

IN THE MATTER OF an Application
for a European Patent
in the name of Merck Patent Gesell-
schaft mit beschränkter Haftung
granted under No. 0014840 , and
IN THE MATTER OF an Application
for a Singapore Registration Patent.

I, WOLFGANG GERSON BARB, B.Sc., Ph.D., F.P.I., a Fellow
of the Institute of Linguists, of "Spring Wood", Hedgerley
Lane, Gerrards Cross, Buckinghamshire, do solemnly and
sincerely declare that I am conversant with the English
and German languages and am a competent translator thereof,
and that the following is a true and correct translation
of the European Patent
No. 0014840 granted to Merck Patent Gesellschaft mit Beschränkter
Haftung

and the Official Certificate attached thereto.

Declared at Europa House,
Marsham Way, Gerrards Cross,
in the County of Buckinghamshire,
this 30th day of March 1983

W. G. Barb

W. G. Barb

35

- (19) European Patent Office
European Patent Office
European Patent Office

(11) Publication No.: 0014840
B1

(12) EUROPEAN PATENT SPECIFICATION

(45) Date of publication of the
patent specification:
06.01.82

(51) Int. Cl.³: C07C49/567, C07C25/18,
C07C45/46, C07C17/00,
C09K3/34

(21) Application No: 80100194.2

(22) Date of application: 16.01.80

(54) Fluorophenyl-trans-cyclohexanes, a process for their preparation, liquid
crystal dielectrics containing them, and an electrooptical display element.

(30) Priority:
24.02.79 DE2907332

(73) Patent proprietor: Merck Patent Gesell-
schaft mit beschränkter Haftung,
Frankfurter Strasse 250, D-6100 Darm-
stadt (DE)

(43) Date of publication of the
application:
03.09.80 Patent Journal 80/18

(45) Publication of the note that
the patent has been granted:
06.01.82 Patent Journal 82/1

(72) Inventor: Dr. Rudolf Eidenschink,
Dessauer Strasse 57, D-6110 Dieburg
(DE)

(84) Designated convention countries:
AT CH DE FR GB IT NL

Inventor: Dr. Ludwig Pohl, Hoffmann-
strasse 54, D-6100 Darmstadt (DE)

56 Citations:
DE-A-2 636 684
1977 SID INTERNATIONAL SYMPOSIUM
DIGEST OF TECHNICAL PAPERS, first
edition, April 1977, Page 77,
Publisher: Lewis Winner, New York,
U.S.A.

ANGEWANDTE CHEMIE, INTERNATIONAL EDI-
TION IN ENGLISH, Volume 16, February
77 Page 100 Weinheim, DE.R.EIDENSCHINK
et al.: "Substituted Phenylcyclohexanes -
A New Class of Liquid-Crystalline Compounds"

Note: Anyone is entitled to lodge an opposition to the granted European patent
at the European Patent Office, within nine months of publication of the note, in
the European Patent Journal, that the European patent has been granted. The
opposition must be filed in writing and must give grounds. It will only be
regarded as lodged when the opposition fee has been paid (Article 99 (1) of the
European Patent Convention).

FEDERAL PRINTING OFFICE BERLIN

EP 0 014 840 B1

Fluorophenyl-trans-cyclohexanes, a process for their preparation, liquid crystal dielectrics containing them, and an electrooptical display element.

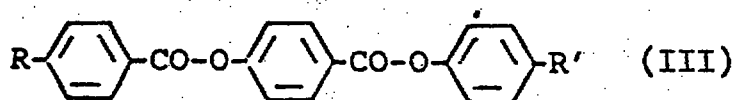
For liquid crystal display elements, the properties of nematic or nematic-cholesteric liquid crystal materials are utilised to effect a significant change in their optical properties, such as light transmission, light scattering, double refraction, reflectance or colour, under the influence of electrical fields. The function of such display elements is based, for example, on the phenomenon of dynamic scattering, the deformation of oriented phases, the Schadt-Helfrich effect in the twisted cell or the nematic-cholesteric phase transition.

For industrial application of these effects in liquid crystal display elements, liquid crystal materials are needed which have to meet a large number of requirements. Particularly important requirements are the chemical stability towards moisture, air and physical influences, such as heat, radiation in the infrared, visible and ultraviolet range and steady and alternating electrical fields. Furthermore, a liquid crystal mesophase in the temperature range from at least 0°C to $+50^{\circ}\text{C}$, and preferably from -10°C to $+70^{\circ}\text{C}$, and a viscosity at room temperature of not more than 60 cP are demanded for liquid crystal materials which can be used industrially. Finally, these materials must not have any characteristic absorption in the range of visible light, that is to say they must be colourless.

A number of liquid crystal compounds which meet the stability requirements demanded of dielectrics for

electronic components and which are also colourless are already known. These include, in particular, the p,p'-disubstituted phenyl benzoates described in German Offenlegungsschrift 2,139,628, the p,p'-disubstituted biphenyl derivatives described in German Offenlegungsschrift 2,356,085 or the phenylcyclohexane derivatives described in German Offenlegungsschrift 2,636,684.

