

PATENT SPECIFICATION

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(54) HEXAHYDROTERPHENYL CYANIDES OF LIQUID CRYSTALLINE NATURE

(71) We, MERCK PATENT GESELLSCHAFT MIT BESCHRANKTER HAFTUNG, of 250 Frankfurter Strasse, 61 Darmstadt, Federal Republic of Germany, a Joint-Stock Company organised under the laws of the Federal Republic of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with liquid crystalline hexahydroterphenyl derivatives and with dielectrics containing these derivatives.

For electro-optical indicating elements, there are utilised, to an increasing extent, the properties of nematic or nematic-cholesteric liquid crystalline materials significantly to change their optical properties, such as light scattering, birefringence, reflecting power or colour, under the influence of electric fields. The function of such indicator elements thereby depends, for example, upon the phenomenon of dynamic scattering, the deformation of aligned phases, the Schadt-Helfrich effect in the twisted cell or the cholesteric-nematic phase transition.

For the technical use of these effects in electronic constructional elements, liquid crystalline materials are needed which must satisfy a plurality of requirements. Especially important are here the chemical stability to moisture, air and physical influences, such as heat, radiation in the infra-red, visible and ultra-violet range and direct and alternating electric fields. Furthermore, there is required of technically usable liquid crystalline materials a liquid crystalline mesophase in

the temperature range of at least 10°C to 60°C and more preferably of 0°C to 60°C, and a low viscosity at ambient temperature, which should preferably be not more than 70 cP. Finally, in the range of visible light, they should exhibit no inherent absorption, i.e. they must be colourless.

A number of liquid crystalline compounds are already known which satisfy the stability requirements demanded of dielectrics for electronic constructional elements and are also colourless. These include, in particular, the *p,p'*-disubstituted benzoic acid phenyl esters described in German Patent Specification No. 2,139,628 and the *p,p'*-disubstituted biphenyl derivatives described in German Patent Specification No. 2,356,086. In both mentioned classes of compounds, as well as in other known series of compounds with liquid crystalline mesophase, there are no individual compounds which, in the required temperature range of 10°C to 60°C, form a liquid crystalline nematic mesophase. Therefore, as a rule, mixtures of two or more compounds are prepared in order to obtain substances which can be used as liquid crystalline dielectrics. For this purpose, at least one compound with a low melting and clear point is mixed with another compound with a distinctly higher melting and clear point, a mixture normally being obtained with a melting point which is below that of the lower melting component, whereas the clear point lies between the clear points of the components. However, dielectrics with optimum properties cannot be prepared in this way since the components with the high melting and clear point almost always impart a high viscosity to the mixture. The switch times of the electro-optical indicator

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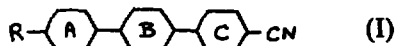
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elements produced with the use of such mixtures are thereby prolonged in an undesirable manner.

It is an object of the present invention to provide liquid crystalline dielectrics which have a nematic phase in the required temperature range and give sufficiently short switch times in liquid crystal cells at ambient temperature.

It has now been found that the new hexahydroterphenyl derivatives of the general formula:—



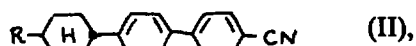
wherein two of the rings A, B and C are aromatic and the third is a *trans*-1,4-disubstituted cyclohexane ring and R is an alkyl or alkoxy radical containing up to 12 carbon atoms, are outstandingly useful as mixture components of liquid crystalline dielectrics.

Although the compounds of general formula (I) preponderantly possess so high a melting point (about 90°C.) and clear point (above 170°C.) that they alone cannot be considered as dielectrics for electronic indicator elements to be operated at ambient temperature, the addition of these compounds (I) to other liquid crystalline substances results in a distinct lowering of the melting point, as well as a favourable increasing of the clear point, without, at the same time, bringing about an undesired increase of the viscosity.

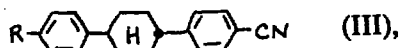
Thus, the present invention provides new hexahydroterphenyl derivatives of general formula (I), as well as a process for the preparation thereof.

Furthermore, the present invention provides liquid crystalline materials which contain at least one known liquid crystalline compound, together with at least one hexahydroterphenyl derivative of general formula (I), as well as dielectrics for electronic constructional elements which consist essentially of such liquid crystalline material mixtures.

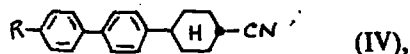
The hexahydroterphenyl derivatives of general formula (I) are either 4 - (4 - *trans* - R - cyclohexyl) - 4' - cyanobiphenyls of the general formula:—



or *trans*-1 - (p - R - phenyl) - 4 - (p - cyanophenyl) - cyclohexanes of the general formula:—



or 4 - R - 4' - (4 - *trans* - cyanocyclohexyl) - biphenyls of the general formula:—



R in general formulae (II), (III) and (IV) having the same meaning as in general formula (I).

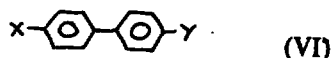
The hexahydroterphenyl derivatives of general formula (I) possess a positive dielectric anisotropy and are, therefore, used as components of liquid crystalline dielectrics for those indicator elements which work on the basis of the Schadt - Helfrich effect in the twisted nematic cell or utilise the phenomenon of cholesteric-nematic phase transition. The compounds of general formulae (II) and (III) have a higher positive dielectric anisotropy.

