

DESCRIPTION

TITLE OF INVENTION:

Composition Containing Hexafluoropropene Trimer

5 TECHNICAL FIELD

[0001] The present disclosure relates to a composition containing a hexafluoropropene trimer.

BACKGROUND ART

10 [0002] Hexafluoropropene (HFP) trimers are used in the form of a composition and the like (PTL 1).

CITATION LIST

PATENT LITERATURE

[0003] PTL 1: WO2018/172919

SUMMARY OF INVENTION

15 TECHNICAL PROBLEM

[0004] The present disclosure aims at providing a composition containing a hexafluoropropene trimer, as a novel mixture with excellent stability.

SOLUTION TO PROBLEM

[0005] The present disclosure encompasses below-described aspects.

20 [Aspect 1] A composition comprising:

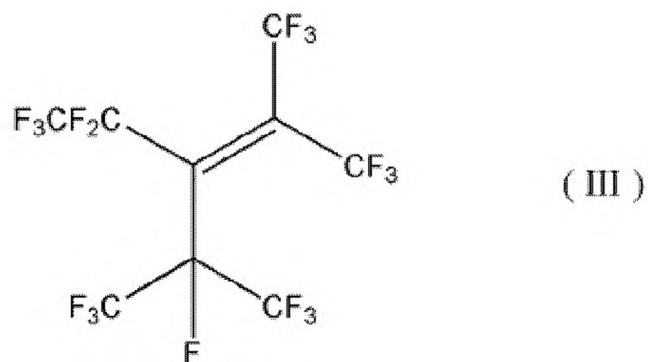
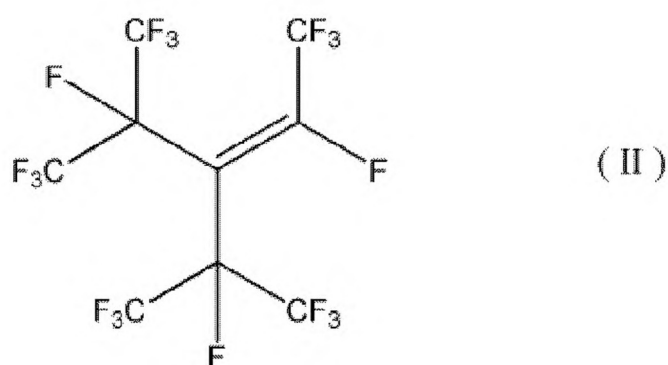
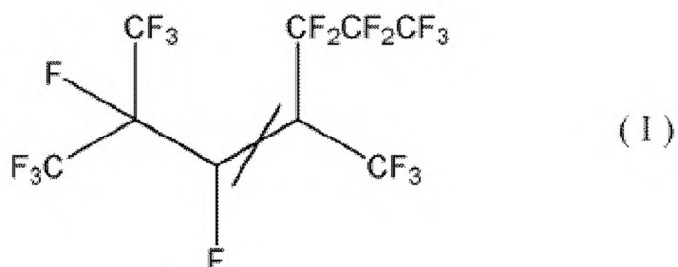
a hexafluoropropene trimer represented by C_9F_{18} ; and

(i) C_mF_{2m} and/or $C_nF_{(2n-2)}$ where m is an integer of 4 to 12 except 9 and n is an integer of 4 to 12, (ii) water where a content of the water relative to a total amount of the hexafluoropropene trimer represented by C_9F_{18} regarded as 100 parts by mass is
25 from 0.0001 to 0.1 parts by mass, and/or (iii) fluoride ions where a content of the fluoride ions relative to the total amount of the hexafluoropropene trimer represented by C_9F_{18} regarded as 100 parts by mass is from 0.0000001 to 5 parts by mass.

[Aspect 2] The composition according to Aspect 1, wherein the hexafluoropropene trimer includes at least one selected from the group consisting of

compounds represented by formulae (I) to (III) below.

[Chem. 1]



5 [Aspect 3] The composition according to Aspect 2, wherein a content of the compound represented by the formula (I) relative to the total amount of the hexafluoropropene trimer is 85 mass% or more.

[Aspect 4] The composition according to Aspect 2, wherein a content of the compound represented by the formula (I) relative to the total amount of the hexafluoropropene trimer is less than 85 mass%.

[Aspect 5] The composition according to Aspect 2, wherein a content of the compound represented by the formula (I) relative to the total amount of the hexafluoropropene trimer is not less than 50 mass% and less than 85 mass%.

[Aspect 6] The composition according to Aspect 1, comprising the
5 hexafluoropropene trimer represented by C_9F_{18} and (i) C_mF_{2m} and/or $C_nF_{(2n-2)}$ where m is an integer of 4 to 12 except 9 and n is an integer of 4 to 12.

