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(54) Solvent composition.

(57) The present application provides:
(1) a solvent comprising a fluorine-containing alcohol,
(2) a solvent composition comprising a fluorine-containing alcohol and water.
(3) a solvent composition comprising a fluorine-containing alcohol, an aliphatic alcohol and water, and
(4) a solvent composition comprising a fluorine-containing alcohol and a surfactant.

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SOLVENT COMPOSITION

FIELD OF THE INVENTION

This invention relates to a solvent composition.

BACKGROUND OF THE INVENTION

Chlorofluoroethanes such as 1,1,2,2-tetrachloro-1,2-difluoroethane (R-112), 1,1,2-trichloro-1,2,2-trifluoroethane (R-113) and the like have heretofore been used as solvent or detergent. These solvents have various excellent properties: they are nonflammable and low in toxicity to organisms; they can selectively solve fat, grease, wax and the like but do not attack plastics, rubber and like high molecular weight materials. However, R-113 and some chlorofluorocarbons are recently pointed out to be responsible for the destruction of the ozone layer in the stratosphere. The destruction of ozone layer will exert an adverse influene on the whole ecosystem including mankind. Thus, the use and production of chlorofluorocarbons which may contribute to the destruction of the ozone layer are now restricted under international agreements and it is expected the use and production thereof would be totally banned.

Various compounds and materials have been proposed as solvents which may replace chlorofluoro carbons. However, they have some defects and cannot fully satisfy the requirements as practical solvent. For example, chlorine containing solvents such as 1,1,1-trichloroethane, trichloroethylene, methylene chloride and the like attack high molecular materials such as plastics and are likely to cause environmental pollution. Alcohols and hydrocarbons are low in detergency and highly inflammable.

SUMMARY OF THE INVENTION

It is the primary object of the present invention to provide new compositions which can replace the conventional chlorofluoroethanes and which have excellent properties as solvent.

Other objects and feature of the invention will become apparent from the following description.

The present invention provides a solvent composition comprising a fluorine-containing alcohol (hereinafter referred to as Composition I).

The present invention provides also a solvent composition comprising a fluorine-containing alcohol and water (hereinafer referred to as Composition II).

A detergent for water soluble flux is generally required to have following properties; it exhibits high cleansing power; it is nonflammable; it has no or little influence on the environment, etc. However, no detergent has been found or proposed which fulfills all these requirements. Thus, purified water is used for eliminating flux because it is harmless, low in cost and does not affect the ecosystem although it has some inherent defects: it cannot wash away the flux retained in narrow spaces or apertures because of its high surface tension; it cannot be dried quickly; treatment of waste water is costly, etc.

We conducted extensive research to find a novel solvent which can effectively remove water soluble flux from substrate such as electronic and electric parts, etc. and found that a fluorine-containing alcohol can be a solvent suitable to substitute water.

We continued our research further and found that compositions comprising a fluorine-containing alcohol and a suitable additive or additives exhibit excellent properties required of general solvent or detergent.

Thus, the present invention provides a solvent composition comprising a fluorine-containing alcohol, an aliphatic alcohol having 1 to 4 carbons and water (hereinafter referred to as Composition III).

The present invention further provides a solvent composition comprising a fluorine-containing alcohol and a surfactant (hereinafter referred to as Composition IV).

DETAILED DESCRIPTION OF THE INVENTION

Each of Compositions I to IV of the invention will be described below in greater detail.

I. Composition I

The fluorine-containing alcohols to be used in Composition I preferably have 2 or more carbon atoms in the molecule wherein at least 2 of hydrogen atoms are substituted by fluorine atoms.

Examples of the fluorine-containing alcohols are as follows.

$$CF_3CH_2OH$$

$$H(CF_2CF_2)_aCH_2OH$$

$$H(CF_2\underset{\displaystyle CF_3}{\underset{|}{CF}})_bCH_2OH$$

$$H(CF_2CF_2)_c\underset{\displaystyle CH_3}{\underset{|}{CHOH}}$$

$$H(CF_2\underset{\displaystyle CF_3}{\underset{|}{CF}})_d\underset{\displaystyle CH_3}{\underset{|}{CHOH}}$$

$$F(CF_2CF_2)_eCH_2OH$$

$$F(CF_2\underset{\displaystyle CF_3}{\underset{|}{CF}})_fCH_2OH$$

$$F(CF_2CF_2)_gCH_2CH_2OH$$

$$F(CF_2\underset{\displaystyle CF_3}{\underset{|}{CF}})_hCH_2CH_2OH$$

$$F(CF_2CF_2)_iCH_2CH_2CH_2OH$$

$$F(CF_2\underset{\displaystyle CF_3}{\underset{|}{CF}})_jCH_2CH_2CH_2OH$$

In the above formulae of the fluorine-containing alcohols, a, c, e, g and i signify each an integer of 1 to 5 and b, d, f, h and j signify each an integer of 1 to 3.