The substituent R in the compounds of the general Formula (I) can be straight-chained or branched. When R is a straight-chained radical and thus signifies a methyl, ethyl, *n*-propyl, *n*-butyl, *n*-pentyl, *n*-hexyl, *n*-heptyl, *n*-octyl, *n*-nonyl, *n*-decyl, *n*-undecyl or *n*-dodecyl radical or a corresponding alkoxy radical, the compounds of general formula (I) usually have an especially high clear point. Compounds of general formula (I) are especially preferred in which R is an alkyl radical containing up to 10 carbon atoms or an alkoxy radical containing up to 8 carbon atoms. However, compounds of general formula (I) in which R is a branched chain substituent are sometimes also of importance since they frequently have better solubility properties in conventional liquid crystalline base mixtures. Such branched chained substituents R preferably contain not more than one chain branching, preferred branched chain substituents being those in which the carbon chain is branched on the binding carbon atom or on one of the next two carbon atoms. Of importance amongst these are those branched chain radicals in which a methyl or ethyl radical is present on a comparatively long carbon chain in the 1-, 2- or 3-position, for example, isopropyl, 1 - methylpropyl, 2 - methylpropyl, 1 - methylbutyl, 2 - methylbutyl, 3 - methylbutyl, 1 - methylpentyl, 1 - ethylpentyl, 2 - methylpentyl, 1 - methylhexyl, 2 - ethylhexyl or 1 - methylheptyl radicals, as well as the corresponding alkoxy radicals.

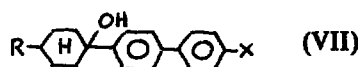
The compounds of general formula (I) can be prepared in the usual way for such compounds, for example, by reacting a compound of the general formula:—



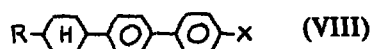
wherein R has the same meaning as in general formula (I), with a compound of the general formula:—



wherein X is a hydrogen atom or an amino group protected against reaction with organometallic reagents by a protective group which is easily split off, for example by a trimethylsilyl group, and Y is -Me¹ or -Me²-Hal, Me¹ and Me² being metal atoms of Main Groups I or II of the Mendeleef Periodic System, preferably lithium or magnesium, and Hal is a halogen atom and preferably a chlorine or bromine atom, and then hydrolysing to give a compound of the general formula:—



After separation of the *cis* - *trans* - isomeric alcohols (VII), these are individually reduced in the usual way, for example, by catalytic hydrogenation, with inversion or retention of the configuration of the benzyl-positioned carbon atom, to give the *trans* - configurational hexahydroterphenyl derivatives of the general formula:—



In these compounds of general formula (VIII), X is converted in the usual way into a nitrile group: thus, for example, a compound of general formula (VIII), in which X is a hydrogen atom, can be reacted in the presence of aluminium chloride with acetyl chloride to give the corresponding acetophenone derivative and this then oxidised, for example with a hypohalite or with iodine, to give a benzoic acid derivative of the general formula:—



The benzoic acid (IX) is then converted in the usual way into a nitrile (II) by successive treatment with thionyl chloride, ammonia and phosphorus oxychloride, *via* the corresponding benzoyl chloride and benzamide.

Furthermore, a compound of general formula (VIII), in which X is a hydrogen atom, can be reacted with thallium tris - trifluoroacetate to give the corresponding phenyl thallium - bis - trifluoroacetate and this then subjected to ultra-violet irradiation in aqueous solution in the presence of potassium cyanide, a nitrile (II) thereby being formed.

Compounds of general formula (VIII), in which X is a protected amino group, are, after splitting off of the protective group, converted into a nitrile (II) by diazotising and reacting with a dicyanocuprate.

In analogous manner, compounds of general formula (III) can also be prepared similarly to compounds of general formula (II) by first reacting a 4-alkyl- (or alkoxy) - phenyl magnesium bromide with 4 - phenylcyclo - hexanone and hydrolysing the reaction mixture to give a *cis* - *trans* - isomeric 1 - (4 - alkyl - or - alkoxy - phenyl) - 4 - phenyl - cyclo - hexanol mixture which, after separation and hydrogenation with inversion or retention of the configuration, is converted into a *trans* - 4 - (4 - alkyl - or - alkoxyphenyl) - 1 - phenyl - cyclohexane. The nitrile group is introduced into these compounds in the 4-position of the unsubstituted phenyl nucleus as described above *via* the corresponding acetophenone derivative or the phenyl thallium bis - trifluoroacetate derivative.

Finally, compounds of general formula (IV) can be obtained by reacting cyclohexene with acetyl chloride in the presence of aluminium chloride to give 4 - acetyl - cyclohexyl chloride which, in turn, is reacted in the presence of aluminium chloride with a compound of the general formula R - (p) - C₆H₄ - C₆H₄ to give a 4 - [4 - (R) - biphenyl - (4')] - 1 - acetylcyclohexane. The acetyl radical in this intermediate product is converted into a carboxyl group by oxidation with hypohalite and this converted, *via* the acid chloride and acid amide, into a nitrile group.

The starting materials for the preparation of the compounds according to the present invention are either known or can be readily prepared by processes known from and described in the literature. Thus, for example, 4 - alkylcyclohexanones (general formula (V), R=alkyl) can be obtained by catalytically hydrogenating the corresponding 4 - alkylphenols and oxidising the 4 - alkylcyclohexanols thus obtained to the corresponding ketones. 4 - Alkoxy-cyclohexanones can be prepared analogously by hydrogenation of the corresponding hydroquinone hemi - ethers and subsequent oxidation of the 4 - alkoxy-cyclohexanols thus obtained.