[Aspect 7] The composition according to Aspect 1, wherein m is an integer of 6 to 11 except 9 and n is an integer of 6 to 11.

[Aspect 8] The composition according to Aspect 1, wherein a content of C_mF_{2m}
10 and/or $C_nF_{(2n-2)}$ relative to the total amount of the hexafluoropropene trimer represented by C_9F_{18} regarded as 100 parts by mass is from 0.0001 to 10 parts by mass.

[Aspect 9] The composition according to Aspect 1, comprising the hexafluoropropene trimer represented by C_9F_{18} and (ii) water.

[Aspect 10] The composition according to Aspect 1, comprising the
15 hexafluoropropene trimer represented by C_9F_{18} and (iii) fluoride ions.

[Aspect 11] The composition according to Aspect 1, further comprising a hexafluoropropene tetramer represented by $C_{12}F_{24}$, wherein a content of the hexafluoropropene trimer relative to a total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer is 80 mass% or more.

[Aspect 12] The composition according to Aspect 11, wherein the content of the
20 hexafluoropropene trimer relative to the total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer is from 90 mass% to 99.99 mass%.

[Aspect 13] The composition according to Aspect 11, wherein the hexafluoropropene tetramer includes 1,1,1,2,5,6,6,6-octafluoro-2,3,5-
25 tris(trifluoromethyl)-4-(perfluoropropyl-2-yl)-3-hexene.

[Aspect 14] The composition according to Aspect 1, further comprising a conductive substance, wherein a content of the conductive substance is 100 ppm by mass or less.

[Aspect 15] The composition according to Aspect 1, further comprising a

conductive substance, wherein a content of insoluble matter of 5 μm or more is 10 pieces/mL or less.

ADVANTAGEOUS EFFECTS OF INVENTION

5 [0006] The above-described composition according to the present disclosure allows for providing a composition as a novel mixture.

DESCRIPTION OF EMBODIMENTS

[0007] Herein, the term "contain" conceptually encompasses "comprise", "consist essentially of", and "consist of". Moreover, herein, a numerical range expressed as "(from) A to B" means not less than A and not more than B.

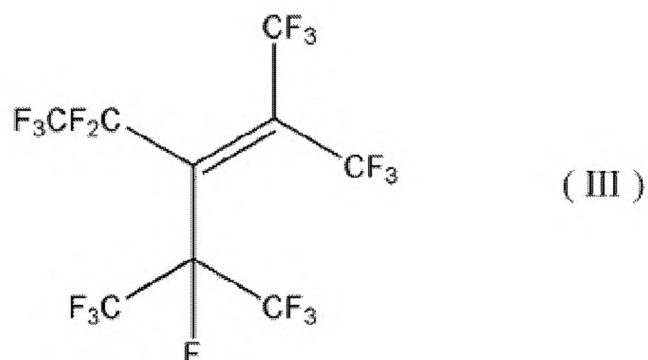
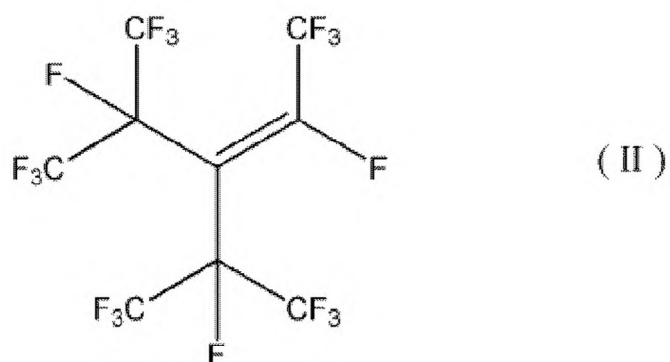
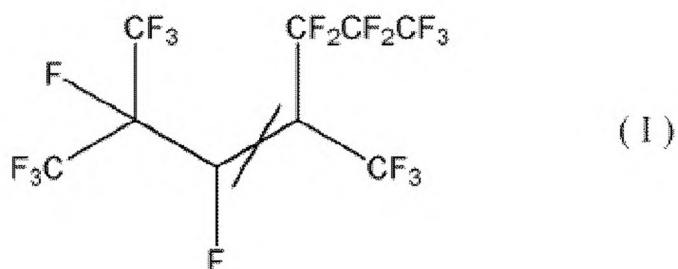
10 [0008] (1. Composition Containing Hexafluoropropene Trimer)

A composition containing a hexafluoropropene (HFP) trimer according to the present disclosure comprises (i) C_mF_{2m} and/or $\text{C}_n\text{F}_{(2n-2)}$ [where m is an integer of 4 to 12 except 9 and n is an integer of 4 to 12], (ii) water (a content of the water relative to a total amount of the hexafluoropropene trimer represented by C_9F_{18} regarded as 100 parts by mass is from 0.0001 to 0.1 parts by mass), and/or (iii) fluoride ions (a content of the fluoride ions relative to the total amount of the hexafluoropropene trimer represented by C_9F_{18} regarded as 100 parts by mass is from 0.0000001 to 5 parts by mass).

