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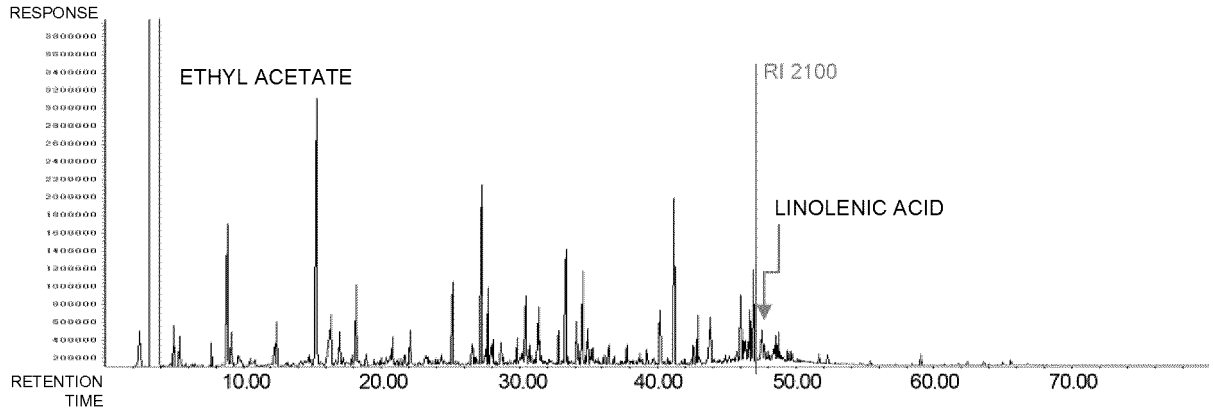
(54) TOBACCO EXTRACT, METHOD FOR PRODUCING TOBACCO EXTRACT, AND NON-COMBUSTION FLAVOR INHALER INCLUDING TOBACCO EXTRACT

(57) An object of the present invention is to provide a tobacco extract that, when utilized for a non-combustion type flavor inhaler, does not generate insoluble particles in a liquid mixture or the like, and can thereby suppress scorching at a heat source section and also suppress a change in smoke flavor. The present invention provides a tobacco extract produced by subjecting all or a portion of a tobacco plant body to steam distillation to obtain a fraction, subjecting the fraction to solvent extraction with a suitable organic solvent, and removing the organic solvent.

Fig. 1



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Description

TECHNICAL FIELD

[0001] The present invention relates to a tobacco extract, a method for producing the tobacco extract, and a non-combustion type flavor inhaler using the tobacco extract.

BACKGROUND ART

[0002] A tobacco extract or a method for extracting the tobacco extract is used for the purpose of ameliorating the flavor of a tobacco raw material or for the purpose of lowering the contents of components in a tobacco raw material. For example, a method for obtaining a tobacco raw material having satisfactory flavor by subjecting a leaf tobacco material to extraction with a low-polar solvent, further subjecting a residue after the extraction to extraction with a highly polar solvent, and pouring an extract extracted with the low-polar solvent back into a resultant residue (PTL1), a method for preparing a tobacco product containing a lowered amount of a phenol-based compound by subjecting a tobacco material to extraction with a solvent to provide an extract and a residue, treating the extract with phenol oxidase to lower the amount of the phenol-based compound, and combining a resultant with a tobacco residue (PTL2), a method of using as refined oil a fraction obtained by subjecting leaf tobacco to steam distillation and mixing the fraction with the other materials (PTL3), and the like are reported.

[0003] In addition, a method for preparing a distillate through distillation (such as distillation under reduced pressure) of a tobacco raw material (PTL4) is reported.

CITATION LIST

PATENT LITERATURE

[0004]

PTL 1: International Publication No. WO 2015/029977
PTL 2: Japanese Translation of PCT International Application Publication No. 2002-520005
PTL 3: Japanese Patent Publication No. 60-045909
PTL 4: Chinese Patent Laid-Open No. 104757703

SUMMARY OF INVENTION

TECHNICAL PROBLEM

[0005] When a tobacco extract is utilized for a non-combustion type flavor inhaler, there exist, as means of extraction, methods such as extraction with a solvent containing an organic solvent, water, or the like, which is a general tobacco-extracting method, high-pressure carbonic acid gas extraction, and steam distillation. However, when a tobacco extract obtained by any of these methods is applied to a non-combustion type flavor inhaler using propylene glycol, glycerin, or the like as a solvent, insoluble particles are generated and quality abnormality such as precipitation brings about a problem in the case where a component insoluble in, for example, propylene glycol or glycerin exists. These insoluble particles contain a large amount of a hydrophobic component containing some glucosides and higher fatty acids, and polymer compounds each having a different cyclic structure and substituent in the case where extraction means is solvent extraction and a volatile organic solvent or a halogen-based organic solvent is used as an extraction solvent. In addition, insoluble particles are also generated in a tobacco extract obtained by water extraction when the tobacco extract is applied to a non-combustion type flavor inhaler using propylene glycol, glycerin, or the like as a solvent. This is because a component insoluble in propylene glycol or glycerin, such as a protein or an inorganic salt, is contained in the water-extracted product. Further, a large amount of a hydrophobic component such as refined oil is contained in a resultant water-insoluble fraction in the case where extraction means is steam distillation. These hydrophobic components are considered to be a main cause for the insoluble particles to be generated when propylene glycol, glycerin, a mixture thereof, or the like is applied as a solvent.

[0006] In addition, in a heating type flavor inhaler which is one embodiment of the non-combustion type flavor inhaler, a mixed liquid is heated at a heat source section to be vaporized at a boiling point of a polyol (such as propylene glycol or glycerin) which is a solvent for the mixed liquid or at a temperature close to the boiling point of the mixed product of the polyols, and a hardly volatile component, which is not gasified at this temperature, retains at the heat source section and scorches to be a cause for a change in flavor.

[0007] Accordingly, a problem to be solved by the present invention is to provide a tobacco extract that, when utilized for a non-combustion type flavor inhaler, does not generate insoluble particles in a mixed liquid or the like, and can thereby suppress scorching at the heat source section and also suppress the change in flavor.

SOLUTION TO PROBLEM

[0008] The present inventors have advanced diligent studies in order to solve the problems to find that a tobacco extract produced by subjecting all or a portion of a tobacco plant body to steam distillation to obtain a fraction, subjecting the fraction to solvent extraction with a suitable organic solvent, and removing the organic solvent has low contents of a hydrophobic component and a hardly volatile component; and that the tobacco extract does not generate insoluble particles in a mixed liquid or the like when utilized for a non-combustion type flavor inhaler, and that the tobacco extract can suppress scorching at a heat source section and also suppress a change in flavor particularly when utilized for a mixed liquid to be used for a heating type flavor inhaler which is one embodiment of the non-combustion type flavor inhaler; and have thereby completed the present invention.

[0009] That is, the present invention includes, but not limited to, the following embodiments and aspects.

[1] A tobacco extract, wherein, when analysis by gas chromatography (a hydrogen flame ionization detector) is performed using a column comprising a stationary phase of 100% of dimethyl polysiloxane, a total peak area of a component group having a retention index (RI) of less than 2100 is 78% or more of a total peak area of the whole.
[2] The tobacco extract according to [1], wherein the total peak area of the component group having a retention index (RI) of less than 2100 is 81% or more of the total peak area of the whole.
[3] The tobacco extract according to [1] or [2], wherein a content of linolenic acid is 0.02% by weight or less of the whole amount of the tobacco extract.
[4] The tobacco extract according to any one of [1] to [3], wherein a peak area of linolenic acid is 0.01% by weight or less of the total peak area of the whole.
[5] The tobacco extract according to any one of [1] to [4], wherein a total peak area of at least one alkaloid selected from nicotine, nornicotine, myosmine, nicotyrine, nicotine-N-oxide, anabasine, anatabine, and cotinine decreases to 5% or less of the total peak area of the whole.
[6] The tobacco extract according to any one of [1] to [5], wherein, when analysis by gas chromatography (a hydrogen flame ionization detector) is performed using a column comprising a stationary phase of 100% of dimethyl polysiloxane, a peak of at least one alkaloid selected from the group consisting of nicotine, nornicotine, myosmine, nicotyrine, nicotine-N-oxide, anabasine, anatabine, and cotinine is not detected.
[7] The tobacco extract according to any one of [1] to [6], produced by a method comprising:

1) a step of subjecting all or a portion of a tobacco plant body to steam distillation, thereby obtaining a fraction;
2) a step of subjecting the obtained fraction to extraction with an organic solvent; and
3) a step of removing the organic solvent from an organic phase obtained by the extraction.

[8] The tobacco extract according to [7], wherein the organic solvent is ethyl acetate or diethyl ether.
[9] The tobacco extract according to [7] or [8], wherein the method further comprises a step of adjusting pH of the fraction to 6.0 or less before the step of subjecting the fraction to extraction with an organic solvent.
[10] The tobacco extract according to [9], wherein the pH of the fraction is 4.0 or less.
[11] The tobacco extract according to any one of [1] to [10], to be used for a non-combustion type flavor inhaler.
[12] The tobacco extract according to any one of [1] to [11], to be used for a heating type flavor inhaler.
[13] A non-combustion type flavor inhaler comprising the tobacco extract according to any one of [1] to [11].
[14] A heating type flavor inhaler comprising the tobacco extract according to any one of [1] to [12].
[15] A mixed liquid for a heating type flavor inhaler, the mixed liquid comprising the tobacco extract according to any one of [1] to [12].
[16] A heating type flavor inhaler comprising the mixed liquid for a heating type flavor inhaler according to [15].
[17] A method for producing the tobacco extract according to any one of [1] to [12], the method comprising:

1) a step of subjecting all or a portion of a tobacco plant body to steam distillation, thereby obtaining a fraction;
2) a step of subjecting the obtained fraction to extraction with an organic solvent; and
3) a step of removing the organic solvent from an organic phase obtained by the extraction.

[18] The method for producing the tobacco extract according to [17], wherein the organic solvent is ethyl acetate or diethyl ether.
[19] The method for producing the tobacco extract according to [17] or [18], further comprising a step of adjusting

pH of the fraction to 6.0 or less before the step of subjecting the fraction to extraction with an organic solvent.
[20] The method for producing the tobacco extract according to [19], wherein the pH of the fraction is 4.0 or less.
[21] Use of the tobacco extract according to any one of [1] to [11] for a non-combustion type flavor inhaler.
[22] Use of the tobacco extract according to any one of [1] to [12] for a heating type flavor inhaler.