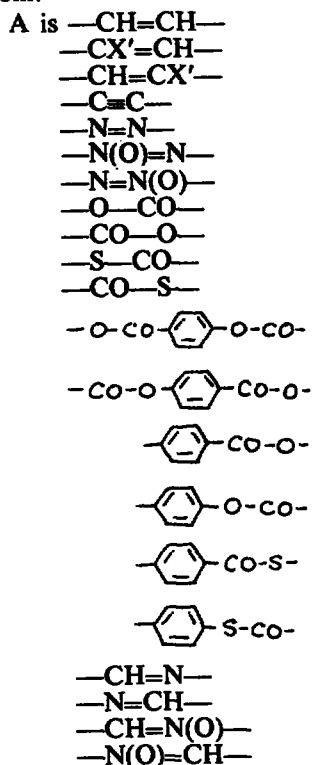
The compounds of the general formula (I) are valuable components of liquid crystalline dielectrics which are to be used for the production of electro-optical indicator elements.

The dielectrics according to the present invention comprise at least one known liquid crystalline component and at least one hexahydroterphenyl derivative of general formula (I). The known components are preferably nematic or nematogenic substances selected from azobenzenes, azoxybenzenes, biphenyls, Schiff bases, especially benzylidene derivatives, phenyl

benzoates, phenyl cyclohexanes, optionally halogenated stilbenes, diphenylacetylene derivatives, diphenyl nitrones and substituted cinnamic acids. The most important compounds which can be used as such further components can be represented by the general formula:—



wherein:



or a carbon-carbon single bond; when A is —CO—O—, —O—CO— or a carbon-carbon single bond, one of the two phenyl rings can be replaced by a *trans*-cyclohexyl ring; X' is a halogen atom and preferably a chlorine atom and R₁ and R₂, which can be the same or different, are alkyl, alkoxy, alkanoyl, alkanoyloxy or alkoxy-carbonyloxy radicals containing up to 18 and preferably up to 8 carbon atoms and one of R₁ and R₂ can also be a cyano, nitro or isonitrile group. In most of these compounds, R₁ and R₂ are preferably different, one of these radicals preferably being an alkyl or alkoxy radical. However, a large number of other variants of the substituents is also usual, many such nematic substances being commercially available.

The dielectrics according to the present invention can contain up to 30, normally 1—20 and preferably 5—15 parts by weight of one or more compounds of general formula (I).

These additions, independently of the nature and composition of the liquid crystalline base substance, increase the clear point by 5 to 40°C., without the viscosity of the base substance and thus the switch time of indicator elements produced with such dielectrics being changed in an undesired way.

By means of appropriate additions, the liquid crystalline dielectrics according to the present invention can be so modified that they can be employed in all previously known types of indicator elements which use liquid crystals with positive dielectric anisotropy. Such additives are known and are described in detail in the relevant literature. For example, substances can be added for changing the dielectric anisotropy and/or the orientation of the nematic phases, substances of this type being described, for example, in German Patent Specifications Nos. 2,209,127; 2,321,632 and 2,611,453.

The following Examples are given for the purpose of illustrating the present invention. In the Examples, m.p. means the melting point and c.p. the clear point of a liquid crystalline substance; boiling temperatures are indicated by b.p. If nothing is stated to the contrary, the parts of percentages are by weight.

Example 1

(a) To a solution of biphenyl magnesium bromide prepared from 46.6 g. 4-bromobiphenyl and 4.9 g magnesium turnings in 250 ml diethyl ether, there is added dropwise with stirring and cooling, in the course of one hour, a solution of 34.0 g 4-*n*-pentylcyclohexanone in 100 ml diethyl ether. The reaction mixture is boiled for a further 1 hour and then poured into a solution of 20 ml. concentrated hydrochloric acid in 400 ml. ice water. The ether phase is separated off and the aqueous phase is shaken out twice with 100 ml. diethyl ether. The combined ether phases are washed with a 5% aqueous sodium bicarbonate solution and then with water and thereafter dried over anhydrous sodium sulphate and evaporated. The residue is separated on a column filled with silica gel. 1-Biphenyl-*cis*-4-*n*-pentyl-*r*-cyclohexanol, eluted with petroleum ether (b.p. 40—60°C) is dissolved in 500 ml ethanol and hydrogenated in the presence of 25 g moist Raney nickel at atmospheric pressure for 72 hours. The *trans* compound, which is eluted with a mixture of petroleum ether (b.p. 40—60°C) and diethyl ether (15 volume percent), is dissolved in 500 ml ethanol and hydrogenated in the presence of 8 g palladium charcoal (5% Pd) at atmospheric pressure for 48 hours. After

filtering of the catalyst, the alcoholic solutions are combined and evaporated. The 4 - [trans - (4 - n - pentyl) - cyclohexyl] - biphenyl obtained in the form of an oil is further worked up without purification.

(b) A suspension of 14.6 g aluminium chloride in 100 ml dichloromethane is successively mixed, while cooling with ice, with 8.6 g acetyl chloride and a solution of 33.7 g 4 - [trans - (4 - n - pentyl) - cyclohexyl] - biphenyl in 100 ml dichloromethane. The reaction mixture is further stirred for 2 hours, poured on to 300 g ice and mixed with sufficient concentrated hydrochloric acid to dissolve the precipitated aluminium hydroxide. The organic phase is separated off and the aqueous phase washed twice with 100 ml amounts of dichloromethane. The combined organic phases are dried over anhydrous calcium chloride and evaporated. The 4 - acetyl - 4' - [trans - (4 - n - pentyl) - cyclohexyl] - biphenyl remaining behind is recrystallised from ethanol; m.p. 125°C; c.p. 200°C.

