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Solvent for the dye of pressure-sensitive recording paper.

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A solvent for the dye of pressure-sensitive recording paper which solvent has no offensive odor and is excellent regarding the color developing rate and developed color density. The solvent is characterized in that it comprises a fraction mainly containing triaryldialkanes having boiling points above 350 °C but not higher than 450 °C which is prepared by disproportionating diarylalkane or a hydrocarbon mixture comprising the same at temperatures in the range of 20 to 500 °C in the presence of a disproportionation catalyst, said diarylalkane having a boiling point in the range of 260 to 320 °C.

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SOLVENT FOR THE DYE OF
PRESSURE-SENSITIVE RECORDING PAPER

BACKGROUND OF THE INVENTION

5 (1) Field of the Invention

This invention relates to a solvent for the dye of pressure-sensitive recording paper. More particularly, the invention relates to a solvent for the dye of pressure-sensitive recording paper which solvent comprises a heavier
10 fraction that is prepared by disproportionating diarylalkanes or a mixture mainly containing diarylalkanes in the presence of a disproportionation catalyst.

(2) Description of the Prior Art

Various kinds of pressure-sensitive recording
15 papers are hitherto well known. For example, a colorless dye, that is a dye-precursor, is dissolved in a solvent and the obtained solution is encapsulated into microcapsules. One surface of a sheet of paper is coated with microcapsules and the surface of another sheet of paper is coated with
20 clay or a polymeric material such as phenol resin (color developer) which produces a color upon reaction with the dye-precursor. When the recording paper is used, the treated surfaces of the above set of paper sheets are put together face to face and local pressure is applied to the
25 paired sheets by handwriting or any other measure, thereby obtaining desired duplicate impressions.

The recording mechanism in the pressure-sensitive recording paper is such that the microcapsules on the surface of a sheet of paper are ruptured by the pressure of handwriting or else to release the dye solution from the microcapsules. The solution containing a dye comes into
5 contact with clay or polymeric material (color developer) on the opposite surface of the other sheet of paper to produce a color.

The dyes for pressure-sensitive recording paper
10 are exemplified by Crystal Violet Lactone, Malachite Green, benzoyl Leucomethylene Blue, Rhodamine B and 3-dialkylamino-7-dialkylamino fluoran.

As the solvents for pressure-sensitive recording paper, diarylalkanes such as diphenylmethane and phenyl-
15 xylylethane are conventionally used. Some of diarylalkanes, however, have offensive odor due to their chemical structures, and thus are not suitable as the solvents for pressure-sensitive recording paper.

Meanwhile, it is known that diphenylethanes are
20 contained in the by-product oil that is produced in a certain preparation process. However, the by-product oil usually contains many kinds of other compounds and it is impossible to separate them simply by distillation. For this reason, even though the diphenylethanes in the by-product oil are not
25 expensive, they are not widely used due to their offensive odor and for other reasons. For example, as disclosed in Japanese Laid-Open Patent Publication No. 56-161195,

it is necessary to treat them in order to remove offensive odor and to add some odor-masking agents.

Other aromatic hydrocarbons such as dibenzyltoluene that is heavier than diarylalkanes are also proposed as the solvents for pressure-sensitive recording paper. Due to their higher molecular weight, however, their pour points and viscosities are generally high and their color developing properties are not always satisfactory.

Accordingly, heavier aromatic hydrocarbons are scarcely used as solvents for pressure-sensitive recording paper.

BRIEF SUMMARY OF THE INVENTION

It is, therefore, the primary object of the present invention to provide an improved solvent for the dye of pressure-sensitive recording paper which is free from the above-described disadvantages of the conventional art.

Another object of the present invention is to provide a solvent for the dye of pressure-sensitive recording paper which solvent has no offensive odor though it is prepared from diarylalkanes, shows excellent color developing property and anti-fading property, and has a sufficiently low pour point and viscosity irrespective of its high boiling point.