In the said categories of compounds and also in other known series of compounds having a liquid crystal mesophase, there are no individual compounds which form a liquid crystal nematic mesophase in the required temperature range from 0°C to 60°C. Therefore, as a rule, mixtures of two or more compounds are prepared in order to obtain substances which can be used as liquid crystal dielectrics. For this purpose, usually at least one compound having a low melting point and clear point is mixed with another compound having a significantly higher melting point and clear point. This usually gives a mixture which has a melting point at a temperature lower than or about the same as the melting point of the component having the lower melting point, whilst the clear point is between the clear points of the components. Components having higher melting points and clear points which have been used hitherto are, for example, 4,4"-disubstituted phenyl p-benzoyloxybenzoate of the formula (III)



- 3 -

or the biphenyl ester derivatives of the formula (IV)

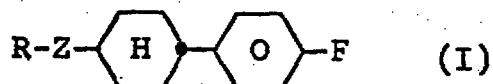


wherein R and R' are alkyl or alkoxy groups and X is a carbonyloxy group. The compounds of the formula (III)

5 have not, however, found a wide field of application because they impart a high viscosity to the liquid crystal dielectrics containing them; as a result, the switching times of the liquid crystal display elements produced with these compounds are prolonged in an undesirable
10 manner. It is true that this effect is not so pronounced when the biphenyl esters of the formula (IV) are used, but these compounds, in particular at low temperatures, are not as readily soluble in the most important liquid crystal base materials as is desirable if the
15 clear point is to be raised.

The invention is thus based on the object of providing liquid crystal dielectrics which have a wide temperature range for the nematic phase, which includes room temperature, and which have as low as possible a
20 viscosity.

This object is achieved, according to the invention, by a process in which one or more fluorophenyl-trans-cyclohexanes of the formula (I)



25 wherein R is an alkyl group with 1-17 carbon atoms and Z is either a carbonyl group or a methylene group, are admixed, as components which lower the viscosity, to

- 4 -

known liquid crystal substances with a high viscosity.

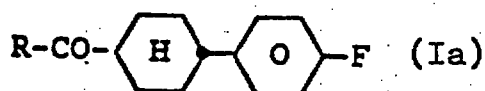
The fluorophenylcyclohexanes of the formula (I) as a rule have monotropic liquid crystal properties, that is to say their clear point is at a lower temperature than their melting point. Their nematic phase can thus be observed only in a supercooled melt, so that they cannot be used as dielectrics for liquid crystal display elements without other mixing components. However, they are very readily soluble in known liquid crystal mixtures and lower the viscosity of such substances to the extent that with the dielectrics resulting therefrom, liquid crystal display elements with short switching times which could scarcely be achieved hitherto can be realised.

The invention thus relates to the fluorophenyl-trans-cyclohexanes of the formula (I) and to processes for their preparation. The invention furthermore relates to the use of the fluorophenylcyclohexanes of the formula (I), mixed with other liquid crystal substances, as dielectrics in liquid crystal display elements.

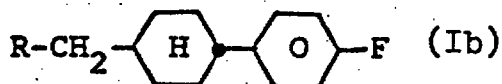
The invention also relates to dielectrics, for liquid crystal display elements, with at least two liquid crystal components, of which at least one is a fluorophenylcyclohexane of the formula (I). Finally, the invention relates to electrooptical display elements based on a liquid crystal cell, in which the liquid crystal cell contains a dielectric containing a fluorophenylcyclohexane of the formula (I).

The fluorophenyl-trans-cyclohexanes of the formula (I) are either ketones of the formula (Ia)

- 5 -



wherein R has the meaning indicated in formula (I), or
4-(trans-4-alkylcyclohexyl)-fluorobenzenes of the formula (Ib)



5 wherein R has the meaning indicated in formula (I). In
all the fluorophenylcyclohexanes of the general formulae
(I), (Ia) and (Ib) according to the invention, the parti-
cular substituents in the 1-positions and 4-positions of
the cyclohexane rings are arranged in the trans configuration;
10 in the formulae, this is indicated by the black marking
on the right-hand side of the cyclohexane rings.

As a rule, the ketones of the formula (Ia) have a
lower dielectric anisotropy than the 4-(4-alkylcyclohexyl)-
fluorobenzenes of the formula (Ib). The ketones (Ia)
15 are thus preferably used in dielectrics for liquid crys-
tal display elements which operate on the basis of the
phenomena of dynamic scattering or of deformation of
oriented phases. The compounds of the formula (Ib) are
correspondingly preferably used in dielectrics with a
20 positive dielectric anisotropy, which are employed in
display elements based on the twisted nematic cell.
However, if technical criteria, such as those of the
dielectric anisotropy or also, for example, of the solu-
bility, play a minor role in selecting the compounds
25 according to the invention, compared with economic view-
points, the ketones (Ia) are preferred, since they can be
prepared more simply than the compounds of the formula (Ib).