(c) 14.0 g 4 - Acetyl - 4' - [trans - (4 - n - pentyl) - cyclohexyl] - biphenyl are, together with 30 g. iodine and 100 ml pyridine, heated on a steambath for 2 hours. After evaporating off the pyridine under reduced pressure, the residue is dissolved in chloroform and purified over a short silica gel column. The eluate is evaporated and the 4' - [trans - (4 - n - pentyl) - cyclohexyl] - 4 - biphenyl - carboxylic acid remaining behind is heated to the boil for 2.5 hours with 30 ml thionyl chloride; after distilling off excess thionyl chloride, the 4' - [trans - (4 - n - pentyl) - cyclohexyl] - 4 - biphenylcarboxylic acid chloride obtained is dissolved in 250 ml. dioxan and this solution is mixed with 100 ml 25% aqueous ammonia solution. The reaction mixture is poured into 1.5 litres ice water and the precipitated 4' - [trans - (4 - n - pentyl) - cyclohexyl] - 4 - biphenyl - carboxylic acid amide formed is filtered off and dried. The dried amide is then dissolved at 40°C. in 170 ml. dimethyl formamide. To this solution are added dropwise at 50°C., in the course of 30 minutes, 26 g phosphorus oxychloride. After 1 hour, the reaction mixture is poured into 300 ml ice water; by several extractions with 100 ml amounts of dichloromethane, washing the extracts with 5% sodium bicarbonate solution and then with water, drying over anhydrous sodium sulphate and evaporation, there is isolated 4 - cyano - 4' - (trans - 4 - n - pentyl - cyclohexyl) - biphenyl, which is purified by recrystallisation from ethanol; m.p. 93°C; c.p. 219°C.

The following compounds are prepared analogously:

4 - cyano - 4' - (trans - 4 - methylcyclohexyl) - biphenyl; m.p. 151°C; c.p. 186°C.	65
4 - cyano - 4' - (trans - 4 - ethylcyclohexyl) - biphenyl,	
4 - cyano - 4' - (trans - 4 - n - propylcyclohexyl) - biphenyl; m.p. 132°C; c.p. 230°C,	70
4 - cyano - 4' - (trans - 4 - isopropylcyclohexyl) - biphenyl,	
4 - cyano - 4' - (trans - 4 - n - butylcyclohexyl) - biphenyl,	75
4 - cyano - 4' - [trans - 4 - (1 - methylpropyl) - cyclohexyl] - biphenyl,	
4 - cyano - 4' - [trans - 4 - (2 - methylpropyl) - cyclohexyl] - biphenyl,	80
4 - cyano - 4' - [trans - 4 - (1 - methylbutyl) - cyclohexyl] - biphenyl,	
4 - cyano - 4' - [trans - 4 - (2 - methylbutyl) - cyclohexyl] - biphenyl,	85
4 - cyano - 4' - [trans - 4 - (3 - methylbutyl) - cyclohexyl] - biphenyl,	
4 - cyano - 4' - (trans - 4 - n - hexylcyclohexyl) - biphenyl,	
4 - cyano - 4' - [trans - 4 - (1 - methylpentyl) - cyclohexyl] - biphenyl,	90
4 - cyano - 4' - [trans - 4 - (2 - methylpentyl) - cyclohexyl] - biphenyl,	
4 - cyano - 4' - (trans - 4 - n - heptylcyclohexyl) - biphenyl; m.p. 77°C; c.p. 206°C,	95
4 - cyano - 4' - [trans - 4 - (1 - methylhexyl) - cyclohexyl] - biphenyl,	
4 - cyano - 4' - [trans - 4 - (1 - ethylpentyl) - cyclohexyl] - biphenyl,	100
4 - cyano - 4' - (trans - 4 - n - octylcyclohexyl) - biphenyl; m.p. 61°C; c.p. 188°C,	
4 - cyano - 4' - [trans - 4 - (1 - methylheptyl) - cyclohexyl] - biphenyl,	105
4 - cyano - 4' - [trans - 4 - (2 - ethylhexyl) - cyclohexyl] - biphenyl,	
4 - cyano - 4' - (trans - 4 - n - nonylcyclohexyl) - biphenyl; m.p. 63°C; c.p. 192°C,	110
4 - cyano - 4' - (trans - 4 - n - decylcyclohexyl) - biphenyl,	
4 - cyano - 4' - (trans - 4 - n - undecylcyclohexyl) - biphenyl,	
4 - cyano - 4' - (trans - 4 - n - dodecylcyclohexyl) - biphenyl.	115

Example 2

(a) To a solution of ethyl magnesium bromide prepared from 45.0 g ethyl bromide and 9.6 g magnesium turnings in 400 ml diethyl ether, there is added dropwise a solution of 49.6 g 4 - amino - 4' - bromobiphenyl in 100 ml diethyl ether. Subsequently, there are added at ambient temperature 43.2 g trimethylsilyl chloride in 200 ml diethyl ether. After heating the reaction mixture under reflux for 1 hour, the solvent is evaporated off and the