15 [0009] The hexafluoropropene trimer is not limited and may be selected from a wide variety of known ones represented by C_9F_{18} .

20 [0010] Specifically, the hexafluoropropene trimer may be a trimer including at least one selected from the group consisting of compounds represented by formulae (I) to (III) below.

[0011] [Chem. 2]



[0012] Herein, the compound represented by the formula (I) includes both E- and Z-diastereomers unless otherwise specified.

- 5 [0013] As the hexafluoropropene trimer contained in the composition according to the present disclosure, only one of the compounds represented by the formulae (I) to (III) may be contained, or a mixture of two or three of them may be contained.

[0014] The proportion of the compound represented by the formula (I) in the total amount of the HFP trimer (namely, the total amount of the compounds represented by

the formulae (I), (II), and (III)) is preferably 1 mass% or more, more preferably 10 mass% or more, further preferably 30 mass% or more, further more preferably 40 mass% or more, particularly preferably 45 mass% or more, particularly preferably 50 mass% or more. In some embodiments, the compound represented by the formula (I)

5 in the total amount of the HFP trimer may be 85 mass% or more. In this case, the HFP trimer mixture has a low viscosity. Moreover, the compound represented by the formula (I) in the total amount of the HFP trimer is preferably 99 mass% or less, more preferably 90 mass% or less, further preferably 85 mass% or less (or less than 85 mass%), yet further preferably 80 mass% or less, particularly preferably 70 mass% or less, most preferably 60 mass% or less. For example, the proportion of the compound represented by the formula (I) in the total amount of the HFP trimer may be from 10 mass% to 90 mass%, or from 30 mass% to 90 mass%, or from 10 mass% to 85 mass%, or from 35 mass% to 85 mass%, or not less than 10 mass% and less than 85 mass%, or not less than 20 mass% and less than 85 mass%, or not less than 30 mass% and less than 85 mass%, or not less than 40 mass% and less than 85 mass%, or not less than 50 mass% and less than 85 mass%, or from 40 mass% to 80 mass%, or from 55 mass% to 80 mass%, or from 60 mass% to 75 mass%, or from 65 mass% to 70 mass%, or from 35 mass% to 60 mass%, or from 50 mass% to 60 mass%, and it may be preferably from 30 mass% to 90 mass%, preferably not less than 40 mass% and less than 85 mass%, more preferably from 40 mass% to 80 mass%, further preferably from 45 mass% to 70 mass%, further more preferably from 50 mass% to 60 mass%.

[0015] Similarly, the proportion of the compound represented by the formula (II) in the total amount of the HFP trimer is preferably 1 mass% or more, more preferably 5 mass% or more, particularly preferably 10 mass% or more. Moreover, the compound represented by the formula (II) in the total amount of the HFP trimer is 70 mass% or less, more preferably 50 mass% or less, yet further preferably 30 mass% or less, particularly preferably 20 mass% or less.

[0016] Similarly, the proportion of the compound represented by the formula (III) in the total amount of the HFP trimer is preferably 1 mass% or more, more preferably 5

mass% or more, particularly preferably 10 mass% or more. Moreover, the compound represented by the formula (III) in the total amount of the HFP trimer regarded as 100 mass% is preferably 70 mass% or less, more preferably 50 mass% or less, yet further preferably 30 mass% or less, particularly preferably 20 mass% or less.

5 [0017] The mass ratio between the compound represented by the formula (II) and the compound represented by the formula (III) in the composition according to the present disclosure is not limited, and, for example, it may be from 1:9 to 9:1, or from 2:8 to 8:2, or from 3:7 to 7:3, or from 4:6 to 6:4, or from 4.5:5.5 to 5.5:4.5.

10 [0018] The compounds represented by the formulae (I) to (III) may be produced by a conventional procedure, and examples of the procedure include, but not limited to, a method described in WO2018/172919. Moreover, the compounds may be obtained by trimerization of HFPs, but this is not limitative and the compounds may be obtained by a method selected from a wide range of methods.

15 [0019] The composition according to the present disclosure may contain a hexafluoropropene trimer represented by C_9F_{18} other than the compounds represented by the formulae (I) to (III).

[0020] The composition according to the present disclosure may contain a hexafluoropropene dimer.

20 [0021] The hexafluoropropene dimer may include (E)-1,1,1,2,3,4,5,5,5-nonafluoro-4-(trifluoromethyl)-2-pentene, (Z)-1,1,1,2,3,4,5,5,5-nonafluoro-4-(trifluoromethyl)-2-pentene, or 1,1,3,4,4,5,5-nonafluoro-2-(trifluoromethyl)-2-pentene.

