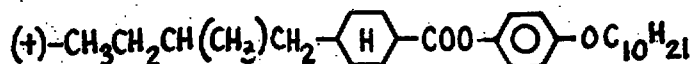
	Selective Reflection	Temperature (°C)	
	Red	-10.0	
	Orange	- 9.5	
5	Yellow	- 8.5	
	Green	- 7.0	5
	Turquoise	- 3.0	
	Blue	+16.0	

Mixture III demonstrates the use of racemic materials ($\pm$) in widening colour ranges in respect of temperature.

10 *Mixture IV* 42.5% by weight 10



28.0% by weight



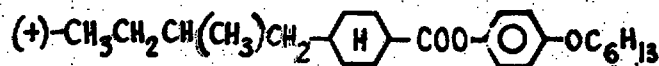
15 29.5% by weight 15



S₀-Ch; 20°C Ch-I 57.4°C.

	Selective Reflection	Temperature (°C)	
20	Infra-red	20	20
	Red	28	
	Yellow	34	
	Green	36	
	Turquoise	45	
25	Blue	47	25
	Isotropic liquid	57.4	

Mixture V
37.5% by weight



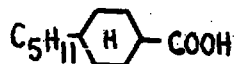
62.5% by weight

30 $(+)\text{-CH}_3\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2\text{-}\langle\text{O}\rangle\text{-}\langle\text{O}\rangle\text{-COO-}\langle\text{O}\rangle\text{-C}_6\text{H}_{13}$ 30

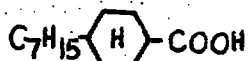
S₀-CH, 27°C; Ch-I, 86°C.

	Selective Reflection	Temperature (°C)	
	Red	27.0	
35	Yellow	29.5	35
	Green	31.0	
	Turquoise	37.0	
	Blue	39.0	
	Isotropic	86.0	

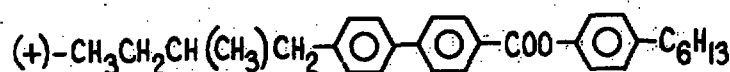
Mixture VI
14% by weight



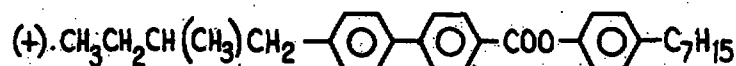
17% by weight



63% by weight



6% by weight



$S_A-\text{Ch}$, 22°C; $\text{Ch}-I$, 107.8°C.

Selective Reflection

Temperature (°C)

Red	27
Yellow	45
Green	54
Turquoise	97
Blue	105
Isotropic	107.8

Examples of the use of mixtures as hereinbefore defined, e.g. in electro-optic phase change devices (when mixed with nematic materials) and in thermochromic devices, are described in copending United Kingdom Patent Application No. 36211/75 (UK Specification No. 1 556 994).

Having regard to the provisions of Section 9 of the Patents Act 1949, attention is directed to the Claims of Patent No. 1 510 360 and Patent No. 1 456 359.

WHAT I CLAIM IS:—

1. An optically active compound having formula (I) given hereinbefore but excluding any compound as claimed in claim 1 of UK Patent Application No. 33860/76 Serial No. 1 592 161.

2. A compound as claimed in claim 1 and which has a positive optical rotation angle (+) and a formula



wherein R^* is an optically active alkyl or alkoxy group and D is an n-alkyl or cyano group.

3. A compound as claimed in claim 2 and wherein R^* is 2-methylbutyl and D is cyano.

4. A compound as claimed in claim 2 and wherein R^* is 2-methylbutyl and D is C_2H_5 .

5. A compound as claimed in claim 2 and wherein R^* is 2-methylbutyl and D is $n\text{-C}_8\text{H}_{17}$.

6. A compound as claimed in claim 2 and wherein R^* is 2-methylbutyl and D is $n\text{-C}_4\text{H}_9$.



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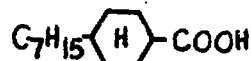
12

Mixture VI
14% by weight



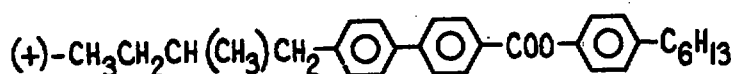
17% by weight

5



5

63% by weight



6% by weight



10

S_A—Ch, 22°C; Ch—I, 107.8°C.

10

Selective Reflection

Temperature (°C)

Red

27

Yellow

45

Green

54

Turquoise

97

Blue

105

Isotropic

107.8

15

15

Examples of the use of mixtures as hereinbefore defined, e.g. in electro-optic phase change devices (when mixed with nematic materials) and in thermochromic devices, are described in copending United Kingdom Patent Application No. 36211/75 (UK Specification No. 1 556 994).

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WHAT I CLAIM IS:—

25

25

1. An optically active compound having formula (I) given hereinbefore but excluding any compound as claimed in claim 1 of UK Patent Application No. 33860/76 Serial No. 1 592 161.

2. A compound as claimed in claim 1 and which has a positive optical rotation angle (+) and a formula



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wherein R* is an optically active alkyl or alkoxy group and D is an n-alkyl or cyano group.

3. A compound as claimed in claim 2 and wherein R* is 2-methylbutyl and D is cyano.

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4. A compound as claimed in claim 2, and wherein R* is 2-methylbutyl and D is C₂H₅.

5. A compound as claimed in claim 2 and wherein R* is 2-methylbutyl and D is n-C₃H₇.

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6. A compound as claimed in claim 2 and wherein R* is 2-methylbutyl and D is n-C₄H₉.



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7. A compound as claimed in claim 2 and wherein R* is 2-methylbutyl and D is n-C₆H₁₁.

8. A compound as claimed in claim 2 and wherein R* is 2-methylbutyl and D is n-C₆H₁₃.

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9. A compound as claimed in claim 2 wherein R* is 4-methylhexane and D is n-heptyl.

5

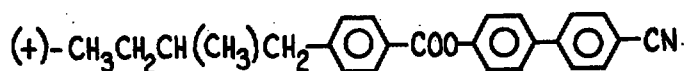
10. A compound as claimed in claim 2 and wherein R* is 2-methylbutyloxy and D is n-hexyl.

10

11. A compound as claimed in claim 2 and wherein R* is 2-methylbutyloxy and D is n-octyloxy.

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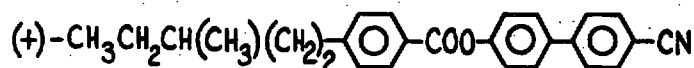
12. A compound as claimed in claim 1 and which has a positive optical rotation angle (+) and a formula:



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13. A compound as claimed in claim 1 and which has a positive optical rotation angle (+) and a formula:

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14. A liquid crystal material comprising a mixture of a compound as claimed in claim 1 together with one or more compounds selected from formulae (II), (III), (IV) or (V) given hereinbefore.

15. A liquid crystal electro-optic device or thermochromic device including as a liquid crystal material as claimed in claim 14.

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16. A liquid crystal mixture as claimed in claim 14 and substantially the same as any one of the specific mixtures described hereinbefore.

J. RUSSELL,
Chartered Patent Agent,
Agent for the Applicant.