(12)

residue is extracted with petroleum ether (b.p. 60—80°C). From the extract, there are obtained by crystallisation 42.0 g 4 - N,N - bis - (trimethylsilyl) - amino - 4' - bromobiphenyl. This is dissolved in 200 ml diethyl ether and mixed at 0°C with 54 ml of a 2 molar solution of butyl lithium solution in hexane. After stirring for 30 minutes, a solution of 18.2 g 4 - *n* - butyloxycyclohexanone in 50 ml diethyl ether is added dropwise thereto. The mixture is boiled for 1 hour and, after cooling to ambient temperature, stirred with 200 ml. 1N aqueous hydrochloric acid. The organic phase is washed with 100 ml 5% aqueous sodium bicarbonate solution and then with 100 ml water, dried with anhydrous sodium sulphate and the solvent removed. The residue is separated on a silica gel column, analogously to Example 1(a), into 1 - (4 - amino - 4' - biphenyl) - *cis* - 4 - *n* - butyloxy - *r* - cyclohexanol and 1 - (4 - amino - 4' - biphenyl) - *trans* - 4 - *n* - butyloxy - *r* - cyclohexanol. The isomers are hydrogenated analogously to Example 1(a) in the presence of Raney nickel or of palladium charcoal.

(b) A solution of 24 g 4 - Amino - 4' - (*trans* - 4 - *n* - butyloxycyclohexyl) - biphenyl, obtained by the hydrogenation described in Example 2(a), in 40 ml of approximately 18% by weight hydrochloric acid is slowly mixed at 0°C, while stirring, with a solution of 5.6 g sodium nitrite in 35 ml water. The diazonium salt solution obtained is covered with 150 ml toluene and, while stirring, slowly mixed with 90 ml of a sodium dicyanocuprate solution prepared from 25 g copper sulphate pentahydrate and 21 g sodium cyanide in 100 ml water. The reaction mixture is further stirred vigorously for 30 minutes at about 80°C. Subsequently, the toluene phase is separated off, washed twice with 50 ml amounts of water, dried over anhydrous sodium sulphate and evaporated to give 4 - cyano - 4' - (*trans* - 4 - *n* - butyloxycyclohexyl) - biphenyl, which is recrystallised from ethanol.

The following compounds are prepared analogously:

- 4 - cyano - 4' - (*trans* - 4 - methoxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - ethoxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - *n* - propyloxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - isopropyloxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (1 - methylpropyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (2 -

- methylpropyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - *n* - pentyloxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (1 - methylbutyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (2 - methylbutyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (3 - methylbutyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - *n* - hexyloxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (1 - methylpentyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (2 - methylpentyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - *n* - heptyloxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (1 - methylhexyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - *n* - octyloxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (1 - methylheptyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - [*trans* - 4 - (2 - ethylhexyloxy) - cyclohexyl] - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - *n* - nonyloxycyclohexyl) - biphenyl,
- 4 - cyano - 4' - (*trans* - 4 - *n* - decyloxycyclohexyl) - biphenyl.

Example 3

(a) To a solution of 4 - *n* - pentylphenyl magnesium bromide, prepared from 45.5 g 4 - *n* - pentylbromobenzene and 4.9 g magnesium turnings in 250 ml diethyl ether, there is added dropwise, while stirring and cooling, a solution of 34.8 g 4 - phenylcyclohexanone in 100 ml diethyl ether. The reaction mixture is boiled for 1 hour and then poured into a solution of 20 ml concentrated hydrochloric acid in 400 ml ice water. The ether phase is separated off and the aqueous phase is shaken out twice with 100 ml amounts of diethyl ether. The combined ether phases are washed with 5% aqueous sodium bicarbonate solution and then with water, dried over anhydrous sodium sulphate and evaporated. The residue is subjected to a separation with a column filled with silica gel using, as elution agents, first petroleum ether (b.p. 40—60°C) and then petroleum ether/diethyl ether mixtures, the proportion of diethyl ether increasing from 2 to 15 vol.%. There are obtained about equal amounts of 1 - (4 - *n* - pentyl - phenyl) - *cis* - 4 - phenyl - *r* - cyclohexanol and 1 - (4 - *n* - pentyl - phenyl) - *trans* - 4 - phenyl - *r* - cyclohexanol in the form of an

oil. The *cis* compound is dissolved in 500 ml ethanol and hydrogenated in the presence of 25 g moist Raney nickel at atmospheric pressure for 72 hours. The *trans* compound is dissolved in 500 ml. ethanol and also hydrogenated for 48 hours in the presence of 8 g palladium charcoal (5% Pd). After filtering off the catalyst, the alcoholic solutions are combined and evaporated. The *trans* - 4 - (4 - *n* - pentylphenyl) - 1 - phenyl - cyclohexane obtained in the form of an oil is further worked up without purification.

(b) A suspension of 14.6 g aluminium chloride in 100 ml dichloromethane is successively mixed, while cooling with ice, with 8.6 g acetyl chloride and a solution of 33.7 g *trans* - 4 - (4 - *n* - pentylphenyl) - 1 - phenyl - cyclohexane in 100 ml dichloromethane. The reaction mixture is further stirred for 2 hours, poured on to 300 g ice and mixed with sufficient concentrated hydrochloric acid to dissolve the precipitated aluminium hydroxide. The organic phase is separated off and the aqueous phase is washed twice with 100 ml amounts of dichloromethane. The combined organic phases are dried over anhydrous calcium chloride and evaporated. The *trans* - 4 - (4 - *n* - pentylphenyl) - 1 - (4 - acetylphenyl) - cyclohexane remaining behind is recrystallised from ethanol.