ADVANTAGEOUS EFFECTS OF INVENTION

[0010] A tobacco extract of the present invention enables providing a non-combustion type flavor inhaler that does not cause a problem such as precipitation by a solvent-insoluble component, suppresses scorching at a heat source section, and also suppresses a change in flavor when the tobacco extract is utilized for the non-combustion type flavor inhaler. In addition, liquid-to-liquid transfer of a fraction with a solvent, the fraction obtained by steam distillation in an extraction step, while keeping the fraction acidic, enables further providing a tobacco extract in which an alkaloid content is reduced or an alkaloid is removed.

BRIEF DESCRIPTION OF DRAWINGS

[0011]

[Fig. 1] Fig. 1 is a chromatogram of a tobacco extract solution by gas chromatography (GC/FID), wherein the tobacco extract solution obtained in such a way that: a fraction obtained by subjecting a tobacco leaf to steam distillation is subjected to liquid-to-liquid transfer using ethyl acetate; an ethyl acetate layer at an upper portion is separated and extracted; ethyl acetate is then removed under reduced pressure; and further, a resultant dried and solidified product (tobacco extract component) is dissolved in ethyl acetate in a weight of 100 times the weight of the dried and solidified product. A vertical line in the chromatogram shows a retention time when a retention index (RI) is 2100. In addition, a peak of linolenic acid is shown by an arrow.
[Fig. 2] Fig. 2 is a chromatogram of a tobacco extract solution by GC/FID, wherein the tobacco extract solution obtained in such a way that: a fraction obtained by subjecting a tobacco leaf to steam distillation is subjected to liquid-to-liquid transfer using diethyl ether; a diethyl ether layer at an upper portion is separated and extracted; diethyl ether is then removed under reduced pressure; and further, a resultant dried and solidified product (tobacco extract component) is dissolved in diethyl ether in a weight of 100 times the weight of the dried and solidified product. A vertical line in the chromatogram shows a retention time when RI is 2100. In addition, a peak of linolenic acid is shown by an arrow.
[Fig. 3] Fig. 3 is a chromatogram of a tobacco extract solution by GC/FID, wherein the tobacco extract solution obtained in such a way that: a fraction obtained by subjecting a tobacco leaf to steam distillation is subjected to liquid-to-liquid transfer using chloroform; a chloroform layer at a lower portion is separated and extracted; chloroform is then removed under reduced pressure; and further, a resultant dried and solidified product (tobacco extract component) is dissolved in chloroform in a weight of 100 times the weight of the dried and solidified product. A vertical line in the chromatogram shows a retention time when RI is 2100. In addition, a peak of linolenic acid is shown by an arrow.
[Fig. 4] Fig. 4 is a chromatogram of a tobacco extract solution by GC/FID, wherein the tobacco extract solution obtained in such a way that: a fraction obtained by subjecting a tobacco leaf to steam distillation is subjected to liquid-to-liquid transfer using n-hexane; a n-hexane layer at an upper portion is separated and extracted; n-hexane is then removed under reduced pressure; and further, a resultant dried and solidified product (tobacco extract component) is dissolved in n-hexane in a weight of 100 times the weight of the dried and solidified product. A vertical line in the chromatogram shows a retention time when RI is 2100. In addition, a peak of linolenic acid is shown by an arrow.
[Fig. 5] Fig. 5 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to solvent immersion extraction with ethyl acetate. A vertical line in the chromatogram shows a retention time when RI is 2100.
[Fig. 6] Fig. 6 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to solvent immersion extraction with diethyl ether. A vertical line in the chromatogram shows a retention time when RI is 2100.
[Fig. 7] Fig. 7 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to solvent immersion extraction with chloroform. A vertical line in the chromatogram shows a retention time when RI is 2100.
[Fig. 8] Fig. 8 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to solvent immersion extraction with n-hexane. A vertical line in the chromatogram shows a retention time when RI is 2100.

[Fig. 9] Fig. 9 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to solvent immersion extraction with acetone. A vertical line in the chromatogram shows a retention time when RI is 2100.

[Fig. 10] Fig. 10 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to solvent immersion extraction with ethanol. A vertical line in the chromatogram shows a retention time when RI is 2100.

[Fig. 11] Fig. 11 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to solvent immersion extraction with methanol. A vertical line in the chromatogram shows a retention time when RI is 2100.

[Fig. 12] Fig. 12 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to warm water extraction to the warm water extraction solution, and then subjecting subjecting the warm water extraction solution to solvent extraction with ethyl acetate,. A vertical line in the chromatogram shows a retention time when RI is 2100.

[Fig. 13] Fig. 13 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to warm water extraction to the warm water extraction solution, and then subjecting the warm water extraction solution to solvent extraction with diethyl ether. A vertical line in the chromatogram shows a retention time when RI is 2100.

[Fig. 14] Fig. 14 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to warm water extraction to the warm water extraction solution, and then subjecting the warm water extraction solution to solvent extraction with chloroform. A vertical line in the chromatogram shows a retention time when RI is 2100.

[Fig. 15] Fig. 15 is a chromatogram of a tobacco extract by GC/FID, wherein the tobacco extract obtained by subjecting a tobacco leaf to warm water extraction to the warm water extraction solution, and then subjecting the warm water extraction solution to solvent extraction with n-hexane. A vertical line in the chromatogram shows a retention time when RI is 2100.

DESCRIPTION OF EMBODIMENTS

[0012] Hereinafter, embodiments and aspects of the present invention will specifically be described, but the present invention is not limited to these.

[0013] Chemical terms and technical terms which are used in relation to the present invention each have a meaning that is generally understood by a person skilled in the art unless otherwise defined in the present specification.

[0014] As one embodiment, the present invention provides a tobacco extract, wherein, when analysis by gas chromatography is performed using a column having a stationary phase of 100% of dimethyl polysiloxane, a total peak area of a component group having a retention index (RI) of less than 2100 is 78% or more of a total peak area of the whole. As a preferred embodiment, the analysis is performed by gas chromatography (hydrogen flame ionization detector).

[0015] The tobacco extract of the present invention is produced using all or a portion of a tobacco plant body as a raw material. The "portion of a tobacco plant body" refers to a part of a tobacco plant body, includes, for example, a leaf (including an upper leaf, a true leaf, an upper middle leaf, a middle leaf, and a lower leaf), a pinched part, an axillary bud, a stem, a trunk, a flower, a root, and a seed, or mixtures thereof, and is preferably a leaf, an axillary bud, and a trunk.

[0016] All or a portion of a tobacco plant body may be used as it is, or all or a portion of a tobacco plant body cut, pulverized, or ground into a slender piece-like, slurry-like, or fine particle-like product may be used. All or a portion of a tobacco plant body harvested from farmland or the like may be used as it is, all or a portion of a tobacco plant body obtained by being left to stand indoors or outdoors for a predetermined period of time to dissipate part of moisture may be used, or all or a portion of a tobacco plant body obtained by almost dissipating moisture with a drier (including a freeze drier) or the like may be used.

[0017] In the present specification, the "retention index (RI)" refers to an index relatively representing a retention ratio of a n-alkane to a compound as an object of analysis based on the numbers of carbon atoms of a linear hydrocarbon (n-alkane) in the analysis by gas chromatography. With respect to the retention index (RI), when a column having a predetermined stationary phase is used, the same compound theoretically has the same value even if the length of the column, the flow rate of a carrier gas, and the like are changed. The retention index (RI) is specifically calculated by the following formula.

[Formula 1]

$$RI = 100n + 100(t_x - t_n)/(t_{n+1} - t_n)$$

n: the numbers of carbon atoms of n-alkane appearing as peak immediately before peak of compound as object of analysis
tx: retention time of peak of compound as object of analysis
t_n: retention time of n-alkane appearing as peak immediately before peak of compound as object of analysis
t_{n+1}: retention time of n-alkane appearing as peak immediately after peak of compound as object of analysis

[0018] In the present specification, a value calculated using a n-alkane mixture in a range from n-hexane (C_6, RI: 600) to n-pentatriacontane (C_{35}, RI:3500) is used as the retention index (RI), but the n-alkane mixture which is used when the retention index (RI) is calculated is not limited to this.

[0019] In the present specification, the column which is used in gas chromatography has, for example, a non-polar or low-polar stationary phase, preferably a non-polar stationary phase.

[0020] As the column having a non-polar stationary phase, a column having a stationary phase of, for example, 100% of dimethyl polysiloxane can be used.

[0021] As the column having a stationary phase of 100% of dimethyl polysiloxane, for example, DB-1 (manufactured by Agilent Technologies, Inc.) can be used, but the column is not limited to this.

[0022] As the column having a low-polar stationary phase, a column having a stationary phase of, for example, 95% of dimethyl polysiloxane/5% of diphenyl siloxane can be used.

[0023] As the column having a stationary phase of 95% of dimethyl polysiloxane/5% of diphenyl siloxane, for example, DB-5 (manufactured by Agilent Technologies, Inc.) can be used, but the column is not limited to this.

[0024] In the present specification, the "hardly volatile component" or the "hardly volatile compound" refers to a substance which dissolves in a polyol to be a solvent and has a boiling point higher than the temperature at which the solvent gasifies, or a tobacco component or a compound having a characteristic that it does not form an azeotropic mixture with solvent and the compound per se does not gasify. Examples of the hardly volatile component or compound include glucosides, proteins, polymer compounds each having a different cyclic structure or substituent, long-chain fatty acids, and long-chain hydrocarbons.

[0025] The hardly volatile component or compound typically has a retention time equal to or longer than the retention time of n-henicosane (C_{21}, retention index (RI): 2100), that is, exhibits a retention index (RI) of 2100 or more in the analysis by gas chromatography using a non-polar or low-polar column.

[0026] Accordingly, in the present invention, the "component group having a retention index (RI) of less than 2100" means a component group having a retention time shorter than the retention time of n-henicosane (C_{21}, retention index (RI): 2100) and a component group which has a relatively low boiling point and gasifies at 200 to 240°C or less.

[0027] In the present invention, the total peak area of the "component group having a retention index (RI) of less than 2100" is, for example, 78% or more of the total peak area of the whole. The total peak area of the "component group having a retention index (RI) of less than 2100" is preferably 80% or more, more preferably 81% or more.

[0028] A "peak area" refers to an area of a part surrounded by a line segment obtained by connecting the spots where tangent lines at right and left inflection points with respect to the top of a waveform of a peak of a separated tobacco component or compound and a base line intersect on a chromatogram obtained by the analysis by gas chromatography.

[0029] The present invention provides as another embodiment the tobacco extract, wherein a content of linolenic acid is 0.02% by weight or less of the whole amount of the tobacco extract.