A further object of the present invention is to provide a solvent for the dye of pressure-sensitive recording paper, which solvent is inexpensive, has desirable properties and can be prepared from a by-product oil fraction of a hydrocarbon mixture mainly containing diarylalkanes that is

obtained from a specific production process.

According to the present invention, the solvent for the dye of pressure-sensitive recording paper is characterized in that the solvent comprises a fraction which is prepared by disproportionating one or more diarylalkanes or a hydrocarbon mixture mainly containing the same at temperatures in the range of 20 to 500°C in the presence of a disproportionation catalyst to obtain a fraction mainly containing triaryldialkanes having boiling points above 350°C but not higher than 450°C converted to atmospheric pressure basis.

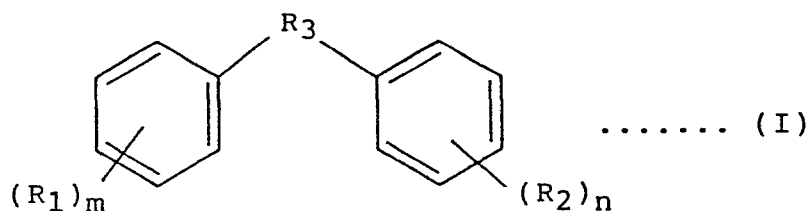
A preferable example of the above-mentioned hydrocarbon mixture mainly containing diarylalkanes is the by-product oil fraction that is obtained in the process to produce alkylated monocyclic aromatic hydrocarbons by alkylating monocyclic aromatic hydrocarbons such as benzene and toluene with olefins such as ethylene in the presence of an alkylation catalyst. When the above by-product oil fraction is used as a starting material, it is inexpensive and a fraction containing triaryldialkanes can be obtained efficiently, in addition, it is desirable because a solvent for the dye of pressure-sensitive recording paper which scarcely has offensive odor can be obtained.

DETAILED DESCRIPTION OF THE INVENTION

The diarylalkanes according to the present invention are represented by the following general formula (I) and the boiling points of the above diarylalkanes or the hydrocarbon mixture mainly containing the same are in the

range of 260 to 320°C and preferably 260 to 310°C.

Diarylalkanes or a hydrocarbon mixture having a boiling point higher than the above range is not desirable because the effect of below-described disproportionation cannot be expected.



wherein each of R_1 and R_2 is a hydrogen atom or a straight chain or branched chain alkyl group; R_3 is a straight chain or branched chain alkylene group; and each of m and n is an integer from 0 to 3. The above diarylalkanes are used singly or in a mixture of two or more kinds.

Exemplified as the above diarylalkanes are diphenylmethane, ditolylmethane, diphenylethane, phenyltolylethane and ditolylethane.

The hydrocarbon mixture mainly containing diarylalkanes that is used as a preferable starting material is a by-product oil that is obtained from a specific preparation process. More particularly, it is a by-product oil fraction which is produced in a process for preparing alkylated monocyclic aromatic hydrocarbons such as alkylbenzene by alkylating monocyclic aromatic hydrocarbons with olefins in the presence of an alkylation catalyst.

The monocyclic aromatic hydrocarbons are benzene and lower alkylbenzenes such as toluene and the olefins are

lower olefins such as ethylene and propylene. Among alkylation catalysts mainly used industrially are Lewis acids such as aluminum chloride and boron fluoride, synthetic zeolites that are typically represented by ZSM-5
5 type zeolite such as ZSM-5 and ZSM-11, and protonic acids such as phosphoric acid.

The above-mentioned alkylation is widely put into practice as a preparation process for lower alkylbenzenes such as ethylbenzene, ethyltoluene and cumene. Ethylbenzene
10 and ethyltoluene which are produced by alkylating benzene and toluene with ethylene, are dehydrogenated into styrene and methylstyrene, respectively, and they are consumed in large quantities for producing polymers.