(c) 14.0 g *trans* - 4 - (4 - *n* - pentylphenyl) - 1 - (4 - acetylphenyl) - cyclohexane are heated for 2 hours on a steambath, together with 30 g iodine and 100 ml pyridine. After evaporation of the pyridine under reduced pressure, the residue is dissolved in chloroform and purified over a short silica gel column. The eluate is evaporated and the 4 - [*trans* - 4 - (4 - *n* - pentylphenyl) - cyclohexyl] - benzoic acid remaining behind is boiled for 2.5 hours and with 30 ml thionyl chloride; after distilling off excess thionyl chloride, the 4 - [*trans* - 4 - (4 - *n* - pentylphenyl) - cyclohexyl] - benzoyl chloride formed is dissolved in 250 ml dioxan and this solution is mixed with 100 ml 25% aqueous ammonia solution. The reaction mixture is poured into 1.5 litres ice water and the precipitated 4 - [*trans* - 4 - (4 - *n* - pentylphenyl) - cyclohexyl] - benzamide is filtered off and dried. The dry amide is dissolved at 40°C in 170 ml dimethyl formamide. To this solution are added dropwise at 50°C, in the course of 30 minutes, 26 g phosphorus oxychloride. After 1 hour, the reaction mixture is poured into 300 ml icewater; after extracting several times with 100 ml amounts of dichloromethane, washing the extracts with 5% aqueous sodium bicarbonate solution and then with water, drying over anhydrous sodium sulphate

and evaporating, there is obtained *trans* - 4 - (4 - *n* - pentylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane, which is purified by recrystallisation from ethanol; m.p. 80°C; c.p. 160°C.

The following compounds are obtained analogously:

trans - 4 - (4 - methylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - ethylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - propylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - isopropylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - butylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (1 - methylpropyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (2 - methylpropyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (1 - methylbutyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (2 - methylbutyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (3 - methylbutyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - hexylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (1 - methylpentyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (2 - methylpentyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - heptylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (1 - methylhexyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - octylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (1 - methylheptyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - [4 - (2 - ethylhexyl) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - nonylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - decylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - undecylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - *n* - dodecylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - methoxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

trans - 4 - (4 - ethoxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,

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	<i>trans</i> - 4 - (4 - <i>n</i> - propyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,	65
	<i>trans</i> - 4 - (4 - isopropyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,	
5	<i>trans</i> - 4 - (4 - <i>n</i> - butyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,	70
	<i>trans</i> - 4 - [4 - (1 - methylpropyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	
10	<i>trans</i> - 4 - [4 - (2 - methylpropyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	75
	<i>trans</i> - 4 - (4 - <i>n</i> - pentyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,	
15	<i>trans</i> - 4 - [4 - (1 - methylbutyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	80
	<i>trans</i> - 4 - [4 - (2 - methylbutyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	
20	<i>trans</i> - 4 - [4 - (3 - methylbutyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	85
	<i>trans</i> - 4 - (4 - <i>n</i> - hexyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,	
25	<i>trans</i> - 4 - [4 - (1 - methylpentyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	90
	<i>trans</i> - 4 - [4 - (2 - methylpentyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	
30	<i>trans</i> - 4 - (4 - <i>n</i> - heptyloxyphenyl) - 1 - (cyanophenyl) - cyclohexane,	95
	<i>trans</i> - 4 - [4 - (1 - methylhexyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	
35	<i>trans</i> - 4 - (4 - <i>n</i> - octyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,	100
	<i>trans</i> - 4 - [4 - (1 - methylheptyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	
40	<i>trans</i> - 4 - [4 - (2 - ethylhexyloxy) - phenyl] - 1 - (4 - cyanophenyl) - cyclohexane,	105
	<i>trans</i> - 4 - (4 - <i>n</i> - nonyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane,	
45	<i>trans</i> - 4 - (4 - <i>n</i> - decyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane.	110
	Example 4 (a) To a suspension of 66.6 g anhydrous aluminium chloride in 200 ml carbon disulphide there are added at -15°C, while stirring, 39 g acetyl chloride and thereafter 41 g cyclohexene. The reaction mixture is stirred for 30 minutes at -15°C and then the upper carbon disulphide layer is removed and replaced by 400 ml of a solution of 105 g 4 - <i>n</i> - butylbiphenyl in carbon disulphide. After the addition of a further 33.3 g aluminium chloride, the reaction mixture is warmed, while stirring, to ambient temperature and further stirred until the evolution of hydrogen chloride ceases. Thereafter, the reaction mixture is poured on to icewater, the precipitated aluminium hydroxide is dissolved by adding hydrochloric acid and the organic phase is separated off, dried and distilled. After stripping off the solvent, 4 - acetyl - 1 - [4' - <i>n</i> - butylbiphenyl] - (4) - cyclohexane remains as a yellowish, wax-like product; yield 64 g. (b) Analogously to Example 1(c), from 50 g 4 - acetyl - 1 - [4' - <i>n</i> - butylbiphenyl] - (4) - cyclohexane, there are obtained, by successive reaction with iodine/pyridine, thionyl chloride, aqueous ammonia solution and phosphorus oxychloride, 21.8 g 4 - cyano - 1 - [4' - <i>n</i> - butylbiphenyl] - (4) - cyclohexane. The following compounds are prepared analogously: 4 - cyano - 1 - [4' - methylbiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - ethylbiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - propylbiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - pentylbiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - hexylbiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - heptylbiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - octylbiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - methoxybiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - ethoxybiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - propyloxybiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - butyloxybiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - pentyloxybiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - hexyloxybiphenyl] - (4) - cyclohexane, 4 - cyano - 1 - [4' - <i>n</i> - heptyloxybiphenyl] - (4) - cyclohexane. The following Examples describe liquid crystalline dielectrics according to the present invention: Example 5 A mixture of 41% <i>trans</i> - 4 - (4 - cyanophenyl) - 1 - <i>n</i> - propylcyclohexane, 35% <i>trans</i> - 4 - (4 - cyanophenyl) - 1 - <i>n</i> - pentylcyclohexane and 24% <i>trans</i> - 4 - (4 - cyanophenyl) - 1 - <i>n</i> - heptylcyclohexane (prepared analogously to Example 1 from phenyl magnesium bromide and the corresponding 4 - <i>n</i> - alkyl cyclohexanones) has a clear point of 52°C and a viscosity of 21 cP at 20°C. After the addition of 12% by weight (referred to the weight of the base mixture) of 4 - cyano -	