[0022] In one aspect, the composition according to the present disclosure may contain a hexafluoropropene tetramer.

25 [0023] The hexafluoropropene tetramer may include 1,1,1,2,5,6,6,6-octafluoro-2,3,5-tris(trifluoromethyl)-4-(perfluoropropyl-2-yl)-3-hexene.

[0024] In the composition according to the present disclosure, the content of the hexafluoropropene trimer relative to the total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer may be 80 mass% or more, preferably 85 mass% or more, more preferably 90 mass% or more, and, for example, it may be 95 mass% or

more, or 98 mass% or more, or 99 mass% or more, or 99.9 mass% or more.

[0025] In the composition according to the present disclosure, the content of the hexafluoropropene trimer relative to the total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer may be preferably 99.999 mass% or less, more preferably 99.99 mass% or less, and, for example, it may be 99.9 mass% or less, or 99 mass% or less, or 95 mass% or less, or 90 mass% or less, or 85 mass% or less.

[0026] In the composition according to the present disclosure, the content of the hexafluoropropene trimer relative to the total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer may be preferably from 80 mass% to 99.999 mass%, more preferably from 85 mass% to 99.99 mass%, and, for example, it may be from 90 mass% to 99.99 mass%, or from 95 mass% to 99.99 mass%, or from 99 mass% to 99.99 mass%, or from 99 mass% to 99.9 mass%.

[0027] The total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer in the composition may be preferably 80 mass% or more, more preferably 85 mass% or more, further preferably 90 mass% or more, further more preferably 95 mass% or more, and, for example, it may be 98 mass% or more, or 99 mass% or more, or 99.9 mass% or more. The total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer in the composition may be substantially 100 mass%. In other words, the composition according to the present disclosure may be a mixture of the hexafluoropropene trimer and the hexafluoropropene tetramer.

[0028] (1-1. C_mF_{2m} and/or $C_nF_{(2n-2)}$)

In one aspect, the composition according to the present disclosure may contain the hexafluoropropene (HFP) trimer represented by C_9F_{18} as well as C_mF_{2m} and/or $C_nF_{(2n-2)}$ [where m is an integer of 4 to 12 except 9 and n is an integer of 4 to 12].

[0029] m is an integer of 4 or more, preferably an integer of 5 or more, more preferably an integer of 6 or more. Moreover, n is an integer of 12 or less, preferably an integer of 11 or less, more preferably an integer of 10 or less. It should be noted that m does not include 9. Particularly preferably, m is 8.

[0030] n is an integer of 4 or more, preferably an integer of 5 or more, more preferably

an integer of 6 or more. Moreover, n is an integer of 12 or less, preferably an integer of 11 or less, more preferably an integer of 10 or less. Particularly preferably, n is 9.

[0031] C_mF_{2m} may be either a chain compound or a cyclic compound that may have a substituted structure. The chain compound may be a so-called alkene, and it may be
5 either linear or branched.

[0032] $C_nF_{(2n-2)}$ may be either a chain compound or a cyclic compound that may have a substituted structure. The chain compound may be a so-called diene or alkyne, and it may be either linear or branched.

[0033] When the composition according to the present disclosure contains C_mF_{2m}
10 and/or $C_nF_{(2n-2)}$, the stability of the HFP trimer is enhanced.

[0034] Preferably, the content of C_mF_{2m} and/or $C_nF_{(2n-2)}$ in the entire composition according to the present disclosure is 0.0001 mass% or more.

[0035] Moreover, the content of C_mF_{2m} and/or $C_nF_{(2n-2)}$ in the entire composition according to the present disclosure is preferably 10 mass% or less, preferably 5 mass%
15 or less, further preferably 1 mass% or less.

[0036] In one aspect, in the composition, the content of C_mF_{2m} and/or $C_nF_{(2n-2)}$ relative to the total amount of the HFP trimer regarded as 100 parts by mass is preferably 0.0001 parts by mass or more, more preferably 0.01 parts by mass or more, further preferably 0.1 parts by mass or more.

[0037] Moreover, in the composition, the content of C_mF_{2m} and/or $C_nF_{(2n-2)}$ relative to the total amount of the HFP trimer regarded as 100 parts by mass is preferably 10 parts
20 by mass or less, more preferably 5 parts by mass or less, further preferably 1 part by mass or less.

[0038] When multiple types of C_mF_{2m} and/or $C_nF_{(2n-2)}$ are contained, the above-
25 mentioned content means the total amount of them.