Among these fluorine-containing alcohols, trifluoroethanol (hereinafter simply referred to as 3FE: boiling point = 77°C), tetrafluoropropanol (hereinafter simply referred to as 4FP: boiling point = 107°C) and pentafluoroethanol (hereinafter referred to as 5FP: boiling point = 81°C) are more preferable. 5FP is most preferable because it is nonflammable. These fluorine-containing alcohols can be used singly or in a mixture of at least two kinds.

Composition I can contain a conventional stabilizer to improve chemical stability for use under severe conditions. Examples of stabilizers are benzotriazol, 2-(2′-hydroxy-5′-methylphenyl)benzotriazol, chlorobenzotriazol and like triazols, etc. These stabilizers are usable singly or in a mixture of 2 or more kinds. Although variable with the kind of stabilizer, the amount thereof is usually about 0.1 to about 10% by weight, preferably about 0.2 to about 5% by weight based on the weight of Composition I.

II. Composition II

The fluorine-containing alcohols to be used in Composition II are the same as in Composition I. Fluorine-containing alcohols in Composition II can be used singly or at least 2 of them can be used. 3FE, 4FP and 5FP are more preferable and 5FP is most preferable also in Composition II.

Composition II comprises up to 80% by weight of water and not less than 20% by weight of at least one of fluorine-containing alcohols. Composition II preferably comprises up to 50% by weight of water and not less than 50% by weight of at least one of fluorine-containing alcohols. Composition II containing more than 50% by weight of fluorine-containing alcohol shows extremely high detergency. The cleansing power of Composition II gradually declines with the increase of water content. When the water content exceeds 80% by weight of Composition II, desired effect as detergent is greatly reduced, hence impractical.

A mixture of 4FP/water = 72.5/27.5 and a mixture of 5FP/water = 96/4 are particularly preferable because they form azeotropic compositions.

A mixture of 3FE and water does not form an azeotrope but behaves as an "azeotrope-like composition".

Stabilizers as indicated above can be incorporated into Composition II in a similar amount to improve stability thereof.

III. Composition III

The fluorine-containing alcohols to be used in Composition III are the same as those used in Composition I and can be used singly or in a mixture of at least two kinds. 3FE, 4FP and 5FP are more preferable and 5FP is most preferable.

Aliphatic alcohols to be used in Composition III are those having 1 to 4 carbon atoms such as methanol, ethanol, butanol, iso-propanol, etc.

Composition III usually comprises about 99 to about 60% by weight of the fluorine-containing alcohol, about 0.5 to about 20% by weight of the aliphatic alcohol and about 0.5 to about 20% by weight of water. Composition III preferably comprises about 91 to about 85% by weight of the fluorine-containing alcohol, about 1 to about 3% by weight of the aliphatic alcohol and about 8 to about 12% by weight of water. When the content of the aliphatic alcohol is more than 3% by weight, Composition III shows increasingly inflammability. In addition, when Composition III contains more than 3% of the aliphatic alcohol and more than 12% of water, it shows a slight tendency to separate into two liquid phases.

Stabilizers as given above can be used in Composition III in a similar amount as in Composition I.

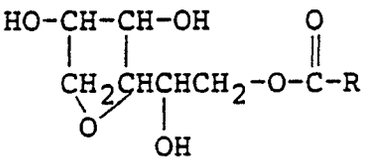
IV. Composition IV

The fluorine-containing alcohols to be used in Composition IV are the same as those in Composition I. They may be used singly or 2 or more of them can be used in mixture.

Examples of surfactants to be used in Composition IV are as shown below.