- 6 -

The substituent R in the compounds of the formula (I) can be straight-chain or branched. If R is straight-chain, that is to say methyl, ethyl, n-propyl, n-butyl, n-pentyl, n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl or n-heptadecyl, the compounds of the formula (I) characterised by this definition as a rule have higher clear points; furthermore, the starting materials for their synthesis are more readily accessible than the starting materials for compounds with a branched substituent R. These compounds, including those in which R is straight-chain alkyl with 1 to 12 carbon atoms, preferably 3 to 10 carbon atoms, are thus particularly preferred in the context of this invention. However, in some cases compounds of the formula (I) with a branched substituent R are also important, since these sometimes have better solubility properties in the customary liquid crystal base mixtures. Such substituents R which are not straight-chain preferably contain not more than one chain branching. Preferred branched substituents are those in which the carbon chain is branched at the carbon atom adjacent to the group Z or at one of the next two carbon atoms. Amongst these substituents, those branched groups in which a methyl or ethyl group is in the 1-, 2- or 3-position on a longer carbon chain are of importance, examples being isopropyl, 1-methylpropyl, 2-methylpropyl, 1-methylbutyl, 2-methylbutyl, 3-methylbutyl, 1-methylpentyl, 1-ethylpentyl, 2-methylpentyl, 1-methylhexyl, 2-ethylhexyl or 1-methylheptyl.

- 7 -

The 4-(4-alkylcyclohexyl)-fluorobenzenes of the formula (Ib) according to the invention can be prepared analogously to the 4-alkyl-1-phenyl-cyclohexanes described in German Offenlegungsschrift 2,636,684. However, the
5 fluorophenylcyclohexanes according to the invention are preferably prepared by a process in which cyclohexene and fluorobenzene are reacted with a carboxylic acid halide of the formula (II)



10 wherein R has the abovementioned meaning and Hal is chlorine or bromine, in the presence of a Lewis acid and if appropriate in the presence of an inert solvent, at a temperature between -50° and $+100^{\circ}$, and, in the compound of the formula (Ia) initially obtained, the carbonyl group is
15 optionally reduced to a methylene group in a manner which is in itself known.

Preferred possible carboxylic acid halides of the formula (II) are the chlorides, which can be prepared from the corresponding fatty acids in a simple manner, for
20 example by reaction with thionyl chloride.

The Lewis acids preferably used in the process according to the invention are aluminium chloride, boron trifluoride, zinc chloride, iron trichloride or antimony pentachloride. Aluminium chloride is preferred for
25 economic reasons.

In principle, all the customary solvents for Friedel-Crafts reactions can be used as solvents for the process according to the invention. Carbon tetra-
chloride, methylene chloride, 1,2-dichloroethane, carbon

- 8 -

disulphide, petroleum ether, n-hexane or cyclohexane are preferably employed. However, the reaction can also be carried out in the absence of an additional solvent.

In this case, it has proved preferable to use an excess
5 of fluorobenzene.

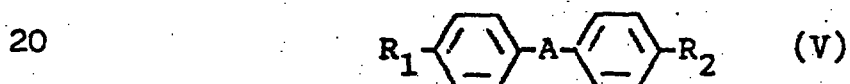
The reaction can be carried out in the temperature range from -50° to $+100^{\circ}$. Advantageously, a lower temperature, preferably between -30° and $+10^{\circ}$, is chosen at the start of the reaction and the reaction mixture is allowed
10 to warm up to room temperature towards the end of the reaction; in some cases it is favourable to warm the mixture, up to the boiling point of the solvent used, for a short time at the end of the reaction in order to bring the reaction to completion. The mixture is worked up in a
15 manner which is in itself customary, preferably by hydrolysis with aqueous hydrochloric acid at temperatures between -10° and $+20^{\circ}$, extraction of the ketone formed, of the formula (Ia), with a suitable organic solvent, for example toluene, and fractional distillation of the
20 extract under reduced pressure. The ketones of the formula (Ia) thus prepared can be used as components of liquid crystal dielectrics without further purification; if purification is desired, the appropriate distillate fractions are preferably recrystallised from methanol or
25 ethanol.