An example of ethylbenzene preparation process
15 will be described, in which benzene is alkylated with ethylene in the presence of aluminum chloride catalyst.

The molar ratio of the feed of benzene to ethylene is, for example, about 10:1 to about 3:1. In liquid phase alkylation, 0.005 to 0.030 part of catalyst is added to one
20 part of produced ethylbenzene. The reaction is carried out at temperatures of 90 to 150°C, pressures of 0.5 to 15 kg/cm² and duration of 20 minutes to 3 hours.

Through the above alkylation, unreacted benzene, aimed ethylbenzene, polyethylbenzenes such as diethylbenzene
25 and triethylbenzene, and the by-product oil fraction containing diarylalkanes are obtained.

After the alkylation, the catalyst is removed by a conventional method. For example, the catalyst is separated by sedimentation in a settler, which is followed by neutralization and repeated water rinsing.

5 Then the unreacted benzene (b.p. 80°C), ethylbenzene (b.p. 136°C) and polyethylbenzene (b.p. 176 to 250°C) are recovered by distillation from the alkylation product to obtain the by-product oil containing diaryl-alkanes, as the bottoms.

10 The by-product oil fraction which is especially preferable for the present invention in the fraction obtained from the process to produce ethylbenzene or ethyltoluene by alkylating benzene or toluene with ethylene. This by-product oil fraction substantially comprises diaryl-
15 alkanes and can be obtained in large quantities at low cost. Furthermore, the effect of disproportionation of the invention can be produced markedly. Accordingly, said by-product oil fraction is desirable as the starting material to be used in the present invention.

20 In the present invention, the above-described starting material is subjected to disproportionation in the presence of a disproportionation catalyst.

 The disproportionation catalysts are exemplified by Lewis acids such as aluminum chloride and ferric chloride,
25 solid acids such as silica-alumina, and synthetic zeolites represented by ZSM-5 type zeolites such as ZSM-5 and ZSM-11, heteropoly acids such as silicotungstic acid, super strong

acids such as trifluoromethane sulfonic acid, and super strongly acidic cation exchange resin such as Nafion (trademark, made by E.I. du Pont de Nemours). However, sulfuric acid and natural clay such as activated clay are
5 not preferable because the disproportionation does not proceed substantially.

The temperatures for the disproportionation can be selected in a wide range of 20 to 500°C depending on the kind of the catalyst. For example, the temperature range of 20 to
10 150°C is suitable for aluminum chloride; 150 to 230°C, for Nafion; and 250 to 500°C, for synthetic zeolite. The disproportionation does not occur at temperatures lower than the above range, while side reactions such as decomposition occur at temperatures higher than the above range,
15 neither of which is, accordingly, desirable.

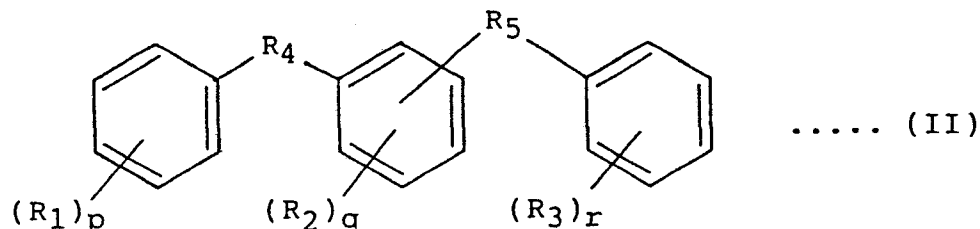
With regard to the type of reaction, any of batchwise and continuous types can be employed.

Preferable reaction times are 20 minutes to 10 hours in batchwise reaction and 0.5 to 10 in LHSV in
20 continuous reaction.

The pressures of disproportionation are not especially limited, however, they are generally in the range of atmospheric pressure to 10 kg/cm².