[0030] Linolenic acid is a linear unsaturated fatty acid having three double bonds and having a molecular formula represented by $C_{18}H_{30}O_2$, is a hardly volatile component having high hydrophobicity, and is insoluble in a relatively high-polar organic solvent, such as propylene glycol, which is used as a solvent for a mixed liquid for a non-combustion type flavor inhaler.

[0031] In the present invention, the content of linolenic acid is, for example, 0.02% by weight or less, preferably 0.01% by weight or less, and more preferably 0.006% by weight or less of the whole amount of the tobacco extract.

[0032] The present invention provides as another embodiment the tobacco extract wherein a total peak area of at least one alkaloid selected from the group consisting of nicotine, nornicotine, myosmine, nicotyrine, nicotine-N-oxide, anabasine, anatabine, and cotinine decreases to 5% or less of the total the peak area of the whole.

[0033] In the present invention, the total peak area of the alkaloid is, for example, 5% or less, preferably 3.5% or less, and more preferably 2.5% or less of the total peak area of the whole, and most preferably, a peak of the alkaloid is not detected.

[0034] The present invention provides as another embodiment the tobacco extract to be used for a non-combustion type flavor inhaler.

[0035] The "non-combustion type flavor inhaler" refers to a tobacco product that generates aerosol by means such as heating an aerosol-generating article held at an aerosol-generating-article-holding part with a heat source without combusting the aerosol-generating article, or atomizing an aerosol-generating article held at an aerosol-generating-article-holding part by ultrasonic waves, thereby delivering the aerosol through a mouthpiece member. Examples of the aerosol-generating article include, but not limited to, a liquid or solid aerosol source containing a flavor component

contained in a tobacco raw material, and tobacco materials such as a compressed tobacco pellet and a tobacco powder.

[0036] The non-combustion type flavor inhaler may be a tobacco product that produces aerosol (may be flavor-imparted aerosol) which a user inhales, and examples thereof include a heating type flavor inhaler of a type that uses a liquid aerosol source, a heating type flavor inhaler of a type that uses aerosol which is generated by heating tobacco as an aerosol source, and a non-heating type flavor inhaler for inhaling flavor of tobacco without heating the tobacco.

[0037] In the present invention, the non-combustion type flavor inhaler is not particularly limited, but preferably, a heating type flavor inhaler of a type that uses a liquid aerosol source can be used.

[0038] Examples of the liquid aerosol source which is used for the non-combustion type flavor inhaler include a mixed liquid for a non-combustion type flavor inhaler, the mixed liquid containing a flavor component in a polyol such as glycerin or propylene glycol, the flavor component contained in a tobacco raw material.

[0039] In the present specification, the mixed liquid for a non-combustion type flavor inhaler can also contain the tobacco extract of the present invention as a material that provides a flavor component contained in a tobacco raw material.

[0040] The present invention provides as another aspect a method for producing the tobacco extract, the method comprising: 1) a step of subjecting all or a portion of a tobacco plant body to steam distillation, thereby obtaining a fraction; 2) a step of subjecting the obtained fraction to extraction with an organic solvent; and 3) a step of removing the organic solvent from an organic phase obtained by the extraction.

[0041] In the present invention, the variety of a tobacco (a plant of the genus Nicotiana) plant body which is used in steam distillation is not particularly limited. For example, flue-cured, burley, and orient can be used.

[0042] In the present invention, all of a tobacco plant body which is used in steam distillation can be used without choosing a site, or a portion of the tobacco plant body can be used by choosing only a desired site. In addition, all or a portion of a tobacco plant body which is used in steam distillation may be utilized for steam distillation without being cut, or may be utilized for steam distillation after being cut appropriately into a desired size, for example, cut into a 2 cm square or so. All or a portion of a tobacco plant body pulverized or ground by an ordinary method into a slender piece-like, slurry-like, or fine particle-like product may be utilized for steam distillation. All or a portion of a tobacco plant body, which is used in steam distillation, harvested from farmland or the like may be used as it is, all or a portion of a tobacco plant body obtained by being left to stand indoors or outdoors for a predetermined period of time to dissipate part of moisture may be used, or all or a portion of a tobacco plant body obtained by almost dissipating moisture with a drier (including a freeze drier) or the like may be used.

[0043] The "steam distillation" is a method of distilling a compound having a low vapor pressure and a high boiling point at a temperature equal to or lower than the boiling point. By continuously introducing heated steam into a distillation container in which an object sample containing a target compound is placed, the distillation container is made into a heated state such that the container is filled with heated steam, and the target compound is thereby distilled out with steam.

[0044] In the present specification, the "liquid-to-liquid transfer" or "liquid-to-liquid extraction" refers to organic solvent extraction of a target compound from a sample in a liquid phase. Specifically, the "liquid-to-liquid transfer" or "liquid-to-liquid extraction" is a method of extracting a compound using an organic solvent as a solvent. By adding an organic solvent to a sample in a liquid phase, the sample containing a target compound, to form two separated phases, and shaking the two separated phases, the target compound is extracted into the added organic solvent utilizing the difference in distribution to the two phases.

[0045] In the present specification, the "solvent immersion extraction" refers to organic solvent extraction of a target compound from a sample in a solid phase. By immersing a sample in a solid phase, the sample containing a target compound, the target compound is extracted into the organic solvent.

[0046] In the present invention, the organic solvent which is used in the extraction step with an organic solvent may be a solvent that is not miscible with water, for example, ethyl acetate, diethyl ether, propyl acetate, isopropyl acetate, and the like, and preferably ethyl acetate or diethyl ether can be used.

[0047] In the present invention, a method which is used in the step of removing the organic solvent from the organic phase obtained by extraction is not particularly limited. Methods such as solvent removal under reduced pressure with an evaporator or the like, solvent removal by heating with a heater, and solvent removal by blasting a purge gas can be used.

[0048] The present invention provides as another embodiment the method for producing the tobacco extract, the method further comprising a step of adjusting pH of the fraction in steam distillation to 3.0 to 6.0. By adjusting the pH of the fraction in steam distillation to the above-described pH, the equilibrium between the molecular form and ionic form of the alkaloid is shifted to the ionic form to inhibit the extraction into the organic solvent, and a tobacco extract in which an alkaloid content is reduced or an alkaloid is removed can thereby be produced.

[0049] In the present invention, the pH of the fraction in steam distillation is adjusted to, for example, 6.0 or less, preferably 5.0 or less, and more preferably 4.0 or less.

[0050] In addition, in the present invention, the pH of the fraction in steam distillation is adjusted to, for example, 1.0 or more, preferably 2.0 or more, and more preferably 3.0 or more.

[0051] In the present specification, the "warm water extraction" refers to solvent extraction using heated water as a

solvent.

[0052] In the warm water extraction, the condition is, for example, such that water is heated to 50 to 60°C to make the temperature of water about 40°C to 45°C when a raw material is immersed therein.

[0053] The present invention provides as another aspect the tobacco extract produced by the above-described production method.

[0054] The tobacco extract is produced using all or a portion of a tobacco plant body as a raw material, the contents of respective components originally contained greatly vary in some cases depending on the seed, the part to be used, and the growth environment of the tobacco plant body which is used as a raw material, therefore it can be considered that the specification of the tobacco extract of the present invention using the contents of components in the extract as indexes is not practical in some cases, but when the above-described production method is used, it is expected that the tobacco extract of the present invention in which a hardly volatile component decreases and/or the alkaloid content is reduced or the alkaloid is removed, and which has constant quality to some extent is obtained, and this tobacco extract exhibits the effects of the present invention.

EXAMPLE

[0055] Hereinafter, Examples of the present invention will be described. The technical scope of the present invention is not limited by these Examples.

Example 1: Production of Tobacco Product by Steam Distillation and Organic Solvent Extraction

[0056] A tobacco product was produced using a United States flue-cured tobacco leaf according to the following procedure.

(1) Steam Distillation

[0057] In a steam distillation apparatus (Herb Oil Maker (Large Type) manufactured by TOKYOSEISAKUSHO Co., Ltd.) the inside of which had been cleaned for about 1 hour after pouring water therein, 2 L of water was poured and heated (250°C) with a heater. After the water was boiled, the United States flue-cured tobacco leaf (500 g) was placed therein to start distillation. Thereafter, distillation was continued, and 1000 mL of a fraction was collected during distillation for 2 hours. The obtained fraction was transferred into a beaker to wrap the beaker and was stored in a 5°C refrigerator overnight.

(2) Organic Solvent Extraction from Fraction

[0058] As an organic solvent, ethyl acetate, diethyl ether, chloroform, and n-hexane were used.

[0059] In a 1 L volume separatory funnel, 500 mL of the fraction (including oil floated on the fraction) was poured, 200 mL of the organic solvent and 30 g of sodium chloride were added, and a resultant mixture was shaken. After the aqueous phase was removed, 500 ml of the rest of the fraction and 30 g of sodium chloride were added to the organic phase, and a resultant mixture was shaken (1000 mL of the fraction in total was subjected to extraction). After the aqueous phase was removed, the organic phase was transferred into a 300 mL Erlenmeyer flask, 20 g of anhydrous sodium sulfate was added, and a resultant mixture was shaken calmly and was then left to stand at room temperature for 30 minutes to perform dehydration.

(3) Removal of Organic Solvent from Organic Phase

[0060] The organic phase after dehydration was filtrated with a filter paper (manufactured by Advantec Toyo Kaisha, Ltd., No. 2, 150 mm) on which a small amount of anhydrous sodium sulfate was placed, and solvent removal under reduced pressure was performed in a 40°C water bath until the filtrate was evaporated to dryness with a rotary evaporator. Further, 5 mL of ethanol having a purity of 99% (Wako Guaranteed Reagent) was added, and the residual organic solvent was completely removed under a reduced pressure condition with a rotary evaporator to obtain 17 mg of a tobacco extract as a dried and solidified product. The organic solvent (the same as used for extraction) in a weight of 100 times the weight of the extract was added to dissolve the extract. A dissolved product was filtrated with a filter (PTFE) having a pore diameter of 0.45 μm to be used as a tobacco extract solution.

(4) Analysis of Tobacco Extract Solution by Gas Chromatography (GC/FID)

[0061] Analysis of the tobacco extract solution obtained in (3) was performed under the following condition.