(1) Nonionic surfactants:

A ... Sorbitan aliphatic acid ester



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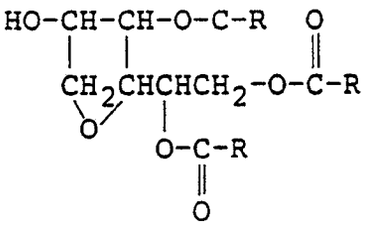
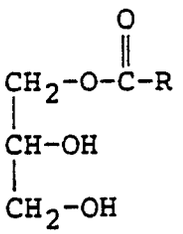


,

wherein R represents alkyl group having 7 to 18 carbons.

B ... Clycerol aliphatic acid ester



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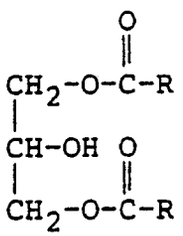
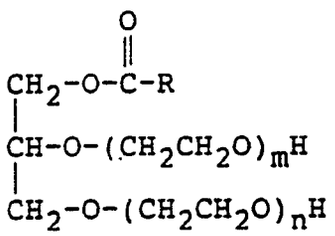


wherein R represents alkyl group having 7 to 18 carbons.

C ... Poly(oxyethylene)glycerol aliphatic acid ester



wherein R represents alkyl group having 7 to 18 carbon atoms and m + n = 5-15.

D ... Poly(oxyethylene)alkyl ether

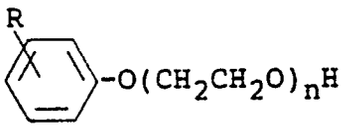
$R\text{-}O\text{-}(CH_2CH_2O)_nH$

wherein R represents alkyl group having 7 to 18 carbon atoms and n = 2 to 40.

E ... Poly(oxyethylene)poly(oxypropylene)alkyl ether

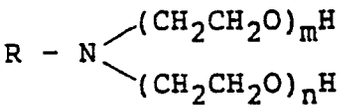
wherein R represents alkyl group having 7 to 18 carbon atoms and n = 1 to 20.

F ... Poly(oxyethylene)alkylphenyl ether



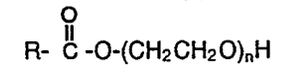
wheren R represents alkyl group having 8 to 9 carbon atoms and n = 2 to 30.

G ... Poly(oxyethylene)alkyl amine



wherein R represents alkyl group having 7 to 18 carbon atoms and m + n = 4-20.

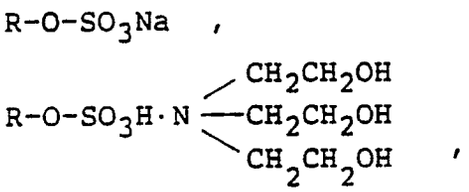
H ... Poly(ethylene glycol)aliphatic acid ester

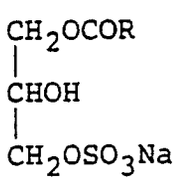


wherein R represents alkyl group having 7 to 18 carbon atoms and n = 1 to 55.

(2) Anionic surfactants:

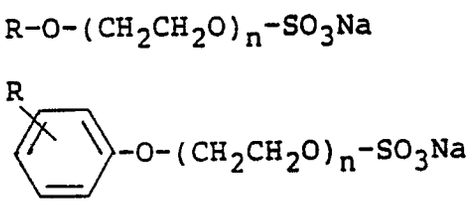
I ... Alkyl sulfate





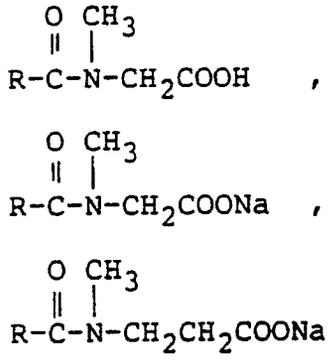
wherein R represents alkyl group having 7 to 18 carbon atoms.

J ... Poly(oxyethylene)alkyl ether sulfate



wherein R represent alkyl group having 7 to 18 carbon atoms and n = 2 to 4.

K ... N-acylamino acids and salts thereof



wherein R represents alkyl group having 7 to 18 carbon atoms.

Composition IV usually comprises about 85 to about 99.5% by weight of the fluorine-containing alcohol and about 15 to about 0.5% by weight of the surfactant. Composition IV preferably comprises about 90 to about 95% by weight of the fluorine-containing alcohol and about 10 to about 5% by weight of the surfactant. When the amount of the surfactant in Composition IV is less than 0.5% by weight, the detergency of the composition is not significantly improved. Use of the surfactant in an amount more than 15% by weight does not achieve more improved cleansing power and may require an additional step for removing the surfactant itself from the article washed.