In the disproportionation according to the
25 invention, monocyclic aromatic hydrocarbons, i.e. benzene and alkylbenzene, such as toluene that are lighter than the starting hydrocarbons and triaryldialkanes that are heavier

than the starting hydrocarbons and are represented by the following general formula (II), are produced.



wherein each of R_1 , R_2 and R_3 is a hydrogen atom or a straight chain or branched chain alkyl group; each of R_4 and R_5 is a straight chain or branched chain alkylene group; and each of p , q and r is an integer from 0 to 3.

The present invention is characterized in that the fraction mainly containing the foregoing triaryldialkanes is used as the solvent for the dye of pressure-sensitive recording paper. Accordingly, after the disproportionation, the catalyst is removed by settling or filtration and the above fraction mainly containing triaryldialkanes is obtained by distillation. The boiling temperature of the fraction that is suitable as the solvent for the dye of pressure-sensitive recording paper is above 350°C but not higher than 450°C, preferably below 420°C as converted to atmospheric pressure basis. In the fraction containing triaryldialkanes having boiling points above 450°C, the viscosity and pour point are too high which are not desirable.

As described above, since monocyclic aromatic hydrocarbons that are lighter than the starting material are produced in the disproportionation, lighter materials can be

continuously removed outside the reaction system especially in a batchwise system. This is desirable because the yield of the above triaryldialkanes can be improved.

5 The fraction mainly containing triaryldialkanes is preferable because it has no offensive odor. In addition, in spite of its higher boiling point, the viscosity and pour point are relatively low. Furthermore, the dissolving property (dissolving power) and color developing property relative to the conventional dyes are also excellent.

10 Accordingly, the above fraction is most suitable for use as the solvent for the dye of pressure-sensitive recording paper. Still further, when the by-product oil fraction in alkylation process is used as a starting material, it is desirable because the solvent can be produced at lower cost.

15 The solvents according to the present invention can be used singly or in a mixture with one or more kinds of known solvents, for example, kerosene, alkylbenzenes such as dodecylbenzene, diarylalkanes such as 1-phenyl-1-xylylethane and 1-phenyl-2-isopropylphenylethane, alkylbiphenyls such as

20 isopropylbiphenyl, and alkylnaphthalenes such as diisopropylnaphthalene.

Among the dyes (dye-precursors), there are typically triarylmethane type compounds, diphenylmethane type compounds, xanthene type compounds, thiazine type compounds, and

25 spiropyran type compounds.

The dye-precursors of triarylmethane type compounds are exemplified by:

3,3-bis(p-dimethylaminophenyl)-6-dimethylaminophthalide
(Crystal Violet Lactone);

- 5 3,3-bis(p-dimethylaminophenyl)phthalide; 3-(p-dimethylaminophenyl)-3-(1,2-dimethylindole-3-yl)-phthalide;
3-(p-dimethylaminophenyl)-3-(2-methylindole-3-yl)phthalide;
3-(p-dimethylaminophenyl)-3-(2-phenylindole-3-yl)phthalide;
3,3-bis(1,2-dimethylindole-3-yl)-5-dimethylaminophthalide;
10 3,3-bis(1,2-dimethylindole-3-yl)-6-dimethylaminophthalide;
3,3-bis(9-ethylcarbazole-3-yl)-5-dimethylaminophthalide;
3,3-bis(2-phenylindole-3-yl)-5-dimethylaminophthalide; and
3-p-dimethylaminophenyl-3-(1-methylpyrrole-2-yl)-6-dimethylaminophthalide.

- 15 The dye-precursors of diphenylmethane type compounds are exemplified by:

4,4-bis-dimethylaminobenzhydrine benzyl ether;
N-halophenyl leuco Auramine; and
N-2,4,5-trichlorophenyl leuco Auramine.