The reduction of the ketones (Ia) to the corresponding 4-(4-alkylcyclohexyl)-fluorobenzenes (Ib) is effected in a manner which is in itself known, for example with hydrazine hydrate and an alkali metal hydroxide or

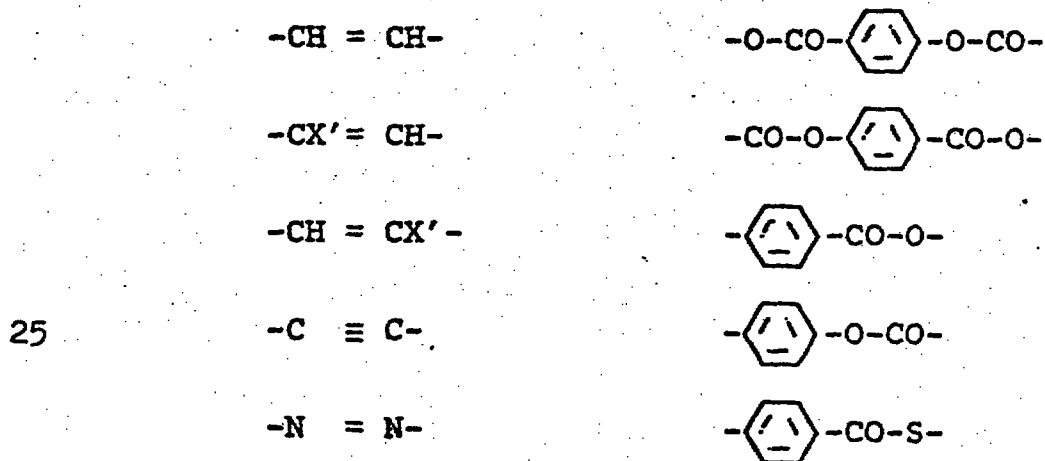
- 9 -

alcoholate in a high-boiling solvent, such as diethylene glycol or dimethylsulphoxide, with zinc and hydrogen chloride in diethyl ether, or with zinc amalgam in aqueous hydrochloric acid. After working up which is in itself customary, the compounds of the formula (Ib) thus obtained are purified by fractional distillation under reduced pressure or by recrystallisation from a suitable solvent, for example ethanol or ethyl acetate.

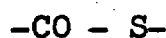
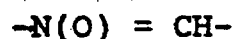
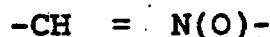
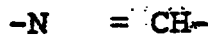
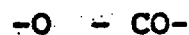
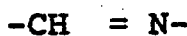
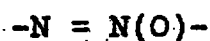
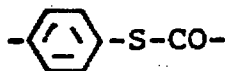
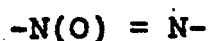
The dielectrics according to the invention consist of two or more components, including at least one of the formula (I). The other components are preferably nematic or nematogenic substances from the categories of azobenzenes, azoxybenzenes, biphenyls, Schiff's bases, in particular benzylidene derivatives, phenyl benzoates, phenylcyclohexanes, optionally halogenated stilbenes, diphenylacetylene derivatives, diphenylnitrones and substituted cinnamic acids. The most important compounds which can be used as other components of this type are characterised by the formula (V):



wherein A is



- 10 -



or a C-C single bond;

if A is $-\text{CO}-\text{O}-$, $-\text{O}-\text{CO}-$ or a C-C single bond, one of the two phenyl rings can also be replaced by a trans-cyclohexyl ring; X' is halogen, preferably Cl. R_1 and R_2 are identical or different and can be alkyl, alkoxy, alkanoyl, alkanoyloxy or alkoxy-carbonyloxy radicals with up to 18, preferably up to 8, C atoms; furthermore, one of these radicals can also be a cyano, nitro or isonitrile group. In most of these compounds, R_1 and R_2 are preferably different, and one of the radicals is usually an alkyl or alkoxy group. However, a large number of other variants of the envisaged substituents are also customary. Many such nematic substances are commercially available.

The liquid crystal dielectrics according to the invention as a rule contain at least 1 and at most 35 parts by weight of one or more fluorophenylcyclohexanes of the formula (I). Those materials which contain 3 to 30, and preferably 5 to 20, parts by weight of at least one fluorophenylcyclohexane of the formula (I) are preferred. The viscosity of the liquid crystal base materials is lowered by an average of 10 - 50 % by these

- 11 -

additives. The switching times of liquid crystal display elements produced with the dielectrics according to the invention are correspondingly shortened. The clear points of the dielectrics according to the invention are indeed lower than those of the liquid crystal base materials. However, by suitable choice of the base materials and of the amount of fluorophenylcyclohexane(s) of the formula (I) added, mixtures are obtained which have clear points which are not achieved even under the warming which occurs during continuous operation of the display elements produced with these mixtures. If the addition of one or more fluorophenylcyclohexanes of the formula (I) to a liquid crystal base material produces a mixture with a clear point which is too low for industrial application, this effect can be compensated, if appropriate, by adding 1-30, and preferably 2-20, parts by weight of one or more biphenyl esters of the formula (IV), of one or more hexahydroterphenyls according to German Offenlegungsschrift 2,701,591 or of one or more of the cyclohexane derivatives according to German Offenlegungsschrift 2,800,553.

By means of suitable additives, the liquid crystal dielectrics according to the invention can be modified in such a way that they can be used in all types of display elements disclosed hitherto. Such additives are known to those skilled in the art and are described in detail in the relevant literature. For example, substances for changing the dielectric anisotropy and/or the orientation of the nematic phases can be added. Such sub-

stances are described, for example, in German Offenlegungsschriften 2,209,127, 2,321,632 and 2,611,453.