Compositions I to IV of the invention does not deplete the stratospheric ozone layer.

Compositions I and II of the invention selectively dissolve away water base flux from the article made of metal, plastics, rubber, etc. to be washed while hardly attacking the article itself. Composition I and II are especially useful for eliminating the flux left on electronic and electric parts after soldering step.

Compositions I and II are more volatile than water and the drying time after cleansing of articles is greatly reduced.

Compositions I and II are safe to handle because they are nonflammable.

Compositions III and IV of the invention dissolve away and remove fat, grease, wax, paint, printing ink, etc. from the substrate made of metal, high molecular compounds such as plastics, rubber, etc. without damaging the substrate. Compositions III and IV are therefore very useful as solvent for eliminating grease and dirt from parts of electronic and electric devices, metal products, detergent for removing releasing agent from mold, etc.

Composition IV of the invention are nonflammable and easy to handle.

Composition IV of the invention containing a surfactant is very stable and easy to maintain the highly improved cleansing properties.

Examples

Given below are examples and comparison examples to clarify the feature of the invention.

Examples 1 to 5

Solvents according to the invention (Composition I and II) were tested for cleansing power against a water soluble flux.

Solvents used were as follows:

(I) 5FP
(II) 5FP/water = 94/6 (azeotrope)
(III) 4FP
(IV) 4FP/water = 72.5/27.5 (azeotrope)
(V) 3FE

To a test piece of printed circuit boad made of phenolic resin (50 mm x 50 mm) was applied a water soluble flux (trademark "solder-right TF-33B", product of Tamura Seisakusho, Japan) and soldering was carried out at 250° C for 5 seconds.

After test piece was cooled to room temperature and immersed in a solvent (300 ml) for 2 minutes, the amount of the flux remaining on the test piece was measured.

The results are given in Table 1 below.

Comparison Examples 1 to 4

The procedure of Example 1 were repeated except that the following solvents or detergents were used in place of 5FP.

Solvents or detergents used were as follows:

(VI) Water
(VII) Methanol
(VIII) Ethanol
(IX) 1,1,1-Trichloroethane

The results are given in Table 1 below.

Table 1

	Residual Flux (*)
(I)	A
(II)	A
(III)	A
(IV)	A
(V)	A
(VI)	B
(VII)	B
(VIII)	C
(IX)	D

* A : Residual flux less than 0.1%

B : Residual flux 0.1 to less than 0.5%

C : Residual flux 0.5 to less than 1%

D : Residual flux 1% or more

The results shown in Table 1 establish that the solvents of the invention exhibit high cleansing power.

Examples 6 to 13 and Comparison Examples 5 to 6

The combustibility of the solvents of the invention (Composition III) and the solvents of Comparison Examples was investigated.

The results are given in Table 2 below.

Solvent mixtures used were as follows:

(1) Comparison Example 5;

5FP/isopropanol (IPA) = 98/2

(2) Comparison Example 6;

5FP/ethanol (Et) = 98/2

The results are given in Table 2 below.

Table 2

	Solvent	Combustibility
Ex. 6	5FP/IPA/Water = 88/2/10	Nonflammable
7	5FP/IPA/Water = 85/3/12	Nonflammable
8	5FP/IPA/Water = 70/20/10	Inflammable
9	5FP/Et/Water = 88/2/10	Nonflammable
10	5FP/Et/Water = 85/3/12	Nonflammable
11	5FP/Et/Water = 70/20/10	Inflammable
12	3FE/IPA/Water = 88/2/10	Inflammable
13	4FP/IPA/Water = 88/2/10	Inflammable
Comp.Ex.5	5FP/IPA = 98/2	Nonflammable
6	5FP/Et = 98/2	Nonflammable

Further, the solvents were tested for cleansing power against water soluble flux following the procedure of Example 1 except that the fluxes (products of Tamura Seisakusho, Japan) indicated below were used.

* Flux (I) ..... trademark "F-AL-1"
* Flux (II) .... trademark "F-AL-4"
* Flux (III) ... trademark "Y-20"
* Flux (IV) .... trademark "HI-15"

The results are given in Table 3 below.