- 20 The xanthene type dye-precursors are exemplified by: Rhodamine B-anilinolactam;

Rhodamine B-(p-nitroanilino)lactam;

Rhodamine B-(p-chloroanilino)lactam;

3-dimethylamino-6-methoxyfluoran;

- 25 3-diethylamino-7-methoxyfluoran;

3-diethylamino-7-chloro-6-methylfluoran;

3-diethylamino-7-(acetylmethylamino)fluoran;

3-diethylamino-7-(dibenzylamino)fluoran;
3-diethylamino-7-(methylbenzylamino)fluoran;
3-diethylamino-7-(chloroethylmethylamino)fluoran;
3-diethylamino-7-(diethylamino)fluoran; and
5 3-diethylamino-6-methyl-7-anilinofluoran.

The thiazine type dye-precursors are exemplified by: benzoyl leuco methylene blue and p-nitrobenzoyl leuco methylene blue.

The spiro type dye-precursors are exemplified by:
10 3-methyl-spiro-dinaphthopyran; 3-ethyl-spiro-dinaphthopyran;
3,3'-dichloro-spiro-dinaphthopyran;
3-benzyl-spiro-dinaphthopyran;
3-methylnaphtho-(3-methoxybenzo)spiropyran; and
3-propyl-spiro-dibenzodipyran.

15 The dye-precursors can be dissolved in the solvent of the invention in the same manner as when using conventional solvents.

Among the color developer, there are clay, polymers, and aromatic carboxylic acids or their metal salts.

20 The polymers are exemplified by phenol-aldehyde polymer, phenol-acetylene polymer, maleic acid-rosin polymer, partially or completely hydrolyzed styrene-maleic anhydride copolymer, partially or completely hydrolyzed ethylene-maleic anhydride copolymer, carboxy polyethylene, and partially or
25 completely hydrolyzed vinyl methyl ether-maleic anhydride copolymer.

The aromatic carboxylic acids and their derivatives are exemplified by: 3,5-di(α -methylbenzyl)salicylic acid; 3-(α -methylbenzyl)-5-(α,α -dimethylbenzyl)salicylic acid; 3-(4'- α' , α' -dimethylbenzyl)phenyl-5-(α,α -dimethylbenzyl)-
5 salicylic acid; 3,5-di-tert-butyl salicylic acid; 3,5-di-tert-octyl salicylic acid; 3-cyclohexyl-5-(α,α -dimethylbenzyl)salicylic acid; 3-phenyl-5-(α,α -dimethylbenzyl)salicylic acid; and 3,5-di(α,α -dimethylbenzyl)salicylic acid. Furthermore,
10 their salts of polyvalent metals such as zinc, aluminum, barium, tin, iron, calcium and lead can also be used.

As the method to prepare the microcapsules of the dye-precursor solution, which is obtained by dissolving a dye-precursor in the solvent, there is a coacervation
15 method in which the fine particles of the dye-precursor solution that are dispersed in water are coated by a protective colloidal material such as gelatin or gum arabic, thereby obtaining the microcapsules which contain the dye-precursor solution. Another method is the inter-
20 facial polymerization method or a in situ polymerization method in which a monomer or a partially condensed polymerizable product is employed and a polymerization initiator, an accelerator or a catalyst is added to cause polymerization on the surfaces of fine particles of the
25 dye-precursor solution, thereby preparing the microcapsules containing the dye-precursor solution. The specific solvent of the present invention can be used in any one of

the above methods.

In the practical process for preparing micro-capsules in the conventional art, an auxiliary solvent has been used in dissolving a dye-precursor in order to control the viscosity and volatility of the dye-precursor solution, the particle size of the fine dispersion in microcapsule formation, the dissolving property to the polymeric material that is coated onto the surface to be recorded, and the rate of color development. However, the specific solvent of the present invention can satisfactorily be used without employing such an auxiliary solvent. Nevertheless, any solvent which does not degrade the characteristics of the solvent of the present invention may be used as an auxiliary solvent. It should be noted also that the specific solvent of the present invention can be used together with conventional solvents.