Gas Chromatography (GC/FID)

[0062]

Apparatus: 7890A GC manufactured by Agilent Technologies, Inc.
Column: DB-1 (manufactured by Agilent Technologies, Inc.), inner diameter 0.25 mm × length 30 m, film thickness 0.25 μm
Injection volume: 1 μL
Injection mode: split (10:1)
Inlet temperature: 290°C
Septum purge flow rate: 5 mL/min
Carrier gas: helium (He)
Column flow rate: 1 mL/min
Oven: 40°C (3 min) - 4°C/min - 290°C (10 min) (75.5 min in total)
Detector: hydrogen flame ionization detector (FID)

Detector temperature: 300°C
Hydrogen (H_2) flow rate: 40 mL/min
Air flow rate: 450 mL/min
Make-up gas (He) flow rate: 1 mL/min

[0063] Chromatograms obtained by the analysis of tobacco extracts by GC/FID in the cases where solvent extraction was performed with ethyl acetate, diethyl ether, chloroform, and n-hexane are shown in Figs. 1 to 4, respectively. In addition, in the case where peaks on a chromatogram are classified by numerical ranges of retention indexes (RI) of 700 to 2099, 2100 to 2299, and 2300 or more, the proportion (%) of the total peak area of the component group in each range in the total peak area of the whole, and the number of peaks of the component group in each range, and the proportion (%) of the number of peaks of the component group in each range in the total number of peaks of the whole are shown in Table 1 below. Further, the contents (% by weight) of linolenic acid and of phytol, which are each a highly hydrophobic, hardly volatile compound, in the whole amount of tobacco extract are shown in Table 2 below.

[Table 1]

	GC (FID) peak area (%)			Number of peaks (number of peaks (%))		
Solvent	RI:700-2099	RI:2100-2299	RI: 2300 or more	RI:700-2099	RI:2100-2299	RI: 2300 or more
Ethyl acetate	86.3*	8.1	5.6	230(82.4)	16(5.7)	33(11.8)
Diethyl ether	82.0*	8.0	10.0	261(84.2)	17(5.5)	32(10.3)
Chloroform	77.9	11.3	10.8	258(82.2)	19(6.1)	37(11.8)
n-Hexane	75.7	11.9	12.3	229(78.4)	18(6.2)	45(15.4)
*: there is a significant difference when $p<0.05$ in a goodness-of-fit test using						

[Table 2]

	Content (% by weight)	
Solvent	Linolenic acid	Phytol
Ethyl acetate	0.003	0.016
Diethyl ether	0.006	0.024
Chloroform	0.023	0.082
n-Hexane	0.074	0.056

[0064] As it is clear from the chromatograms in Figs. 1 to 4, it was found that the peaks of the component group which has a relatively high boiling point and is hardly volatile, the component group having a retention index (RI) of 2100 or more, are small, and the peak of linolenic acid, which is an index of a highly hydrophobic, hardly volatile compound, is

also small in any of the cases where the fraction after steam distillation was subjected to solvent extraction with ethyl acetate, diethyl ether, chloroform, or n-hexane. Particularly in the case where solvent extraction was performed with either ethyl acetate or diethyl ether, the peaks of the component group having a RI of 2100 or more are smaller, and the peak of linolenic acid was smaller even as compared to the cases where solvent extraction was performed with chloroform or n-hexane. As it is clear from Table 1, in the cases where solvent extraction was performed with ethyl acetate or diethyl ether, it was found that the proportion of the peak area of the component group which has a relatively low boiling point and easily volatilizes, the component group having a RI of less than 2100, exceeds 78%. In addition, as it is clear from Table 2, it was found that the content of linolenic acid is about 0.1% by weight or less in any of the cases where solvent extraction was performed with ethyl acetate, diethyl ether, chloroform, or n-hexane. Particularly in the cases where solvent extraction was performed with ethyl acetate or diethyl ether, the content of linolenic acid was about 0.02% by weight or less, and was extremely lower even as compared to the cases where solvent extraction was performed with chloroform or n-hexane. Accordingly, it was demonstrated that a tobacco extract in which a hardly volatile component is significantly decreased is obtained by subjecting the fraction after steam distillation to solvent extraction with ethyl acetate or diethyl ether.

Example 2: Test of Solubility of Tobacco Extract in Propylene Glycol

[0065] Solubility of the tobacco extracts each obtained as a dried and solidified product in Example 1 in propylene glycol, which is also used as a solvent for a heating type flavor inhaler, was tested. Specifically, a predetermined amount of propylene glycol was added, a resultant mixture was warmed to 42°C and returned back to room temperature, particle size distributions of 150 μm (primary particle) and 1500 μm (aggregate) were then measured with a particle size distribution analyzer (LV-950A manufactured by HORIBA, Ltd.), and thereby the existence or non-existence of insoluble particles was checked.

[0066] The tobacco extracts each produced by subjecting the fraction after steam distillation to liquid-to-liquid extraction with ethyl acetate or diethyl ether dissolved by adding propylene glycol in a weight of 4 times the weight of the extracts, and insoluble particles of 0.45 μm or larger were not recognized.

[0067] On the other hand, the tobacco extracts each produced by subjecting the fraction after steam distillation to solvent extraction with chloroform or n-hexane did not dissolve completely because insoluble particles having a diameter of 0.45 μm or larger were recognized when propylene glycol in a weight of 4 times the weight of the extracts was added. When propylene glycol was added stepwise, insoluble particles of 0.45 μm or larger were recognized in the tobacco extract produced by subjecting the fraction after steam distillation to solvent extraction with chloroform even in the case where propylene glycol in a weight of 20 times the weight of the extract was added, and in the tobacco extract produced by subjecting the fraction after steam distillation to solvent extraction with n-hexane even in the case where propylene glycol in a weight of 50 times the weight of the extract was added. It is considered that a hydrophobic component in the tobacco leaf was extracted and such insoluble particles were thereby generated.

[0068] Accordingly, it was demonstrated that a tobacco extract in which a hydrophobic component in the tobacco leaf is significantly decreased and which easily dissolves into propylene glycol is obtained by subjecting the fraction after steam distillation to solvent extraction with ethyl acetate or diethyl ether.

Comparative Example 1: Production of Tobacco Extract by Organic Solvent Extraction and Test of Solubility in Propylene Glycol

(1) Production of Tobacco Extract by Organic Solvent Extraction

[0069] As an organic solvent, ethyl acetate, diethyl ether, chloroform, n-hexane, acetone, ethanol, and methanol were used.

[0070] A United States flue-cured tobacco leaf (10 g), which is the same as in Example 1, cut into a 2 cm square or so was placed in a 500 mL volume conical beaker, and 100 mL of the organic solvent was added to immerse the tobacco leaf. Shaking extraction was performed at room temperature for 1 hour (solvent immersion extraction), and the organic solvent after extraction was filtrated with a filter paper (manufactured by Advantec Toyo Kaisha, Ltd., No. 2, 150 mm) on which a small amount of anhydrous sodium sulfate was placed, and solvent removal under reduced pressure was performed in a 40°C water bath until the filtrate was evaporated to dryness with a rotary evaporator to obtain 9.3 mg to 16.0 mg of a tobacco extract as a dried and solidified product. The organic solvent used for extracting the extract in an amount of 1 mL was added to make an analysis sample, and analysis by GC/FID was performed under the same condition as in Example 1(4). However, the injection mode was split (50:1).

[0071] Chromatograms obtained by the analysis by GC/FID are shown in Figs. 5 to 11. In addition, in the case where peaks on a chromatogram are classified by numerical ranges of retention indexes (RI) of 700 to 2099, 2100 to 2299, and 2300 or more, the proportion (%) of the total peak area of the component group in each range in the total peak area

of the whole, and the number of peaks of the component group in each range, and the proportion (%) of the number of peaks of the component group in each range in the total number of peaks of the whole are shown in Table 3 below.

[Table 3]

	GC (FID) peak area (%)			Number of peaks (number of peaks (%))		
Solvent	RI:700-2099	RI:2100-2299	RI: 2300 or more	RI:700-2099	RI:2100-2299	RI: 2300 or more
Ethyl acetate	27.4	18.5	54.1	139(60.7)	12(5.2)	78(34.1)
Diethyl ether	23.1	16.2	60.6	89(53.0)	13(7.7)	66(39.3)
Chloroform	52.8	13.3	33.9	152(61.5)	15(6.1)	80(32.4)
n-Hexane	24.4	19.7	55.9	63(43.4)	17(11.7)	65(44.8)
Acetone	37.1	18.3	44.6	93(56.4)	10(6.1)	62(37.6)
Ethanol	48.7	20.9	30.4	132(73.7)	7(3.9)	40(22.3)
Methanol	73.2	9.5	17.3	35(17.4)	9(4.5)	157(78.1)

[0072] As it is clear from the chromatograms in Figs. 5 to 11, it was found that in all of the organic solvents, the peaks of the component group which has a relatively high boiling point and is hardly volatile, the component group having a RI of 2100 or more, are larger than the results in Example 1 (Figs. 1 to 4) as compared to the component group which has a relatively low boiling point and easily volatilizes, the component group having a RI of less than 2100. As it is clear from Table 3, it was found that in all of the organic solvents, the proportion of the peak area of the component group which has a relatively low boiling point and easily volatilizes, the component group having a RI of less than 2100, is lower as compared to the results in Example 1 (Table 1), as low as 70% or slightly higher at most (in the case of methanol), and the number of peaks is also smaller. On the other hand, it was also found that there is a tendency that the proportion of the peak area of the component group which has a relatively high boiling point and is hardly volatile, the component group having a RI of 2100 or more, was higher as compared to the results in Example 1 (Table 1), and the number of peaks was also larger. Accordingly, it was demonstrated that a tobacco extract in which a hardly volatile component decreases effectively is not obtained by only organic solvent extraction.

Test of Solubility of Tobacco Extract in Propylene Glycol

[0073] When the same test as in Example 2 was performed for the tobacco extracts produced by only organic solvent extraction, insoluble particles of 0.45 μm or larger were recognized in all of the tobacco extracts even in the case where propylene glycol in a weight of 50 times the weight of the extracts was added. Accordingly, it was demonstrated that a tobacco extract in which a hydrophobic component in the tobacco leaf is significantly decreased, and which easily dissolves into propylene glycol is not obtained by only organic solvent extraction.

Comparative Example 2: Production of Tobacco Extract by Warm Water Extraction and Organic Solvent Extraction

(1) Warm Water Extraction

[0074] Distilled water was heated to prepare 500 mL of about 60°C warm water, and a United States flue-cured tobacco leaf (50 g) cut into a 2 cm square or so was placed therein to perform extraction for 10 minutes while keeping the temperature in such a way as to be 40 to 45°C under stirring with a stirrer. The warm water after extraction was cooled to room temperature in a 6°C refrigerator and was filtrated with a filter paper (ADVANTEC, No. 5A) to remove a solid, thereby obtaining 470 mL of a warm water extract solution.