Table 3

	Residual Flux			
	(I)	(II)	(III)	(IV)
Ex. 6	B	A	B	B
7	B	A	B	B
8	A	A	B	A
9	B	A	C	B
10	B	A	B	B
11	A	A	B	A
12	B	B	B	B
13	B	B	B	B
Comp. Ex. 5	C	B	C	C
6	C	B	C	C

Next, the influence of solvents of Example 6 to 13 on plastics (weight increase by swelling of the material) was inspected.

Immediately after a test piece of plastics (5 mm x 50 mm x 2 mm) was immersed and kept in a solvent at 50° C for 1 hour, the test piece was weighed to find the weight increase. The results are given in Table 4 below.

The plastics used were as follows.

(a) ..... polystyrene
(b) ..... epoxy resin
(c) ..... polypropylene
(d) ..... phenolic resin

Table 4

	Weight increase			
Example	(a)	(b)	(c)	(d)
6	A	A	A	A
7	A	A	A	A
8	A	A	A	A
9	A	A	A	A
10	A	A	A	A
11	A	A	A	A
12	A	A	A	A
13	A	A	A	A

* A : Increase of less than 5%
B : Increase of 5% to 50%
C : Increase more than 50%

Examples 14 to 25

Solvents according to the invention (Composition IV) were tested for cleansing power against a water soluble flux.

The solvents used comprise 93% by weight of 3FE, 4FP or 5FP and 7% by weight of surfactant or surfactants shown below. When the solvent contains two kinds of surfactants, they are in an equal amount.

(1) Sorbitan monolaurate
(2) Glyceryl monostearate
(3) Poly(oxyethylene)glyceryl monostearate
(4) Poly(oxyethylene)lauryl ether
(5) Poly(oxyethylene)poly(oxypropylene) cetyl ether
(6) Poly(oxyethylene)nonylphenyl ether
(7) Poly(oxyethylene)stearyl amine
(8) Poly(ethylene glycol)monostearate
(9) Sodium dodecyl sulfate
(10) Poly(oxyethylene)lauryl ether sodium sulfate
(11) Sodium N-lauroylsarcosinate

The solvents were used in a similar manner as in Example 1 except that the flux used was a product of Tamura Seisakusho, Japan sold under the trademark of "F-AL-1".

The results are shown in Table 5 below.

Table 5

Example	Surfactant	Residual flux (%)(*) 3FE	4FP	5FP
14	(1)	B	B	B
15	(2)	A	B	B
16	(3)	B	B	B
17	(4)	B	A	A
18	(5)	B	B	A
19	(6)	B	B	B
20	(7)	B	B	B
21	(8)	A	B	B
22	(9)	B	B	B
23	(10)	B	B	B
24	(11)	B	B	A
25	(1)/(4)	B	B	B
26	(1)/(9)	B	B	A
27	(2)/(5)	B	B	B
28	(2)/(11)	B	A	A
29	(3)/(6)	B	B	B
30	(3)/(9)	B	B	B
31	(4)/(8)	B	B	B
32	(4)/(11)	A	A	B

Table 5 (continued)

Example	Surfactant	Residual flux (%)(*) 3FE	4FP	5FP
33	(5)/(10)	B	B	B
34	(6)/(7)	A	B	B
35	(6)/(11)	B	A	B
36	(7)/(8)	B	B	B
37	(8)/(10)	B	B	B
38	(10)/(11)	B	B	B
Comp.Ex.7		C	C	C

* Criteria are the same as in Table 1.

Claims

1 A solvent comprising a fluorine-containing alcohol.

2. A solvent according to claim 1 wherein the fluorine-containing alcohol has at least 2 carbon atoms in the molecule wherein at least 2 of hydrogen atoms are substituted by fluorine atoms.

3. A solvent composition comprising a fluorine-containing alcohol and water.

4. A solvent composition according to claim 3 wherein a solvent according to claim 1 wherein the fluorine-containing alcohol has at least 2 carbon atoms in the molecule wherein at least 2 of hydrogen atoms are substituted by fluorine atoms.

5. A solvent composition according to claim 3 which comprises up to 80% by weight of water and not less than 20% by weight of a fluorine-containing alcohol.

6. A solvent composition according to claim 5 which comprises up to 50% by weight of water and not less than 50% by weight of a fluorine-containing alcohol.

7. A solvent composition according to claim 6 which comprises 72.5% by weight of tetrafluoropropanol and 27.5% by weight of water.