[0039] When the content of C_mF_{2m} and/or $C_nF_{(2n-2)}$ falls within the above-mentioned range, degradation of the HFP trimer represented by C_9F_{18} can be reduced and thereby an increase of fluoride ions and a rise of acidity can be reduced.

[0040] (1-2. Water)

In one aspect, the composition according to the present disclosure may contain water. The content of water in the composition may be 1 ppm by mass or more, and preferably, it is 5 ppm by mass or more. When the content of water is set to a certain level or more, such as 1 ppm by mass or more, for example, electrification of the composition that can occur when the composition becomes less stable can be reduced. Moreover, the content of water in the composition is 1000 ppm by mass or less, preferably 500 ppm by mass or less, more preferably 100 ppm by mass or less, yet further preferably 20 ppm by mass or less. When the content of water is set to a certain level or less, such as 1000 ppm by mass or less, for example, degradation of the HFP trimer represented by C_9F_{18} during heating can be reduced and thereby an excessive increase of fluoride ions and a rise of acidity can be reduced.

[0041] In one aspect, the content of water relative to the total amount of the compound represented by C_9F_{18} regarded as 100 parts by mass is preferably 0.0001 parts by mass or more, more preferably 0.0005 parts by mass or more, further preferably 0.001 parts by mass or more.

[0042] Moreover, the content of water relative to the total amount of the compound represented by C_9F_{18} regarded as 100 parts by mass is preferably 10 parts by mass or less, more preferably 5 parts by mass or less, further preferably 1 part by mass or less.

[0043] When the composition according to the present disclosure contains a certain amount of water, dielectric strength is enhanced. The dielectric strength of the composition according to the present disclosure may be preferably 40 kV or more, preferably 50 kV or more, further preferably 60 kV or more.

[0044] (1-3. Fluoride Ions)

In one aspect, the composition for heat transfer fluid according to the present disclosure may contain fluoride ions in a content of 0.0000001 mass% or more relative to the entire composition. The amount of fluoride ions in the entire composition is preferably 0.000001 mass% or more, more preferably 0.0001 mass% or more, further preferably 0.001 mass% or more. When the concentration of fluoride ions is 0.0000001 mass% or more, stability of the composition can be maintained and

electrification of the composition can be reduced.

[0045] Moreover, the amount of fluoride ions contained in the composition according to the present disclosure is 5 mass% or less, preferably 1 mass% or less, more preferably 0.1 mass% or less relative to the entire composition. When the amount of fluoride ions is 5 mass% or less, degradation of the compound represented by C_9F_{18} during heating can be reduced.

[0046] In one aspect, the amount of fluoride ions relative to the total amount of the compound represented by C_9F_{18} regarded as 100 parts by mass is preferably 0.0000001 parts by mass or more, more preferably 0.000001 parts by mass or more, more preferably 0.0001 parts by mass or more, further preferably 0.001 parts by mass or more.

[0047] Moreover, the amount of fluoride ions relative to the total amount of the compound represented by C_9F_{18} regarded as 100 parts by mass is preferably 5 parts by mass or less, more preferably 1 part by mass or less, further preferably 0.1 parts by mass or less.

[0048] The amount of fluoride ions can be measured in the following manner, for example. To a specimen, an equal amount (weight) of distilled water is added, and the resultant is shaken for about 20 seconds to extract F ions into the aqueous layer. Then, 2.5 to 3.0 mL of the aqueous layer is taken out with a pipette and mixed with twice the amount of TISAB solution (total ionic strength adjustment buffer), and the resulting liquid is used as a sample for measurement with a fluoride ion meter.

[0049] The source of the fluoride ions is not limited and may be selected from a wide range of known fluoride ion sources. Specific examples include ions from hydrogen fluoride, sodium fluoride, sodium hydrogen fluoride, potassium fluoride, potassium hydrogen fluoride, lithium fluoride, cesium fluoride, calcium fluoride, magnesium fluoride, aluminum fluoride, zinc fluoride, silver fluoride, and iron fluoride. The source of the fluoride ions may include only one type of them, or may include a plurality of types of them. Preferably, the source of the fluoride ions is hydrogen fluoride.

[0050] (1-4. Other Components)

The composition according to the present disclosure may contain perfluorotripropylamine, in addition to the hexafluoropropene trimer. The composition containing the hexafluoropropene trimer and perfluorotripropylamine may be a quasi-azeotropic liquid. In the case where the composition is a quasi-azeotropic liquid, the composition does not undergo significant compositional changes even when it is vaporized, and therefore it has enhanced handleability.

[0051] Herein, the quasi-azeotropic liquid means a liquid where the molar fraction of each of the components of the quasi-azeotropic liquid in the gas phase differs by 10% or less from that in the liquid phase.