(2) Organic Solvent Extraction from Warm Water Extract Solution

[0075] As an organic solvent, ethyl acetate, diethyl ether, chloroform, and n-hexane were used.

[0076] In a 500 mL volume separatory funnel, 100 mL of the warm water extract solution was poured, 50 mL of the organic solvent and 20 g of sodium chloride were added, and a resultant mixture was shaken for 5 minutes. After the mixture was shaken, the mixture was left to stand in a 6°C refrigerator for 3 hours to ascertain that the organic phase and the aqueous phase were separated satisfactory, and the aqueous phase was removed. After the aqueous phase was removed, the organic phase was filtrated with a filter paper (manufactured by Advantec Toyo Kaisha, Ltd., No. 2,

150 mm) on which a small amount of anhydrous sodium sulfate was placed, and solvent removal under reduced pressure was performed in a 40°C water bath until the filtrate was evaporated to dryness with a rotary evaporator to obtain 8.2 to 12.5 mg of a tobacco extract as a dried and solidified product. The organic solvent used for extracting the extract in an amount of 1 mL was added to make an analysis sample, and analysis by GC/FID was performed under the same condition as in Example 1(4).

[0077] Chromatograms obtained by the analysis by GC/FID are shown in Figs. 12 to 15. In addition, in the case where peaks on a chromatogram are classified by numerical ranges of retention indexes (RI) of 700 to 2099, 2100 to 2299, and 2300 or more, the proportion (%) of the total peak area of the component group in each range in the total peak area of the whole, and the number of peaks of the component group in each range, and the proportion (%) of the number of peaks of the component group in each range in the total number of peaks of the whole are shown in Table 4 below.

[Table 4]

	GC (FID) peak area (%)			Number of peaks (number of peaks (%))		
Solvent	RI:700-2099	RI:2100-2299	RI: 2300 or more	RI:700-2099	RI:2100-2299	RI: 2300 or more
Ethyl acetate	54.9	14.6	30.5	133(78.2)	15(8.8)	22(12.9)
Diethyl ether	40.4	15.4	44.2	30(44.1)	14(20.6)	24(35.3)
Chloroform	50.2	12.0	37.8	116(82.9)	11(7.9)	13(9.3)
n-Hexane	34.8	17.6	47.6	46(63.9)	14(19.4)	12(16.7)

[0078] As it is clear from the chromatograms in Figs. 12 to 15, it was found in the case where the warm water extract solution was subjected to organic solvent extraction, the peaks of all of the detected component groups are smaller than the results in Example 1 (Figs. 1 to 4) in all of the organic solvents. In addition, it was found that the peaks of the component group which has a relatively high boiling point and is hardly volatile, the component group having a RI of 2100 or more, are larger than the results in Example 1 (Figs. 1 to 4) as compared to the peaks of the component group which has a relatively low boiling point and easily volatilizes, the component group having a RI of less than 2100. As it is clear from Table 4, it was found that in all of the organic solvents, the proportion of the peak area of the component group which has a relatively low boiling point and easily volatilizes, the component group having a RI of less than 2100, is lower as compared to the results in Example 1 (Table 1), as low as 55% or slightly lower at most (in the case of ethyl acetate), and the number of peaks is also smaller. On the other hand, it was also found that the proportion of the peak area of the component group which has a relatively high boiling point and is hardly volatile, the component group having a RI of 2100 or more, is higher as compared to the results in Example 1 (Table 1), and the number of peaks is larger. Accordingly, it was demonstrated that a tobacco extract in which a hardly volatile component decreases effectively is not obtained, and on top of that, loss of components in the process of extraction is large by warm water extraction and organic solvent extraction.

Example 3: Production of Tobacco Extract in Which Alkaloid Content Is Reduced or Alkaloid Is Removed

[0079] A tobacco extract in which the alkaloid content was reduced or the alkaloid was removed was produced using a United States flue-cured tobacco leaf according to the following procedure. That is, by adjusting the pH of the fraction in steam distillation, the fraction obtained in the same manner as in Example 1, the equilibrium between the molecular form and ionic form of the alkaloid is shifted to the ionic form to inhibit the extraction into the organic solvent, and thereby the alkaloid content was reduced or the alkaloid was removed.

(1) Steam Distillation

[0080] A flue-cured tobacco leaf was subjected to steam distillation in the same manner as in Example 1 (1) to obtain 1000 mL of a fraction.

(2) Organic Solvent Extraction from Fraction

[0081] As an organic solvent, diethyl ether was used.

[0082] To the fraction obtained in (1), 1.5 M sulfuric acid was gradually added under stirring to adjust pH to 6.0, 5.0, 4.0, or 3.0. The fraction whose pH was adjusted was subjected to solvent extraction with diethyl ether according to the same procedure as in Example 1 (2) to dehydrate the diethyl ether phase.

(3) Removal of Organic Solvent from Organic Phase

[0083] Diethyl glycol was removed from the diethyl ether phase after dehydration according to the same procedure as in Example 1 (3) to obtain 18 mg of a tobacco extract as a solidified and dried product. The tobacco extract was dissolved by adding diethyl ether in a weight of 100 times the weight of the extract. A dissolved product was filtrated with a filter (PTFE) having a pore diameter of 0.45 μm to be used as a tobacco extract solution.

(4) Analysis of Tobacco Extract by Gas Chromatography (GC/FID)

[0084] Analysis of the tobacco extract obtained in (3) by GC/FID was performed under the same condition as in Example 1 (4).

[0085] The proportion (%) of the peak area of each alkaloid of nicotine, nornicotine, myosmine, nicotyrine, nicotine-N-oxide, anabasine, anatabine, and cotinine in the total peak area of the whole on the chromatograms obtained by analysis of the tobacco extract by GC/FID in the cases where the fraction was subjected to solvent extraction with diethyl ether at a pH of 6.0, 5.0, 4.0, or 3.0 is shown in Table 5 below.

[Table 5]

		GC (FID) peak area (%)			
Alkaloid	RI	pH6.0	pH5.0	pH4.0	pH3.0
Nicotine	1348	2.4	1.6	ND	ND
Nornicotine	1362	0.1	0.1	ND	ND
Myosmine	1368	0.4	0.2	ND	ND
Nicotyrine	1428	0.2	0.1	ND	ND
Nicotine-N-oxide	1429	ND	ND	ND	ND
Anabasine	1432	ND	ND	ND	ND
Anatabine	1456	ND	ND	ND	ND
Cotinine	1622	ND	ND	ND	ND
ND: not detected (ascertained by both of analysis by GC and analysis by GC/MS)					

[0086] As it is clear from Table 5, it was found that nicotine (RI: 1348) which was detected in the cases where solvent extraction with diethyl ether was performed at a pH of 6.0 or 5.0 was not detected at a pH of 4.0 or 3.0. The total proportion of the peak area of each alkaloid was 3.1% in the case where solvent extraction was performed at a pH of 6.0, 2.0% in the case where solvent extraction was performed at a pH of 5.0, and a peak of the above-described alkaloids was not detected in the cases where solvent extraction was performed at a pH of 4.0 or 3.0. Accordingly, it was demonstrated that the alkaloid content was reduced at a pH of 6.0 and 5.0, and the alkaloid was removed at a pH of 4.0 and 3.0.

Example 4: Production of Flue-cured Tobacco Extract by Steam Distillation and Organic Solvent Extraction and Test of Solubility in Propylene Glycol

[0087] A tobacco extract was produced using a French flue-cured tobacco according to the following procedure.

(1) The French flue-cured tobacco (958.5 g) was subjected to steam distillation by heating (setting: 280°C, actual temperature: 126 to 128°C) a steam distillation apparatus (Herb Oil Maker (for 3 kg) manufactured by TOKYOSEI-SAKUSHO Co., Ltd.) with 3 L of water to obtain 2.3 L of a fraction. The dropping speed of the fraction was 9.5 to 10 ml/min.

(2) The obtained fraction was divided into 500 ml of proportions, the proportions were each subjected to extraction with any one of various organic solvents (ethyl acetate, diethyl ether, chloroform, and n-hexane) according to the same procedures as in Example 1 (2) and (3) to remove the organic solvent, thereby obtaining 187 to 280 mg of a tobacco extract as a solidified and dried product.

[0088] The solubility of the above-described tobacco extracts in propylene glycol was tested. Specifically, the existence or non-existence of insoluble particles was checked visually by adding a predetermined amount of propylene glycol. In

addition, the particle size distribution was measured with a wet type particle analyzer (LA-960 manufactured by HORIBA, Ltd.) to check the existence or non-existence of insoluble particles having a particle diameter of 0.45 μm or larger. The results are shown in Tables 6 and 7 below.

[Table 6]

[0089]

Table 6. Check of insoluble particles by visual observation

	Weight of propylene glycol added (based on weight of tobacco extract)		
Organic solvent used for extraction	Equal amount	20 times	50 times
Ethyl acetate	X	○	○
Diethyl ether	X	○	○
Chloroform	X	X	○
n-Hexane	X	X	X
○: insoluble particles do not exist, x: insoluble particles exist			

[Table 7]

[0090]

Table 7. Check of insoluble particles having a particle diameter of 0.45 μm or larger by wet type particle analyzer

	Weight of propylene glycol added (based on weight of tobacco extract)		
Organic solvent used for extraction	Equal amount	20 times	50 times
Ethyl acetate	X	○	○
Diethyl ether	X	○	○
Chloroform	X	X	○
n-Hexane	X	X	X
○: insoluble particles do not exist, x: insoluble particles exist			

[0091] As it is clear from Tables 6 and 7, the flue-cured tobacco extracts produced by subjecting the fraction after steam distillation to solvent extraction with ethyl acetate or diethyl ether dissolved by adding propylene glycol in a weight of 20 times and of 50 times the weight of the extracts.

[0092] On the other hand, the flue-cured tobacco extract produced by subjecting the fraction after steam distillation to solvent extraction with chloroform did not dissolve and insoluble particles having a particle diameter of 0.45 μm or larger were recognized in the case where propylene glycol in a weight of 20 times the weight of the extract was added, and the flue-cured tobacco extract produced by subjecting the fraction after steam distillation to solvent extraction with chloroform dissolved finally by adding propylene glycol in a weight of 50 times the weight of the extract. In addition, the flue-cured tobacco extract produced by subjecting the fraction after steam distillation to solvent extraction with n-hexane did not dissolve, and insoluble particles of 0.45 μm or larger were recognized even in the case where propylene glycol in a weight of 50 times the weight of the extract was added.