The present invention will be described in more detail with reference to the following examples.

Example 1

Using aluminum chloride catalyst, alkylation was carried out by reacting batchwise benzene with ethylene in a molar ratio (benzene:ethylene) of 5:1 at 130°C and 4.9 kg/cm² for 1 hour.

Unreacted benzene, ethylbenzene and polyethylbenzene were distilled off from the above obtained reaction mixture to recover a by-product oil fraction of boiling points 260 to 310°C (converted to atmospheric pressure) that

contained the following diarylalkanes.

	Composition	% by weight
	C ₁₄ - C ₁₆ diarylethane	71
	O t h e r s	29
5	T o t a l	100

(Triaryldialkane was scarcely contained.)

The above by-product oil fraction had strong offensive odor and could not be used as a solvent.

Then, 30 g of aluminum chloride was added to 2000 ml
10 of the above by-product oil fraction and it was disproportionated at 80°C for 5 hours with stirring.

After the disproportionation, the catalyst was deactivated and the disproportionation product was rinsed with water and dried. It was followed by distillation to
15 obtain a C₆ - C₉ monocyclic aromatic hydrocarbon fraction of 80 to 160°C in boiling points (yield: 5.0%), unreacted by-product oil fraction and heavier fraction of 351 to 400°C in boiling points (yield: 14.8%).

It was confirmed that the above heavier fraction
20 mainly contained triaryldialkanes by GC-mass spectrum analysis. Furthermore, even though the fraction has high boiling points, the pour point and viscosity of the fraction were low as follows:

	Pour point	-27.5°C
25	Viscosity	18.5 cSt (at 40°C)

This fraction had almost no odor.

Then, 5.0 g of crystal Violet was added to 100 g

of the above fraction and it was emulsified by adding 100 g of gelatin, and water was added to make up 600 g as the whole. The pH of the above was adjusted to 4.5 with adding an aqueous solution of CMC and the capsule membranes were
5 cured by glutaraldehyde to prepare microcapsule slurry.

A paste (CMC aqueous solution) and a blocking agent were added to the above obtained microcapsule slurry and they were mixed well. The above mixture was applied uniformly to sheets of quality paper using a wire bar and
10 they were dried. The coated paper sheets were used as test papers where the the quantity of coated microcapsules was determined by weighing the test paper that was maintained at $25 \pm 1^{\circ}\text{C}$ and 60% humidity.

This test paper was combined with another sheet of
15 paper that was coated with acid clay to obtain a set of pressure-sensitive recording paper. This pressure-sensitive recording paper was applied with a load of 675 kg/cm^2 for 1 minute and color densities at 1 minute and 60 minutes after removing the load were obtained by determining the
20 absorbances at 610 nm. The results are shown in the following Table 1.

For comparison purpose, 10% by weight of activated clay powder was added to the foregoing by-product oil fraction and it was treated at 180°C for 1 hour. Meanwhile,
25 to the by-product oil fraction was added 5% by volume of 97 wt.% sulfuric acid and it was shaken at room temperature for 30 minutes. The two of treated by-product oil fractions

were analyzed by GC-mass spectrum where the formation of triaryldialkane was hardly observed. Furthermore, the odor was hardly changed after the above treatments.

Example 2

5 Using synthetic zeolite ZSM-5, toluene was alkylated with ethylene under the following conditions:

Reaction temperature	500°C
Toluene/ethylene	5 (molar ratio)
WHSV	10

10 After the alkylation, unreacted toluene, ethyltoluene and polyethyltoluene were distilled off from the reaction mixture to obtain a heavier fraction, which had the following property and composition:

By-Product Oil Fraction

15 Boiling Point	260 - 300°C
Composition	% by weight
C ₁₄ - C ₁₅ diarylmethane	59.1
C ₁₆ diarylethane	25.9
O t h e r s	15.0
20 T o t a l	100.0

(Triaryldialkane was scarcely contained)

The odor of the by-product oil fraction obtained here was better than the by-product oil fraction obtained in Example 1, however, it had considerable offensive odor yet.