4' - (*trans* - 4 - *n* - pentylcyclohexyl) - biphenyl, the dielectric has a clear point of 72°C and a viscosity of 29 cP at 20°C.

Example 6

- 5 A mixture of 37% 4 - *n* - pentyl - 4' - cyanobiphenyl, 43% 4 - *n* - heptyl - 4' - cyanobiphenyl, as well as 10% each of 4 - *n* - pentyloxy - 4' - cyanobiphenyl and 4 - *n* - heptyloxy - 4' - cyanobiphenyl, has a
10 clear point of 45°C. By the addition of 18% by weight (referred to the weight of the base mixture) of *trans* - 4 - (4 - *n* - pentylphenyl) - 1 - (4 - cyanophenyl) - cyclohexane, the clear point increases to
15 66°C, whereas the viscosity remains practically unchanged (37 cP at 20°C).

Example 7

- 20 A mixture of 67% anisic acid 4 - *n* - pentylphenyl ester and 33% 4 - *n* - hexyloxybenzoic acid 4' - *n* - pentylphenyl ester has a clear point of 49°C and a viscosity of 59 cP at 20°C. By the addition of 21.5% by weight 4 - cyano - 4' - *trans* - 4 - *n* - heptylcyclohexyl - biphenyl, a
25 mixture is obtained which has a clear point of 83°C and a viscosity of 50 cP at 20°C.

Example 8

- 30 A mixture of 40% 4 - *n* - butyl - 4' - methoxyazoxybenzene, 22% 4 - ethyl - 4' - methoxyazoxybenzene, 20% dimethylaminobenzonitrile and 18% 4 - (4 - *n* - butylbenzoyloxy) - benzoic acid 4' - *n* - butylphenyl ester has a clear point of 31°C and a viscosity of 50 cP at 20°C. By
35 the addition of 15% by weight *trans* - 4 - (4 - *n* - hexyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane, the clear point increases to 57°C., whereas the viscosity decreases to 45 cP at 20°C.

WHAT WE CLAIM IS:—

1. Hexahydroterphenyl derivatives of the general formula:—



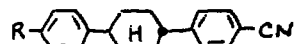
- 45 wherein two of the rings A, B and C are aromatic and the third is a *trans* - 1,4 - disubstituted cyclohexane ring and R is an alkyl or alkoxy radical containing up to 12 carbon atoms.

2. Hexahydroterphenyl derivatives of the general formula:—



wherein R has the same meaning as in claim 1.

3. Hexahydroterphenyl derivatives of the general formula:—



wherein R has the same meaning as in claim 1.

4. Hexahydroterphenyl derivatives of the general formula:—



wherein R has the same meaning as in claim 1.

5. Hexahydroterphenyl derivatives according to any of the preceding claims, wherein R is a straight-chained alkyl or alkoxy radical. 65

6. Hexahydroterphenyl derivatives according to any of the preceding claims, wherein R is an alkyl radical containing up to 10 carbon atoms. 70

7. Hexahydroterphenyl derivatives according to any of claims 1 to 5, wherein R is an alkoxy radical containing up to 8 carbon atoms. 75

8. Hexahydroterphenyl derivatives according to any of claims 1 to 4, wherein R is a branched-chain alkyl or alkoxy radical.

9. Hexahydroterphenyl derivatives according to claim 1 which are hereinbefore specifically exemplified. 80

10. A compound of the group:

4 - cyano - 4' - (*trans* - 4 - *n* - propylcyclohexyl) - biphenyl, 85

4 - cyano - 4' - (*trans* - 4 - *n* - butylcyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - pentylcyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - hexylcyclohexyl) - biphenyl, 90

4 - cyano - 4' - (*trans* - 4 - *n* - heptylcyclohexyl) - biphenyl, and

4 - cyano - 4' - (*trans* - 4 - *n* - octylcyclohexyl) - biphenyl.

11. A compound of the group: 95

4 - cyano - 4' - (*trans* - 4 - methoxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - ethoxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - propyloxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - butyloxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - pentyloxycyclohexyl) - biphenyl, 105

4 - cyano - 4' - (*trans* - 4 - *n* - hexyloxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - heptyloxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - octyloxycyclohexyl) - biphenyl, 110

4 - cyano - 4' - [*trans* - 4 - (2 - methylbutyloxy) - cyclohexyl] - biphenyl, and

4 - cyano - 4' - [*trans* - 4 - (2 - ethylhexyloxy) - cyclohexyl] - biphenyl. 115



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4' - (*trans* - 4 - *n* - pentylcyclohexyl) - biphenyl, the dielectric has a clear point of 72°C and a viscosity of 29 cP at 20°C.