The examples which follow are intended to illustrate the invention without restricting it. In the
5 examples, m. designates the melting point and Cl designates the clear point of a liquid crystal substance in degrees Centigrade; boiling points are designated b.p.. Unless stated otherwise, parts or percentage data are parts by weight or per cent by weight respectively.

10 Example 1

A mixture of 82 g of cyclohexene and 132 g of n-heptanoyl chloride is added dropwise to a suspension of 160 g of aluminium chloride in 192 g of fluorobenzene in the course of 2 hours, whilst stirring and cooling to
15 -10° . The reaction mixture is subsequently stirred at $+20^{\circ}$ for a further 16 hours and then poured into a mixture of 300 g of ice and 300 ml of fuming hydrochloric acid. The organic phase is separated off, the aqueous phase is extracted twice more, with 200 ml of toluene
20 each time, and the combined organic phases are dried over calcium chloride and distilled. After distilling off the excess fluorobenzene and the toluene, the residue is distilled under reduced pressure (5-10 mm Hg). A first fraction of about 10 % of the residue is discarded as
25 first runnings; the subsequent 4-(trans-4-n-heptanoyl-cyclohexyl)-fluorobenzene distilled off, which makes up about 65 % of the residue, is taken up in the same amount by volume of methanol and the product is crystallised at -20° ; yield 73 g, m. 47° , Cl- 10° .

- 13 -

The following compounds are obtained analogously:

- 4-(trans-4-acetylcyclohexyl)-fluorobenzene,
4-(trans-4-propionylcyclohexyl)-fluorobenzene, m. 53° , Cl- 20°
4-(trans-4-n-butyrylcyclohexyl)-fluorobenzene,
5 4-(trans-4-isobutyrylcyclohexyl)-fluorobenzene,
4-(trans-4-n-pentanoylcyclohexyl)-fluorobenzene, m. 35° , Cl- 30°
4-(trans-4-(2-methylbutyryl)-cyclohexyl)-fluorobenzene,
4-(trans-4-(3-methylbutyryl)-cyclohexyl)-fluorobenzene,
4-(trans-4-n-hexanoylcyclohexyl)-fluorobenzene,
10 4-(trans-4-(2-methylpentanoyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-(3-methylpentanoyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-(2-methylhexanoyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-(2-ethylpentanoyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-n-octanoylcyclohexyl)-fluorobenzene,
15 4-(trans-4-n-nonanoylcyclohexyl)-fluorobenzene, m. 54° , Cl- 10°
4-(trans-4-n-(3-ethylheptanoyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-n-decanoylcyclohexyl)-fluorobenzene, m. 58° , Cl- 20°
4-(trans-4-n-undecanoylcyclohexyl)-fluorobenzene,
4-(trans-4-n-dodecanoylcyclohexyl)-fluorobenzene,
20 4-(trans-4-n-tridecanoylcyclohexyl)-fluorobenzene,
4-(trans-4-n-tetradecanoylcyclohexyl)-fluorobenzene, m. 60° , Cl- 5°
4-(trans-4-n-pentadecanoylcyclohexyl)-fluorobenzene,
4-(trans-4-n-hexadecanoylcyclohexyl)-fluorobenzene,
4-(trans-4-n-heptadecanoylcyclohexyl)-fluorobenzene and
25 4-(trans-4-n-octadecanoylcyclohexyl)-fluorobenzene.

Example 2

A solution of 58 g of 4-(trans-4-n-heptanoylcyclohexyl)-fluorobenzene, 45 g of potassium hydroxide and 30 g of hydrazine hydrate in 200 ml of diethylene glycol

- 14 -

is warmed from 100° to 195° in the course of 3 hours.

After cooling the reaction mixture, 300 ml of water are added and the mixture is extracted with 200 ml of methylene chloride. The solvent and, as an azeotrope with the solvent, the residual moisture are distilled off from the extract and the 4-(trans-4-n-heptylcyclohexyl)-fluorobenzene which remains is recrystallised from ethyl acetate; yield 47 g, m. 35°, Cl-15°.

The following compounds are obtained analogously:

- 10 4-(trans-4-ethylcyclohexyl)-fluorobenzene,
4-(trans-4-n-propylcyclohexyl)-fluorobenzene, m. 32°, Cl-40°
4-(trans-4-n-butylcyclohexyl)-fluorobenzene,
4-(trans-4-isobutylcyclohexyl)-fluorobenzene,
4-(trans-4-n-pentylcyclohexyl)-fluorobenzene, m. 36°, Cl-25°
- 15 4-(trans-4-(2-methylbutyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-(3-methylbutyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-n-hexylcyclohexyl)-fluorobenzene,
4-(trans-4-(2-methylphenyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-(3-methylpentyl)-cyclohexyl)-fluorobenzene,
- 20 4-(trans-4-(2-methylhexyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-(2-ethylpentyl)-cyclohexyl)-fluorobenzene,
4-(trans-4-n-octylcyclohexyl)-fluorobenzene,
4-(trans-4-n-nonylcyclohexyl)-fluorobenzene, m. 39°, Cl-5°
4-(trans-4-(3-ethylheptyl)-cyclohexyl)-fluorobenzene,
- 25 4-(trans-4-n-decylcyclohexyl)-fluorobenzene, m. 46°, Cl-5°
4-(trans-4-n-undecylcyclohexyl)-fluorobenzene,
4-(trans-4-n-dodecylcyclohexyl)-fluorobenzene,
4-(trans-4-n-tridecylcyclohexyl)-fluorobenzene,
4-(trans-4-n-tetradecylcyclohexyl)-fluorobenzene, m. 57°, Cl. 15°

50

- 15 -

4-(trans-4-n-pentadecylcyclohexyl)-fluorobenzene,
4-(trans-4-n-hexadecylcyclohexyl)-fluorobenzene,
4-(trans-4-n-heptadecylcyclohexyl)-fluorobenzene and
4-(trans-4-n-octadecylcyclohexyl)-fluorobenzene.

5 The examples which follow relate to liquid crystal dielectrics according to the invention:

Example 3

A mixture of 27 % of 4-(trans-4-n-propylcyclohexyl)-benzonitrile, 39 % of 4-(trans-4-n-pentylcyclohexyl)-
10 benzonitrile, 13 % of 4-(trans-4-n-pentylcyclohexyl)-4'-cyanobiphenyl and 21 % of 4-(trans-4-n-nonylcyclohexyl)-fluorobenzene has a nematic phase in the temperature range from -10° to $+55^{\circ}$. The dielectric anisotropy is +10.2 and the viscosity is 20 cSt. The corresponding
15 dielectric which contains no fluorophenylcyclohexane according to the invention but is otherwise identical in composition has a viscosity of 32 cSt.

The dielectric according to the invention is outstandingly suitable for use in display elements based on
20 the twisted nematic cell.

Example 4

A mixture of 24 % of 4-(trans-4-n-butylcyclohexyl)-benzonitrile, 16.5 % of 4-ethyl-4'-cyanobiphenyl, 12.0 % of 4-n-propoxy-4'-cyanobiphenyl, 17.5 % of 4-
25 (trans-4-n-pentylcyclohexyl)-4'-cyanobiphenyl, 7 % of 4'-cyanophenyl 4-(trans-4-n-pentylcyclohexyl)-benzoate and 23 % of 4-(trans-4-n-heptylcyclohexyl)-fluorobenzene has a nematic phase in the temperature range from -6° to $+61^{\circ}$. The dielectric anisotropy is +12.5 and the

- 16 -

viscosity is 32 cSt. The corresponding dielectric which contains no fluorophenylcyclohexane according to the invention but is otherwise identical in composition has a viscosity of 40 cSt.

- 5 The dielectric according to the invention is very suitable for display units based on the twisted nematic cell, and in particular also in the form of matrix displays.

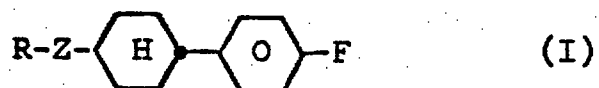
Example 5

- 10 A mixture of 47 % of 4-(trans-4-n-propylcyclohexyl)-1-n-butyryloxybenzene, 19 % of 4-n-propylcyclohexyl 4-(trans-4-n-butylcyclohexyl)-benzoate, 14 % of 4-n-propylcyclohexyl 4-(trans-4-ethylcyclohexyl)-benzoate and 20 % of 4-(trans-4-propionylcyclohexyl)-fluorobenzene
- 15 has a nematic phase in the temperature range from -5° to $+65^{\circ}$. The dielectric anisotropy is -0.2 and the viscosity is 30 cSt. The corresponding dielectric which contains no fluorophenylcyclohexane according to the invention but is otherwise identical in composition has a
- 20 viscosity of 36 cSt.

This dielectric according to the invention is very particularly suitable for display elements which operate on the basis of dynamic scattering.

Patent Claims for the contracting states CH, DE, FR, GB, IT and NL

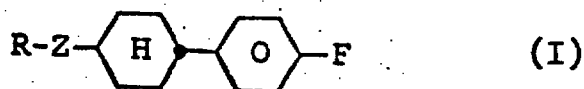
1. Fluorophenyl-trans-cyclohexanes of the formula (I)



wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group.

2. Fluorophenylcyclohexanes according to Claim 1, characterised in that R is a straight-chain alkyl group.

3. Process for the preparation of fluorophenyl-trans-cyclohexanes of the formula (I)



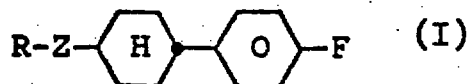
wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, characterised in that cyclohexene and fluorobenzene are reacted with a carboxylic acid halide of the formula (II)



wherein R has the abovementioned meaning and Hal is chlorine or bromine, in the presence of a Lewis acid and if appropriate in the presence of an inert solvent, at a temperature between -50° and $+100^{\circ}$, and, in the initially obtained compound of the formula (I) wherein Z denotes a carbonyl group, this group is optionally reduced to a methylene group in a manner which is in itself known.