[0052] The perfluorotripropylamine is also referred to as tris(heptafluoropropyl)amine or N,N-bis(heptafluoropropyl)(heptafluoropropyl)amine, and represented by the general formula $N(CF_2CF_2CF_3)_a(CF(CF_3)CF_3)_{3-a}$ (where a is an integer of 0 to 3). The perfluorotripropylamine may include only one type of compounds represented by the above general formula, or may include a plurality of types of them. $N(CF_2CF_2CF_3)_3$ is preferable, and $N(CF_2CF_2CF_3)_a(CF(CF_3)CF_3)_{3-a}$ (where a is an integer of 0 to 2) may be included as impurities. Specific examples include a product under the trade name "Fluorinert (registered trademark)" (manufactured by 3M) (FC-3283) and the like.

[0053] The composition according to the present disclosure may further contain perfluoropolyether, a methoxytridecafluoroheptene isomer mixture, perfluorotributylamine, and the like.

[0054] The perfluoropolyether is preferably represented by the general formula:



where

R and R' are the same as or different from each other and each is a monovalent group represented by $-C_mF_{2m+1}$, in which m is an integer of 1 to 8, and

R^l is a divalent fluoropolyoxyalkylene group containing 2 to 20 repeating units, in which the repeating unit is represented by:

(i) $-CFXO-$ (where X is F or CF_3);

- (ii) $-\text{CF}_2\text{CFXO}-$ (where X is F or CF_3);
- (iii) $-\text{CFXCF}_2\text{O}-$ (where X is F or CF_3);
- (iv) $-\text{CF}_2\text{CF}_2\text{CF}_2\text{O}-$; or
- (v) $-\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_2\text{O}-$, or

5 Rf^1 is a divalent group represented by:

(vi) $-(\text{CF}_2)_n\text{-CFY-O}-$ (where n is an integer of 0 to 3, Y is a monovalent group represented by the general formula $-\text{ORf}^2\text{Z}$, Rf^2 is a divalent fluoropolyoxyalkylene group containing 2 to 20 repeating units represented by $-\text{CFXO}-$, $-\text{CF}_2\text{CFXO}-$, $-\text{CF}_2\text{CF}_2\text{CF}_2\text{O}-$, or $-\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_2\text{O}-$, Xs are the same as or different from each other and each is F or CF_3 , and Z is a monovalent C_{1-5} perfluoroalkyl group).

[0055] Specific examples of the perfluoropolyether include products under the trade names GALDEN (registered trademark) "HT135" and GALDEN (registered trademark) "HT110" (both manufactured by Solvay), and the like.

15 [0056] Specifically, the methoxytridecafluoroheptene isomer mixture includes methyl-perfluoroheptene ether (MPHE) ($\text{C}_7\text{F}_{13}\text{OCH}_3$). Specific examples include a product under the trade name "Opteon SF10" (manufactured by Chemours) and the like.

[0057] When the composition according to the present disclosure contains perfluorotripropylamine, perfluoropolyether, and a methoxytridecafluoroheptene isomer mixture, the content ratio of these components does not substantially affect the properties of the entire composition because these components have similar properties to those of the compound represented by C_9F_{18} . Therefore, in this case, the composition according to the present disclosure preferably contains the compound represented by C_9F_{18} in 40 mass% to 99.9 mass%, more preferably 60 mass% to 99.9 mass%, further preferably 80 mass% to 99.9 mass%, relative to the entire composition.

25 [0058] (1-5. Other Components)

The composition according to the present invention can be used together with a component other than the composition. As long as the effect and the purpose of the composition are not impaired, the composition according to the present disclosure may

contain a freely-selected additive other than the composition in addition to the above-mentioned compounds represented by the formulae (I) to (III) as well as C_mF_{2m} and/or $C_nF_{(2n-2)}$, water, and/or fluoride ions. Examples of the freely-selected additive include a stabilizer and the like.

5 [0059] The stabilizer exhibits stabilizing effects and thereby it functions as a so-called acid scavenger or antioxidant. Examples of major stabilizing effects include effects to capture radicals produced in the system to prevent degradation of the HFP trimer, acid scavenging effects to capture acids produced in the system to prevent further degradation of the HFP trimer due to acid, and the like.

10 [0060] The stabilizer may be selected from a wide range of known stabilizers. Among these, preferably, for effectively reducing metal corrosion caused by the composition, one or more types of stabilizers selected from the group consisting of unsaturated alcohol-based stabilizers, nitro-based stabilizers, amine-based stabilizers, phenol-based stabilizers, and epoxy-based stabilizers are used.

15 [0061] The unsaturated alcohol-based stabilizer may be selected from a wide range of known ones. For example, one or more selected from the group consisting of 3-buten-2-ol, 2-buten-1-ol, 4-propen-1-ol, 1-propen-3-ol, 2-methyl-3-buten-2-ol, 3-methyl-3-buten-2-ol, 3-methyl-2-buten-1-ol, 2-hexen-1-ol, 2,4-hexadien-1-ol, and oleyl alcohol can be used.