[0093] Accordingly, it was demonstrated that a tobacco extract in which a hydrophobic component in a tobacco leaf is significantly decreased and which easily dissolves in propylene glycol is obtained also from French flue-cured by subjecting the fraction after steam distillation to solvent extraction with ethyl acetate or diethyl ether.

INDUSTRIAL APPLICABILITY

[0094] The present invention can provide a tobacco extract in which the amount of a hardly volatile component in a tobacco leaf is significantly decreased, a method for producing the tobacco extract, a non-combustion type flavor inhaler comprising the tobacco extract, and further, a heating type flavor inhaler which is one embodiment of the non-combustion

type flavor inhaler comprising the non-combustion type flavor inhaler. In addition, the present invention can further provide a tobacco extract in which an alkaloid content is reduced or an alkaloid is removed, a method for producing the tobacco extract, a non-combustion type flavor inhaler comprising the tobacco extract, and, further, a heating type flavor inhaler which is one embodiment of the non-combustion type flavor inhaler comprising the tobacco extract.

Claims

1. A tobacco extract, wherein, when analysis by gas chromatography (a hydrogen flame ionization detector) is performed using a column comprising a stationary phase of 100% of dimethyl polysiloxane, a total peak area of a component group having a retention index (RI) of less than 2100 is 78% or more of a total peak area of the whole.

2. The tobacco extract according to claim 1, wherein the total peak area of the component group having a retention index (RI) of less than 2100 is 81% or more of the total peak area of the whole.

3. The tobacco extract according to claim 1 or 2, wherein a content of linolenic acid is 0.02% by weight or less of the whole amount of the tobacco extract.

4. The tobacco extract according to any one of claims 1 to 3, wherein a peak area of linolenic acid is 0.01% by weight or less of the total peak area of the whole.

5. The tobacco extract according to any one of claims 1 to 4, wherein, when analysis by gas chromatography (a hydrogen flame ionization detector) is performed using a column comprising a stationary phase of 100% of dimethyl polysiloxane, a total peak area of at least one alkaloid selected from nicotine, nornicotine, myosmine, nicotyrine, nicotine-N-oxide, anabasine, anatabine, and cotinine decreases to 5% or less of the total peak area of the whole

6. The tobacco extract according to any one of claims 1 to 5, wherein a peak of at least one alkaloid selected from the group consisting of nicotine, nornicotine, myosmine, nicotyrine, nicotine-N-oxide, anabasine, anatabine, and cotinine is not detected.

7. The tobacco extract according to any one of claims 1 to 6, produced by a method comprising:

    1) a step of subjecting all or a portion of a tobacco plant body to steam distillation, thereby obtaining a fraction;
    2) a step of subjecting the obtained fraction to extraction with an organic solvent; and
    3) a step of removing the organic solvent from an organic phase obtained by the extraction.

8. The tobacco extract according to claim 7, wherein the organic solvent is ethyl acetate or diethyl ether.

9. The tobacco extract according to claim 7 or 8, wherein the method further comprises a step of adjusting pH of the fraction to 6.0 or less before the step of subjecting the fraction to extraction with an organic solvent.

10. The tobacco extract according to claim 9, wherein the pH of the fraction is 4.0 or less.

11. The tobacco extract according to any one of claims 1 to 10, to be used for a non-combustion type flavor inhaler.

12. The tobacco extract according to any one of claims 1 to 11, to be used for a heating type flavor inhaler.

13. A non-combustion type flavor inhaler comprising the tobacco extract according to any one of claims 1 to 11.

14. A heating type flavor inhaler comprising the tobacco extract according to any one of claims 1 to 12.

15. A mixed liquid for a heating type flavor inhaler, the mixed liquid comprising the tobacco extract according to any one of claims 1 to 12.

16. A heating type flavor inhaler comprising the mixed liquid for a heating type flavor inhaler according to claim 15.

17. A method for producing the tobacco extract according to any one of claims 1 to 12, the method comprising:

1) a step of subjecting all or a portion of a tobacco plant body to steam distillation, thereby obtaining a fraction;
2) a step of subjecting the obtained fraction to extraction with an organic solvent; and
3) a step of removing the organic solvent from an organic phase obtained by the extraction.

18. The method for producing the tobacco extract according to claim 17, wherein the organic solvent is ethyl acetate or diethyl ether.

19. The method for producing the tobacco extract according to claim 17 or 18, further comprising a step of adjusting pH of the fraction to 6.0 or less before the step of subjecting the fraction to extraction with an organic solvent.

20. The method for producing the tobacco extract according to claim 19, wherein the pH of the fraction is 4.0 or less.

21. Use of the tobacco extract according to any one of claims 1 to 11 for a non-combustion type flavor inhaler.

22. Use of the tobacco extract according to any one of claims 1 to 12 for a heating type flavor inhaler.

Fig. 1

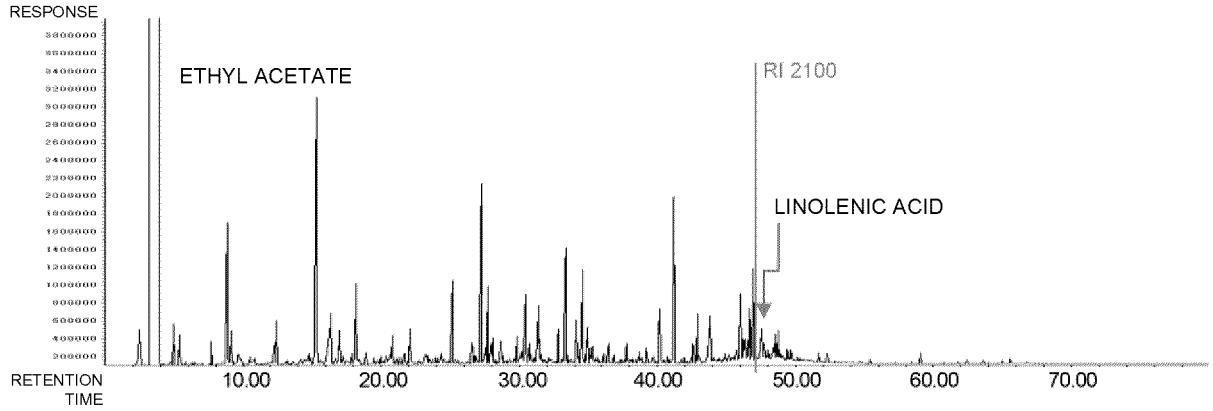
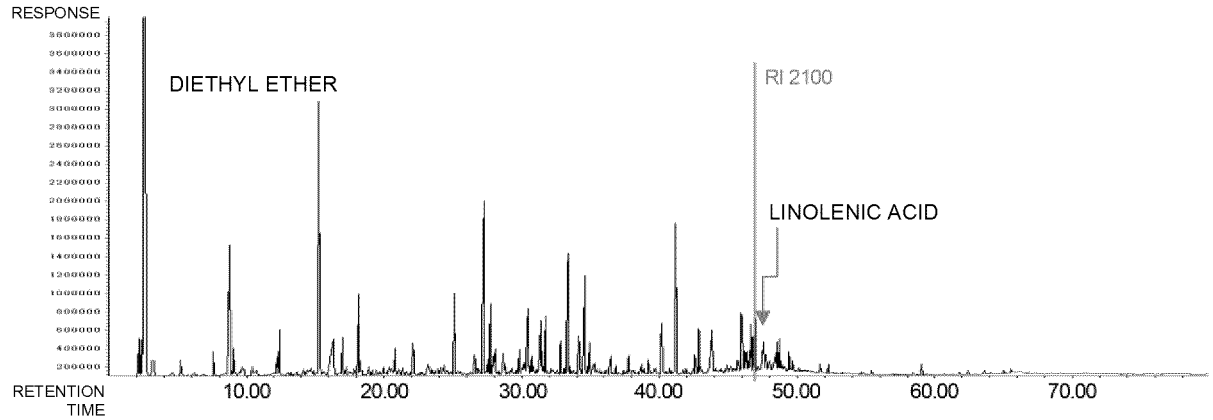


Fig. 2



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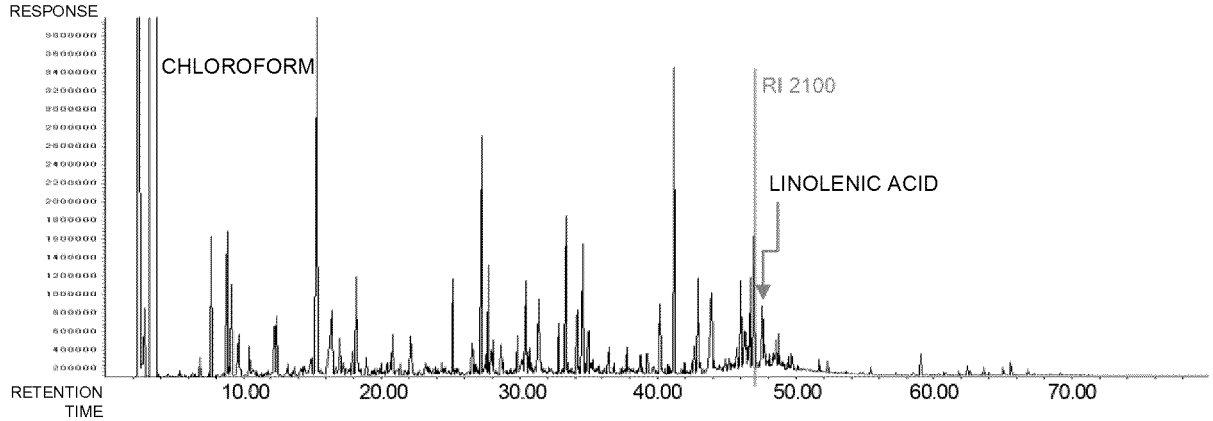