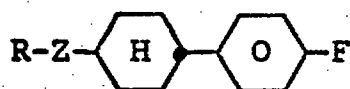
4. Use of a fluorophenyl-trans-cyclohexane of the formula (I)

- 2 -



wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, as a component of a liquid crystal dielectric for electro-optical display elements.

5. Liquid crystal dielectric with at least two components, characterised in that it contains at least one fluorophenyl-trans-cyclohexane of the formula (I)

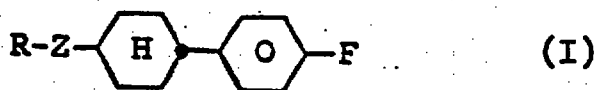


wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group.

6. Electrooptical display element based on a liquid crystal cell, characterised in that the liquid crystal cell contains a dielectric according to Claim 5.

Patent Claims for the contracting state AT

1. Process for the preparation of fluorophenyl-trans-cyclohexanes of the formula (I)



wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, characterised in that cyclohexene and fluorobenzene are reacted with a carboxylic acid halide of the formula (II)

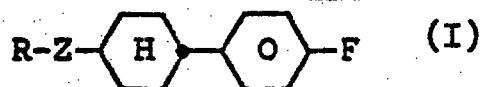


wherein R has the abovementioned meaning and Hal is chlorine or bromine, in the presence of a Lewis acid and if appropriate in the presence of an inert solvent, at a

- 3 -

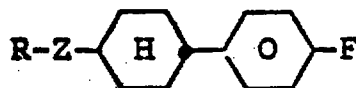
temperature between -50° and $+100^{\circ}$, and, in the initially obtained compound of the formula (I) wherein Z denotes a carbonyl group, this group is optionally reduced to a methylene group in a manner which is in itself known.

2. Use of a fluorophenyl-trans-cyclohexane of the formula (I)



wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, as a component of a liquid crystal dielectric for electro-optical display elements.

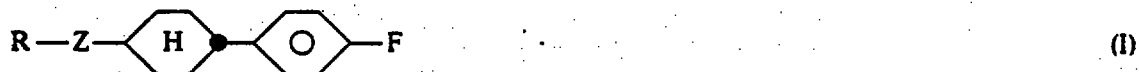
3. Liquid crystal dielectric with at least two components, characterised in that it contains at least one fluorophenyl-trans-cyclohexane of the formula (I)



wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group.

4. Electrooptical display element based on a liquid crystal cell, characterised in that the liquid crystal cell contains a dielectric according to Claim 3.

(Translation of German text)

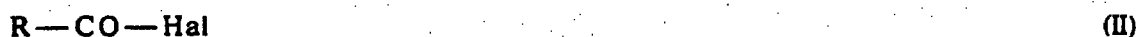
Claims for the Contracting States CH, DE, FR, GB, IT, NL (As printed)**1. Fluorophenyl-trans-cyclohexanes of the formula (I)**

wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group.

2. Fluorophenylcyclohexanes according to Claim 1, characterised in that R is a straight-chain alkyl group.

3. Process for the preparation of fluorophenyl-trans-cyclohexanes of the formula (I)

wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, characterised in that cyclohexene and fluorobenzene are reacted with a carboxylic acid halide of the formula (II)



wherein R has the abovementioned meaning and Hal is chlorine or bromine, in the presence of a Lewis acid and if appropriate in the presence of an inert solvent, at a temperature between -50° and $+100^\circ$, and, in the initially obtained compound of the formula (I) wherein Z denotes a carbonyl group, this group is optionally reduced to a methylene group in a manner which is in itself known.

4. Use of a fluorophenyl-trans-cyclohexane of the formula (I)

wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, as a component of a liquid crystal dielectric for electrooptical display elements.

5. Liquid crystal dielectric with at least two components, characterised in that it contains at least one fluorophenyl-trans-cyclohexane of the formula (I)



wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group.

6. Electrooptical display element based on a liquid crystal cell, characterised in that the liquid crystal cell contains a dielectric according to Claim 5.

Claims for the Contracting State AT (As printed)**1. Process for the preparation of fluorophenyl-trans-cyclohexanes of the formula (I)**

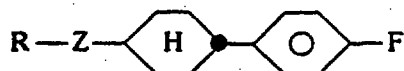
wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, characterised in that cyclohexene and fluorobenzene are reacted with a carboxylic acid halide of the formula (II)



(II)

wherein R has the abovementioned meaning and Hal is chlorine or bromine, in the presence of a Lewis acid and if appropriate in the presence of an inert solvent, at a temperature between -50° and $+100^{\circ}$, and, in the initially obtained compound of the formula (I) wherein Z denotes a carbonyl group, this group is optionally reduced to a methylene group in a manner which is in itself known.