8. A solvent composition according to claim 6 which comprises 96% by wight of pentafluoropropanol and 4% by weight of water.

9. A solvent composition comprising a fluorine-containing alcohol, an aliphatic alcohol and water.

10. A solvent composition according to claim 9 which comprises about 99 to about 60% by weight of fluorine-containing alcohol, about 0.5 to about 20% by weight of aliphatic alcohol and about 0.5 to about 20% by weight of water.

11. A solvent composition according to claim 10 which comprises about 91 to about 85% by weight of fluorine-containing alcohol, about 1 to about 3% by weight of aliphatic alcohol and about 8 to about 12% by weight of water.

12. A solvent composition comprising a fluorine-containing alcohol and a surfactant.

13. A solvent composition according to claim 12 which comprises about 85 to about 99.5% by weight of fluorine-containing alcohol and about 15 to about 0.5% by weight of surfactant.

14. A solvent composition according to claim 13 which comprises about 90 to about 95% by weight of fluorine-containing alcohol and about 10 to about 5% by weight of surfactant.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 90 10 7828

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	PATENT ABSTRACTS OF JAPAN vol. 9, no. 96 (C-278)(1819), 25 April 1985; & JP-A-59226086 (BAIKIN KOGYO K.K.) 19.12.1984 * abstract * ---	1-4,8	C 11 D 7/50 C 11 D 7/26 C 11 D 7/28
X	PATENT ABSTRACTS OF JAPAN vol. 7, no. 252 (C-194)(1397), 9 November 1983; & JP-A-58138770 (DAIKIN KOGYO K.K.) 17.08.1983 * abstract * ---	1,3,4	
A	idem ---	8	
X	PATENT ABSTRACTS OF JAPAN vol. 7, no. 232 (C-190)(1377), 14 October 1983; & JP-A-58122980 (SANYO DENKI K.K.) 21.07.1983 * abstract * ---	1,3,4	
A	idem ---	5,6	
X	PATENT ABSTRACTS OF JAPAN vol. 8, no. 138 (C-231)(1575), 27 June 1984; & JP-A-5948089 (DAIKIN KOGYO K.K.) 19.03.1984 * abstract * ---	1,2	TECHNICAL FIELDS SEARCHED (Int. Cl.5) C 11 D C 23 G
X	PATENT ABSTRACTS OF JAPAN vol. 11, no. 38 (C-401)(2485), 4 February 1987; & JP-A-61205236 (DAIKIN IND. LTD.) 11.09.1986 * abstract * ---	1	
X	CHEMICAL ABSTRACTS vol. 76, no. 16, 17 April 1972, page 68, abstract no. 87014y, Columbus, Ohio, US; M. SPEI: "Influence of detergents on low-angle x-ray diffraction patterns of alpha-keratin" & Appl. Polym. Symp., 1971, no. 18, part 1, pages 659-662 * abstract * --- -/-	1-4	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	31-07-1990	PELLI-WABLAT B

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
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EUROPEAN SEARCH REPORT

Application Number

EP 90 10 7828

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	CHEMICAL ABSTRACTS vol. 100, no. 22, 28 May 1984, page 45, abstract no. 175940s, Columbus, Ohio, US; & JP-A-58219236 (MATSUSHITA) 20.12.1983 * abstract * ---	1-4	
X	CHEMICAL ABSTRACTS vol. 106, no. 25, 22 June 1987, page 264, abstract no. 209643p, Columbus, Ohio, US; M. JOSEPH et al.: "Circular dichroism studies on a synthetic peptide corresponding to the membrane-spanning region of vesicular stomatis virus G protein and its fatty acyl derivative" & Biochim. Biophys. Acta, 1987, vol. 911, no. 2, page 231-237 * abstract * ---	1,9	
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X	CHEMICAL ABSTRACTS vol. 101, no. 19, 5 November 1984, page 746, abstract no. 171735b, Columbus, Ohio, US; M.V.R. RAO et al.: "Solution conformation of poly(L-lysyl-L-glutamic acid) and poly(L-lysyl-L-glutamine) & Int. J. Pept. Protein Res., 1984, vol. 24, no. 1, pages 48-54 * abstract * ---	1	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	idem ---	9	
A	EP-A-0 095 171 (DAIKIN KOGYO CO.) * claims; page 2, lines 35-39; page 3; page 4, lines 1-3 * -----	1-4	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	31-07-1990	PELLI-WABLAT B

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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