20 [0062] The nitro-based stabilizer may be selected from a wide range of known ones. As aliphatic nitro compounds, nitromethane, nitroethane, 1-nitropropane, 2-nitropropane, and the like may be mentioned, for example. As aromatic nitro compounds, one or more selected from the group consisting of nitrobenzene, o-, m-, and p-dinitrobenzenes, o-, m-, and p-nitrotoluenes, dimethylnitrobenzene, m-nitroacetophenone, o-, m-, and p-nitrophenols, o-nitroanisole, m-nitroanisole, and p-nitroanisole can be used, for example.

25 [0063] The amine-based stabilizer may be selected from a wide range of known ones. For example, one or more selected from the group consisting of pentylamine, hexylamine, diisopropylamine, diisobutylamine, di-n-propylamine, diallylamine,

triethylamine, N-methylaniline, pyridine, morpholine, N-methylmorpholine, triallylamine, allylamine, α -methylbenzylamine, dimethylamine, trimethylamine, ethylamine, diethylamine, triethylamine, propylamine, isopropylamine, dipropylamine, tripropylamine, butylamine, isobutylamine, dibutylamine, tributylamine, dipentylamine, tripentylamine, 2-ethylhexylamine, aniline, N,N-dimethylaniline, N,N-diethylaniline, ethylenediamine, propylenediamine, diethylenetriamine, tetraethylenepentamine, benzylamine, dibenzylamine, diphenylamine, and diethylhydroxylamine can be used.

[0064] The phenol-based stabilizer may be selected from a wide range of known ones. For example, one or more selected from the group consisting of 2,6-di-tert-butyl-4-methylphenol, 3-cresol, phenol, 1,2-benzenediol, 2-isopropyl-5-methylphenol, and 2-methoxyphenol can be used.

[0065] The epoxy-based stabilizer may be selected from a wide range of known ones. For example, one or more selected from the group consisting of butylene oxide, 1,2-propylene oxide, 1,2-butylene oxide, butyl glycidyl ether, diethylene glycol diglycidyl ether, and 1,2-epoxy-3-phenoxypropane can be used.

[0066] Use of a combination of stabilizers with different stabilizing effects can more effectively prevent degradation of the HFP trimer that can occur from various causes, and therefore, it is preferable to use one or more selected from the group consisting of the epoxy-based stabilizers, the unsaturated alcohol-based stabilizers, the nitro-based stabilizers, and the phenol-based stabilizers mentioned above.

[0067] From the viewpoint of effectively reducing acid release from the HFP trimer to reduce metal corrosion caused by the liquid composition, the content of the stabilizer in the entire composition according to the present disclosure is preferably 0.0001 mass% or more, more preferably 0.01 mass% or more. On the other hand, in consideration of avoiding undesired changes of the physical properties of the liquid composition due to excessive addition of stabilizers, the content of the stabilizer in the entire composition according to the present disclosure is preferably 10 mass% or less, more preferably 5 mass% or less.

[0068] (1-6. Impurities)

[0069] The composition according to the present disclosure may contain conductive substances. The content of conductive substances is 100 ppm by mass or less, preferably 75 ppm by mass or less, more preferably 50 ppm by mass or less, further preferably 10 ppm by mass or less. Specific types of conductive substances that can enter into the device through a gap to cause a short circuit are metals and metal ions, and therefore, as needed, study and analysis on the content of conductive substances according to the present disclosure can be performed in terms of "at least one of metals and metal ions".

[0070] In another aspect, in the composition according to the present disclosure, the content of insoluble matter of 5 μm or more (including solid matter such as resin fragments of the container and dirt and dust from the air, both conductive and not conductive) is preferably 10 pieces/mL or less, more preferably 5 pieces/mL or less, further preferably 3 pieces/mL or less.

[0071] (1-7. Method of Producing Composition with Reduced Impurities)

The present disclosure provides a method of producing a composition with reduced impurities. The method of producing a composition according to the present disclosure has a step to perform purification treatment of a composition containing both a compound represented by C_9F_{18} and conductive substances to obtain the composition from which the conductive substances are reduced, characterized in that:

(1) the purification treatment is a treatment performed with the use of at least one selected from the group consisting of filtration filter, ion-exchange resin, metal-ion removal filter, metal-ion removing agent, distillation, fractionation, centrifugation, and electrostatic attraction.

In the present disclosure, "reducing (reduced)" in the context of purification means reducing the content ratio of conductive substances in the composition.