25 The above by-product oil fraction (2000 ml) was disproportionated at 200°C for 3 hours under atmospheric pressure by using 50 g of strongly acidic cation exchange

resin (trademark: Nafion made by du Pont de Nemours).

During the disproportionation, the produced lighter fractions of benzene and C₇ - C₉ alkylbenzenes such as toluene were removed continuously from the reaction system.

5 After the disproportionation, the catalyst was filtered off and 1550 ml of the filtrate was distilled to recover Fraction 2 containing diarylalkanes corresponding to unreacted fraction and Fraction 3 containing heavier triaryl-
10 dialkane. Incidentally, the lighter fraction which was removed during the disproportionation was also collected, it is also shown in the following:

Fraction	Boiling Point	Recovery
Lighter fraction	80 - 160°C	9.9%
Fraction 3	351 - 400°C	29.5%

15 It was confirmed that the above Fraction 3 mainly contained triaryldialkanes by GC-mass spectrum analysis. This fraction had almost no odor and the properties of the fraction were low as follows:

Pour point	-35°C
20 Viscosity	16.8 cSt (at 40°C)

 Using the above Fraction 3, pressure-sensitive recording papers were prepared in the like manner as Example 1 and color densities and rates of color development were determined. The results are shown in the following Table 1.

25 For comparison purpose, other pressure-sensitive recording papers were prepared in the same manner as Example 1 by using, as solvents, diisopropylnaphthalene (trademark:

KMC made by Kureha Chemical Industry) and partially hydrogenated terphenyl (trademark: HB-40 made by Hüls), and similar tests were carried out. Their results are also shown in Table 1.

5

T a b l e 1

Example	Time	
	1 minute	60 minutes
Example 1	39.7	40.9
Example 2	39.6	40.8
" (K M C)	34.5	38.8
" (HB-40)	30.7	38.0

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CLAIMS

1. A solvent for the dye of pressure-sensitive recording paper comprising a fraction mainly containing triaryldialkanes having boiling points above 350°C but not higher than 450°C, which fraction is obtainable by disproportionating one or more diarylalkanes or a hydrocarbon mixture comprising the same at temperatures in the range of 20 to 500°C in the presence of a disproportionation catalyst, said diarylalkane(s) having boiling points in the range of 260 to 320°C.

2. The solvent of claim 1, wherein said disproportionation catalyst is a Lewis acid.

3. The solvent of claim 2, wherein said Lewis acid is aluminum chloride.

4. The solvent of claim 1, wherein said disproportionation catalyst is a solid acid.

5. The solvent of claim 4, wherein said solid acid is a synthetic zeolite.

6. The solvent of any of the claims 1 to 5, wherein said hydrocarbon mixture is the by-product oil fraction mainly containing diarylalkanes and being obtained in the process to prepare an alkylated monocyclic aromatic hydrocarbon by
5 alkylating a monocyclic aromatic hydrocarbon with an olefin in the presence of an alkylation catalyst.

7. The solvent of claim 6, wherein said monocyclic aromatic hydrocarbon is benzene or toluene.

8. The solvent of claim 6 or 7, wherein said
10 olefin is ethylene.

9. The solvent of any of the claims 6 to 8, wherein said alkylation catalyst is a Lewis acid or a solid acid.

10. The solvent of claim 9, wherein said Lewis
15 acid is aluminum chloride.

11. The solvent of claim 9, wherein said solid acid is a synthetic zeolite.

12. A pressure-sensitive recording material comprising a dye-precursor which produces a color when
20 said dye-precursor is brought into contact with a color developer, wherein the solvent for said dye-precursor is a solvent according to any of the claims 1 to 11.