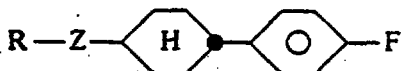
2. Use of a fluorophenyl-trans-cyclohexane of the formula (I)



(I)

wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group, as a component of a liquid crystal dielectric for electrooptical display elements.

3. Liquid crystal dielectric with at least two components, characterised in that it contains at least one fluorophenyl-trans-cyclohexane of the formula (I)

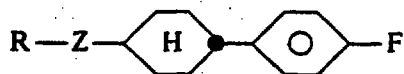


wherein R is an alkyl group with 1 to 17 carbon atoms and Z is either a carbonyl group or a methylene group.

4. Electrooptical display element based on a liquid crystal cell, characterised in that the liquid crystal cell contains a dielectric according to Claim 3.

Revendications pour les Etats contractants CH, DE, FR, GB, IT, NL (As printed)

1. Fluorophényl-trans-cyclohexanes de formule I



(I)

dans laquelle R représente un groupe alkyle en C1—C17 et Z représente un groupe carbonyle ou un groupe méthylène.

2. Fluorophénylcyclohexanes selon la revendication 1, caractérisés en ce que R représente un groupe alkyle à chaîne droite.

3. Procédé de préparation des fluorophényl-trans-cyclohexanes de formule I



(I)

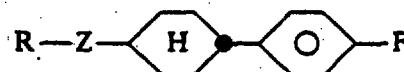
dans laquelle R représente un groupe alkyle en C1—C17 et Z représente un groupe carbonyle ou un groupe méthylène, caractérisé en ce que l'on fait réagir le cyclohexène et le fluorobenzène en présence d'un acide de Lewis avec un halogénure d'acide carboxylique de formule II



(II)

dans laquelle R a la signification indiquée ci-dessus et Hal représente le chlore ou le brome, éventuellement en présence d'un solvant inerte, à une température allant de -50° à $+100^{\circ}$ C et le cas échéant, dans le composé de formule I obtenu en premier et dans lequel Z représente un groupe carbonyle, on réduit ce dernier, de manière connue en soi, en un groupe méthylène.

4. Utilisation d'un fluorophényl-trans-cyclohexane de formule I



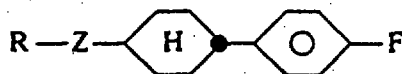
(I)

dans laquelle R représente un groupe alkyle en C1—C17 et Z représente un groupe carbonyle ou un groupe méthylène, en tant que composant d'un diélectrique à cristaux liquides pour éléments d'affichage électro-optiques.

57

- 6 -

5. Diélectrique à cristaux liquides à au moins deux composants, caractérisé en ce qu'il contient au moins un fluorophényl-trans-cyclohexane de formule

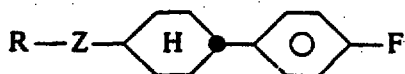


dans laquelle R représente un groupe alkyle en C1—C17 et Z représente un groupe carbonyle ou un groupe méthylène.

6. Élément d'affichage électro-optique à base d'une cellule à cristaux liquides, caractérisé en ce que la cellule à cristaux liquides contient un diélectrique selon la revendication 5.

Revendications pour l'Etat contractant AT (As printed)

1. Procédé de préparation des fluorophényl-trans-cyclohexanes de formule I



(I)

dans laquelle R représente un groupe alkyle en C1—C17 et Z représente un groupe carbonyle ou un groupe méthylène, caractérisé en ce que l'on fait réagir le cyclohexène et le fluorobenzène en présence d'un acide de Lewis avec un halogénure d'acide carboxylique de formule II



(II)

dans laquelle R a la signification indiquée ci-dessus et Hal représente le chlore ou le brome, éventuellement en présence d'un solvant inerte, à une température allant de -50 à $+100^\circ\text{C}$ et le cas échéant, dans le composé de formule I obtenu en premier et dans lequel Z représente un groupe carbonyle, on réduit ce dernier, de manière connue en soi, en un groupe méthylène.

2. Utilisation d'un fluorophényl-trans-cyclohexane de formule I



(I)

dans laquelle R représente un groupe alkyle en C1—C17 et Z représente un groupe carbonyle ou un groupe méthylène, en tant que composant d'un diélectrique à cristaux liquides pour éléments d'affichage électro-optiques.

3. Diélectrique à cristaux liquides à au moins deux composants, caractérisé en ce qu'il contient au moins un fluorophényl-trans-cyclohexane de formule



dans laquelle R représente un groupe alkyle en C1—C17 et Z représente un groupe carbonyle ou un groupe méthylène.

4. Élément d'affichage électro-optique à base d'une cellule à cristaux liquides, caractérisé en ce que la cellule à cristaux liquides contient un diélectrique selon la revendication 3.