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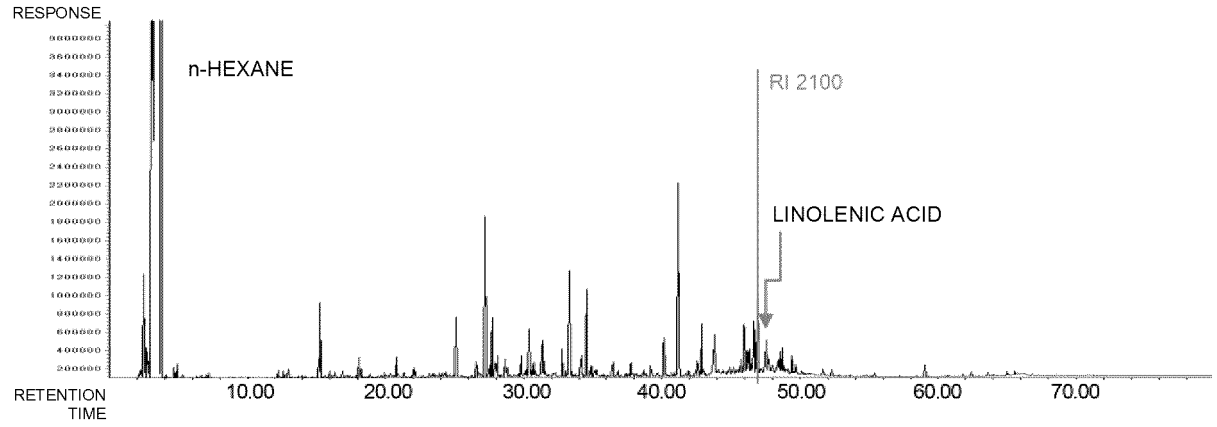

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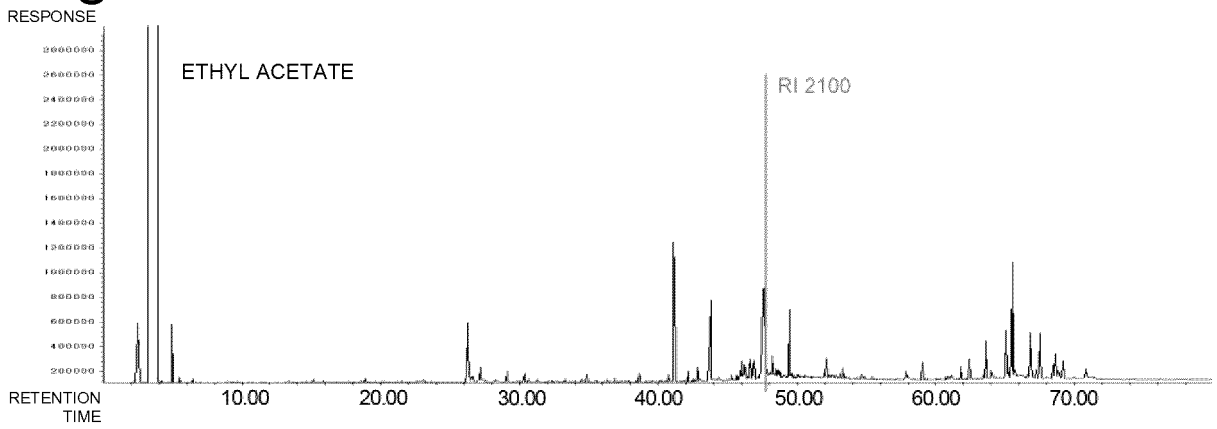

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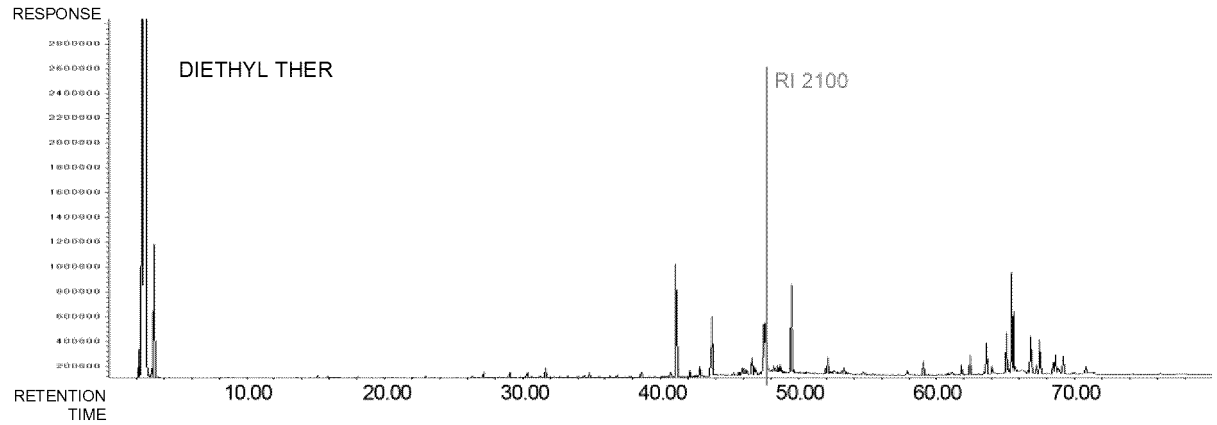

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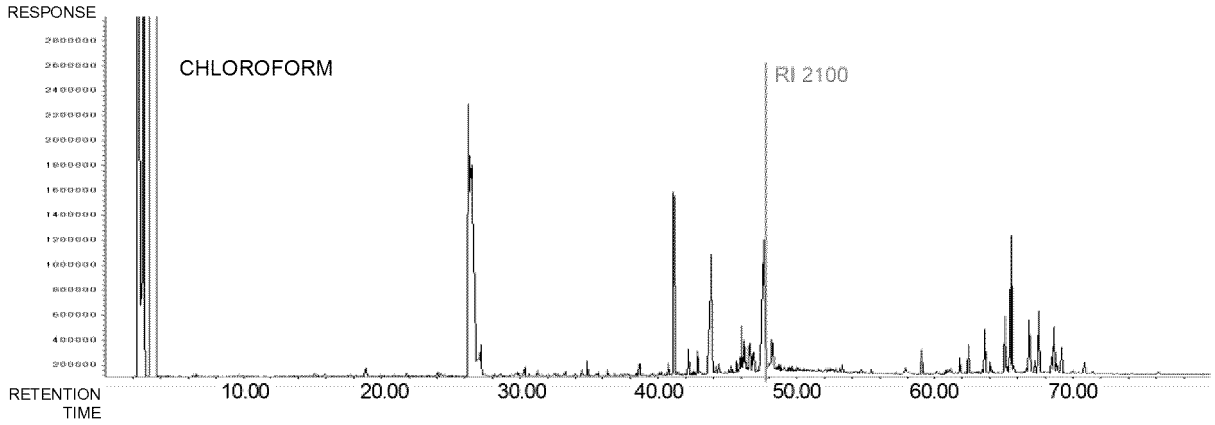