Example 6

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10 clear point of 45°C. By the addition of 18% by weight (referred to the weight of the base mixture) of *trans* - 4 - (4 - *n* - pentylphenyl) - 1 - (4 - cyanophenyl) -
15 cyclohexane, the clear point increases to 66°C, whereas the viscosity remains practically unchanged (37 cP at 20°C).

Example 7

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25 of 21.5% by weight 4 - cyano - 4' - (*trans* - 4 - *n* - heptylcyclohexyl) - biphenyl, a mixture is obtained which has a clear point of 83°C and a viscosity of 50 cP at 20°C.

Example 8

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35 4' - *n* - butylphenyl ester has a clear point of 31°C and a viscosity of 50 cP at 20°C. By the addition of 15% by weight *trans* - 4 - (4 - *n* - hexyloxyphenyl) - 1 - (4 - cyanophenyl) - cyclohexane, the clear point increases to 57°C., whereas the viscosity decreases to 45 cP at 20°C.

WHAT WE CLAIM IS:—

1. Hexahydroterphenyl derivatives of the general formula:—



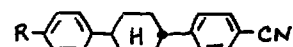
45 wherein two of the rings A, B and C are aromatic and the third is a *trans* - 1,4 - disubstituted cyclohexane ring and R is an alkyl or alkoxy radical containing up to 12 carbon atoms.

50 2. Hexahydroterphenyl derivatives of the general formula:—



wherein R has the same meaning as in claim 1.

55 3. Hexahydroterphenyl derivatives of the general formula:—



wherein R has the same meaning as in claim 1.

4. Hexahydroterphenyl derivatives of the general formula:—



wherein R has the same meaning as in claim 1.

5. Hexahydroterphenyl derivatives according to any of the preceding claims, wherein R is a straight-chained alkyl or alkoxy radical. 65

6. Hexahydroterphenyl derivatives according to any of the preceding claims, wherein R is an alkyl radical containing up to 10 carbon atoms. 70

7. Hexahydroterphenyl derivatives according to any of claims 1 to 5, wherein R is an alkoxy radical containing up to 8 carbon atoms. 75

8. Hexahydroterphenyl derivatives according to any of claims 1 to 4, wherein R is a branched-chain alkyl or alkoxy radical.

9. Hexahydroterphenyl derivatives according to claim 1 which are hereinbefore specifically exemplified. 80

10. A compound of the group:

4 - cyano - 4' - (*trans* - 4 - *n* - propylcyclohexyl) - biphenyl, 85

4 - cyano - 4' - (*trans* - 4 - *n* - butylcyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - pentylcyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - hexylcyclohexyl) - biphenyl, 90

4 - cyano - 4' - (*trans* - 4 - *n* - heptylcyclohexyl) - biphenyl, and

4 - cyano - 4' - (*trans* - 4 - *n* - octylcyclohexyl) - biphenyl.

11. A compound of the group: 95

4 - cyano - 4' - (*trans* - 4 - methoxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - ethoxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - propyloxycyclohexyl) - biphenyl, 100

4 - cyano - 4' - (*trans* - 4 - *n* - butyloxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - pentyloxycyclohexyl) - biphenyl, 105

4 - cyano - 4' - (*trans* - 4 - *n* - hexyloxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - heptyloxycyclohexyl) - biphenyl,

4 - cyano - 4' - (*trans* - 4 - *n* - octyloxycyclohexyl) - biphenyl, 110

4 - cyano - 4' - [*trans* - 4 - (2 - methylbutyloxy) - cyclohexyl] - biphenyl, and

4 - cyano - 4' - [*trans* - 4 - (2 - ethylhexyloxy) - cyclohexyl] - biphenyl. 115

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12. A compound of the group:
trans - 4 - (4 - *n* - propylphenyl) - 1 -
5 (4 - cyanophenyl) - cyclohexane,
trans - 4 - (4 - *n* - butylphenyl) - 1 - (4 -
cyanophenyl) - cyclohexane,
trans - 4 - (4 - *n* - pentylphenyl) - 1 -
(4 - cyanophenyl) - cyclohexane,
trans - 4 - (4 - *n* - hexylphenyl) - 1 -
10 (4 - cyanophenyl) - cyclohexane,
trans - 4 - (4 - *n* - heptylphenyl) - 1 -
(4 - cyanophenyl) - cyclohexane, and
trans - 4 - (4 - *n* - octylphenyl) - 1 - (4 -
cyanophenyl) - cyclohexane.
13. A process for the preparation of
15 compounds of the general formula given in
claim 1, substantially as hereinbefore
described and exemplified.
14. Compounds of the general formula
20 given in claim 1, whenever prepared by the
process according to claim 13.

15. A liquid crystalline dielectric
containing at least one known liquid
crystalline component and at least one
hexahydroterphenyl derivative of the
general formula given in claim 1. 25
16. A liquid crystalline dielectric
according to claim 15, wherein it contains
up to 30 percent by weight of the
hexahydroterphenyl derivative.
17. Liquid crystalline dielectrics 30
according to claim 15, substantially as
hereinbefore described and exemplified.

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