Fig. 8

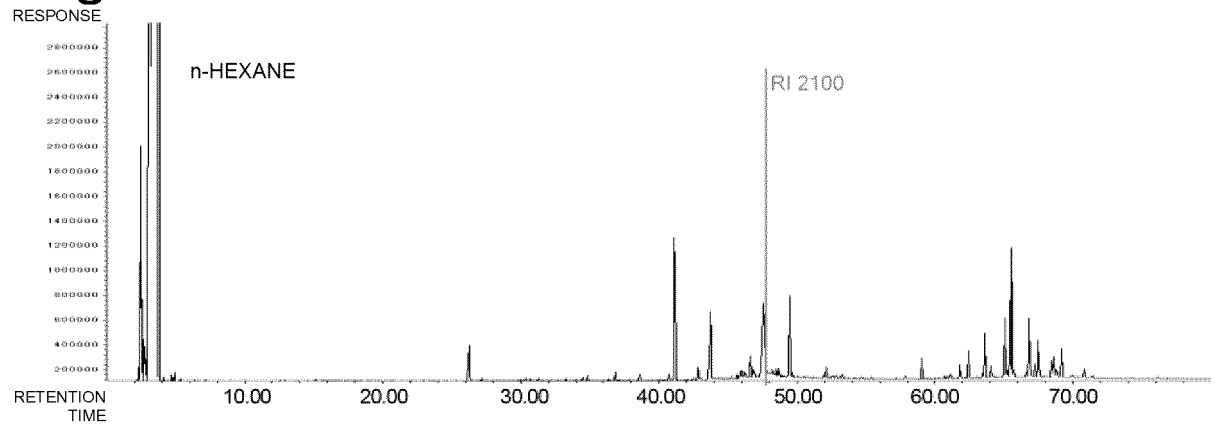


Fig. 9

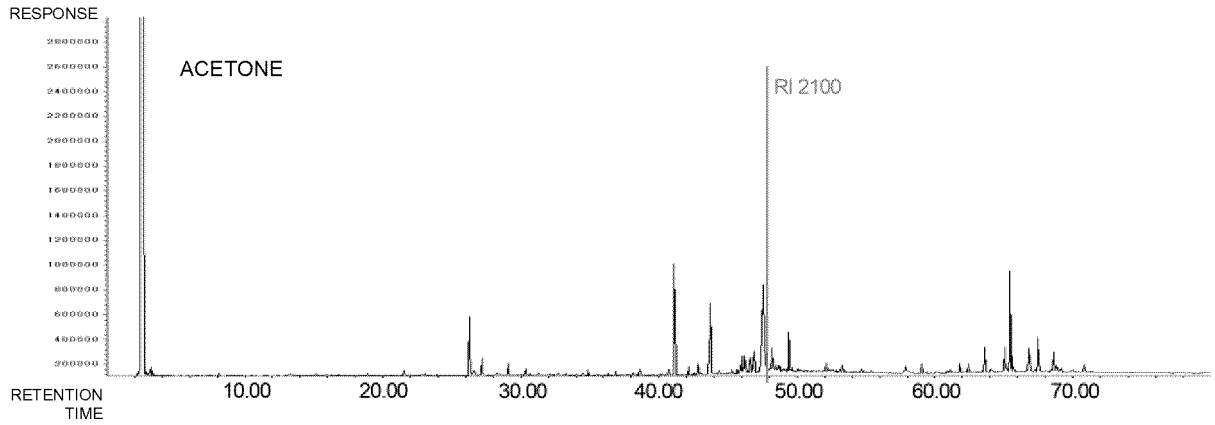


Fig. 10

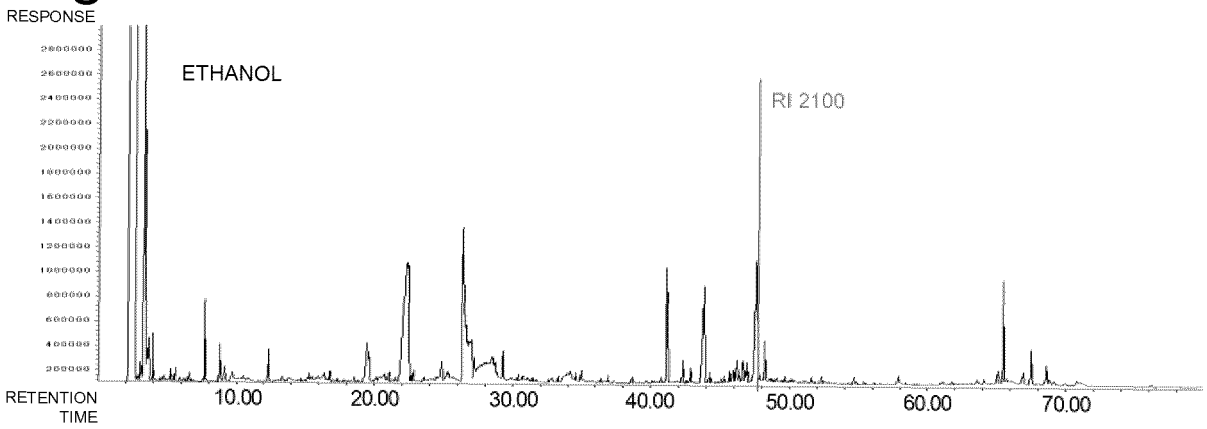


Fig. 11

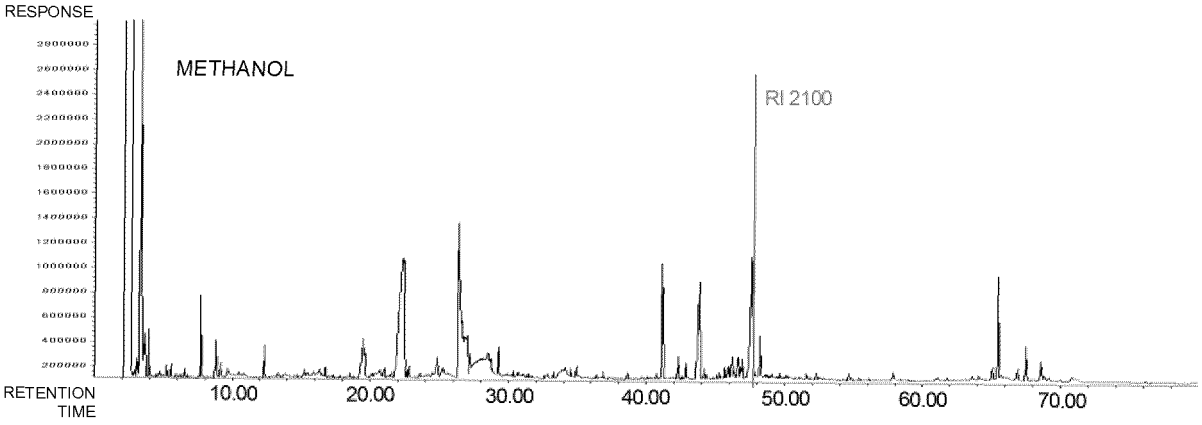


Fig. 12

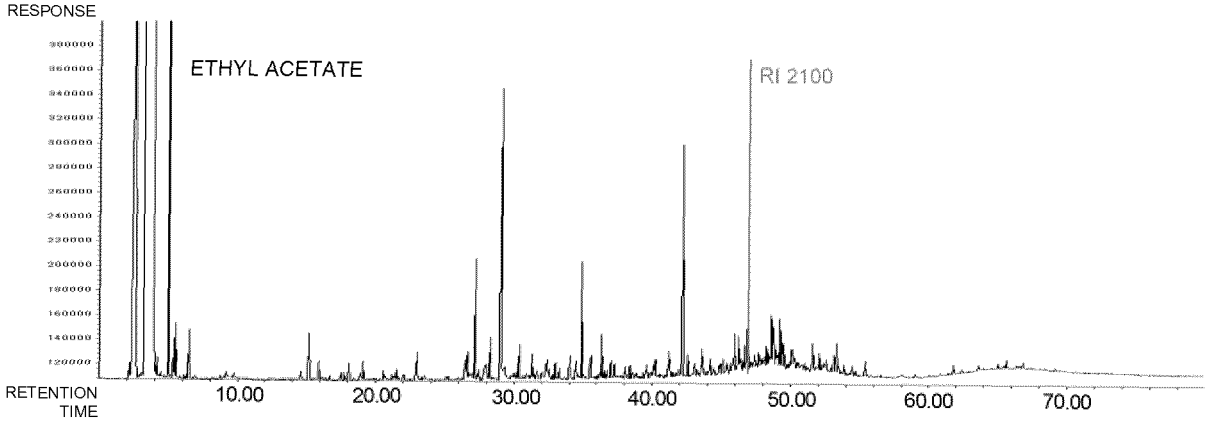


Fig. 13

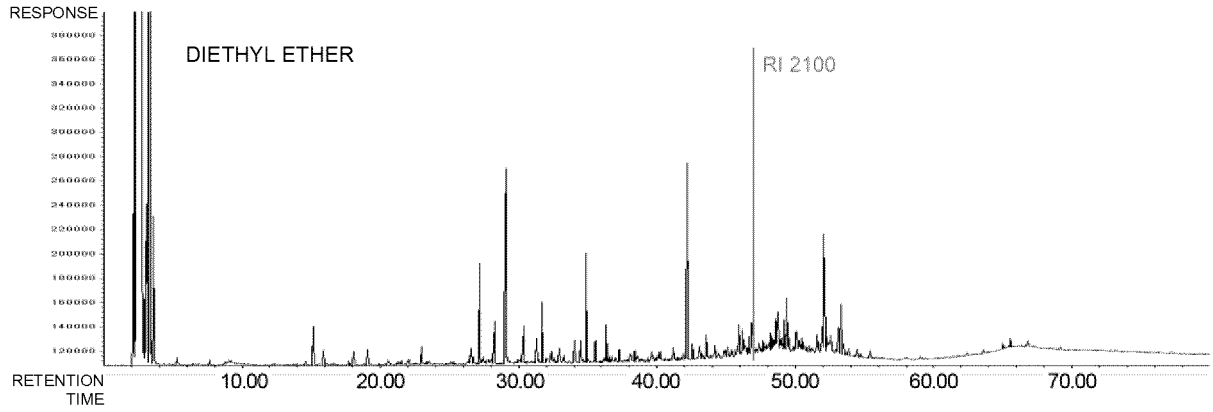


Fig. 14

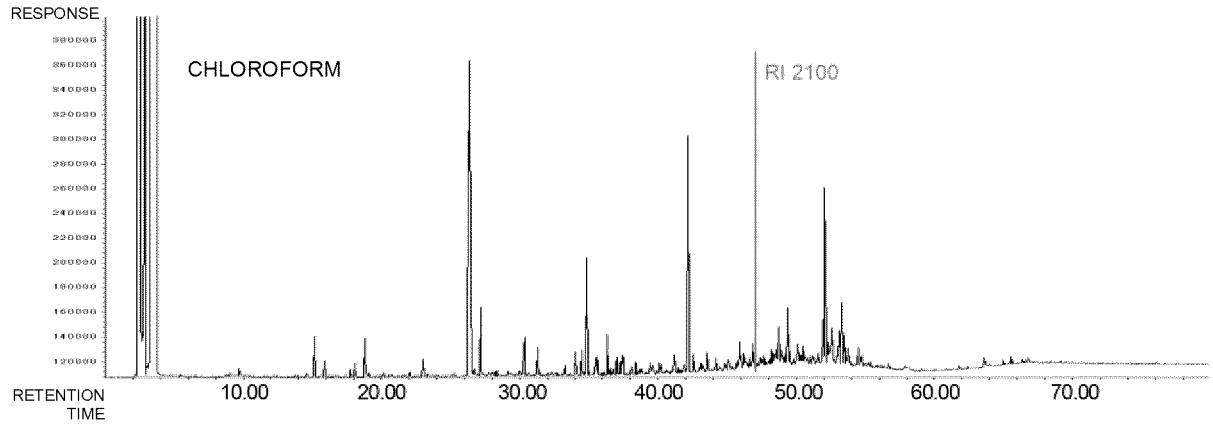
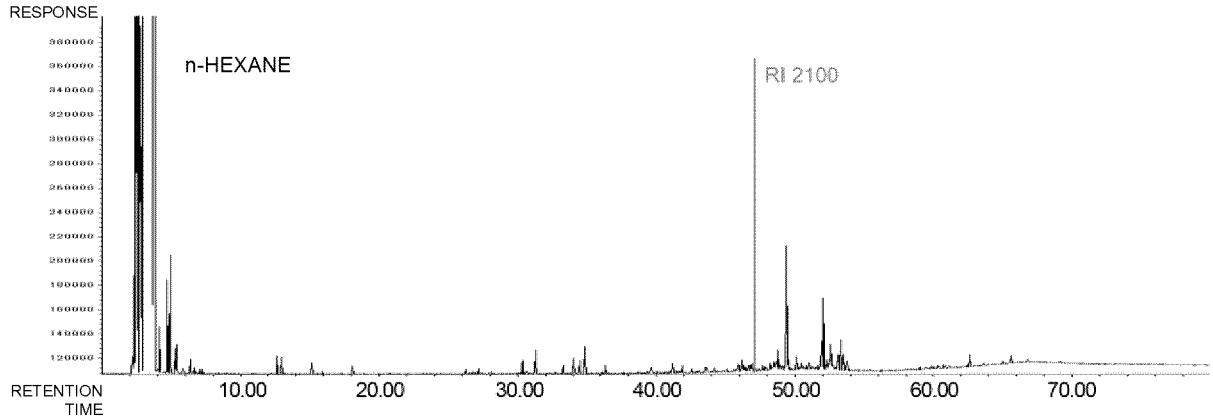


Fig. 15



INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2018/047439

A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl. A24B15/26(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
Int.Cl. A24B15/26

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan	1922-1996
Published unexamined utility model applications of Japan	1971-2019
Registered utility model specifications of Japan	1996-2019
Published registered utility model applications of Japan	1994-2019

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 11-37986 A (NKK CORP.) 12 February 1999, entire text, all drawings (Family: none)	1-22
A	CN 106324130 A (NAT TABACCO QUALITY SUPERVISION & INSPECTION CT) 11 January 2017, entire text, all drawings (Family: none)	1-22

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:
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"P" document published prior to the international filing date but later than the priority date claimed
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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
11 March 2019 (11.03.2019)	26 March 2019 (26.03.2019)

Name and mailing address of the ISA/	Authorized officer
Japan Patent Office 3-4-3, Kasumigaseki, Chiyoda-ku, Tokyo 100-8915, Japan	Telephone No.

Form PCT/ISA/210 (second sheet) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- WO 2015029977 A **[0004]**
- JP 2002520005 W **[0004]**
- JP 60045909 A **[0004]**
- CN 104757703 **[0004]**