[0072] According to the research performed by the inventors of the present disclosure, a composition containing a compound represented by C_9F_{18} contains conductive substances (such as metals, metal ions, carbon, conductive polymers, superconducting

ceramic, and the like). The conductive substances include those inevitably contaminating due to the process of production of the composition and also those contaminating during use after production. In the case where the composition is used for transferring heat to and/or from a device, the conductive substances can enter into the device through a gap to cause a short circuit. Moreover, they can cause clogging of the piping through which the composition is circulated, during repeated use of the composition. A conventionally known composition, because it contains conductive substances, can also be expressed as "a composition containing both a compound represented by C₉F₁₈ and conductive substances". The method of producing a composition according to the present disclosure is characterized in that it performs a specific purification treatment of a composition containing both an HFP trimer and conductive substances (hereinafter also called "a pre-purification composition") to obtain the composition from which the conductive substances are reduced.

[0073] (1-7-1) Composition Containing Both Compound Represented by C₉F₁₈ and Conductive Substances (Pre-Purification Composition)

The pre-purification composition may contain both a compound represented by C₉F₁₈ and conductive substances.

[0074] (1-7-2) Compound Represented by C₉F₁₈

The compound represented by C₉F₁₈ is the above-mentioned hexafluoropropene trimer.

[0075] (1-7-3) Compound That Can Be Additionally Contained

The composition according to the present disclosure may contain an additional compound (also called "an additional component") that is different from the compound represented by C₉F₁₈. The additional component may be of one or more types, and the additional component may be a hexafluoropropene dimer, a hexafluoropropene tetramer, and the like, for example.

[0076] The hexafluoropropene dimer may include (E)-1,1,1,2,3,4,5,5,5-nonafluoro-4-(trifluoromethyl)-2-pentene, (Z)-1,1,1,2,3,4,5,5,5-nonafluoro-4-(trifluoromethyl)-2-pentene, 1,1,3,4,4,5,5-nonafluoro-2-(trifluoromethyl)-2-pentene.

[0077] The hexafluoropropene tetramer may include 1,1,1,2,5,6,6,6-octafluoro-2,3,5-tris(trifluoromethyl)-4-(perfluoropropyl-2-yl)-3-hexene.

[0078] The composition according to the present disclosure, when containing both the compound represented by C_9F_{18} according to the present disclosure and an additional component, preferably contains the compound represented by C_9F_{18} according to the present disclosure in 80 mass% or more relative to the entire composition, more preferably 85 mass% or more, further preferably 90 mass% or more, most preferably 95 mass% or more. In the production method according to the present disclosure, the properties of the composition containing both the compound represented by C_9F_{18} and conductive substances (the pre-purification composition) such as the kinematic viscosity, coagulation point, and boiling point can affect the efficiency of removal of the conductive substances from the pre-purification composition in relation to a certain purification treatment (a treatment performed with the use of at least one selected from the group consisting of filtration filter, ion-exchange resin, metal-ion removal filter, metal-ion removing agent, distillation, fractionation, centrifugation, and electrostatic attraction), and therefore it is preferable that no additional component be contained; however, in the case where an additional component is contained, it is preferable that the compound represented by C_9F_{18} be contained in 80 mass% or more relative to the entire composition as mentioned above.

[0079] Whether or not an additional component is contained in addition to the compound represented by C_9F_{18} according to the present disclosure, the composition containing both the compound represented by C_9F_{18} and conductive substances (the pre-purification composition) may be selected from a wide range including, for example, synthesized products and commercially available products of the compound represented by C_9F_{18} according to the present disclosure as described above, as well as such synthesized products and commercially available products that were contaminated with conductive substances after a certain period of use.

[0080] (1-7-4) Conductive Substances

Examples of the conductive substances include at least one selected from the

group consisting of metals, metal ions, metal oxides, metal nitrides, carbon, conductive polymers, and superconducting ceramic. In the case where the composition containing conductive substances is used for a device, the conductive substances can enter into the device through a gap to cause a short circuit. Moreover, they can cause clogging of the piping through which the compound represented by C_9F_{18} is circulated, during repeated use of the composition according to the present disclosure. Hence, for the purpose of enhancing the performance of the compound represented by C_9F_{18} , it is important to reduce the content of conductive substances. The shape and the size of the conductive substances vary depending on the type of the conductive substances, and typically, the conductive substances are of about 0.001 to 10 μm .

[0081] The type of the metal of the above-mentioned metals, metal ions, metal oxides, and metal nitrides as conductive substances may be, for example, Al, Ba, Be, Bi, Ca, Co, Cr, Cu, Fe, Ga, K, Li, Mg, Mn, Na, Ni, Pb, Sr, V, Zn, and the like.

[0082] The metal as a conductive substance may be either in the form of an elementary substance or in the form of alloy. Typically, the metal as a conductive substance is contained in the form of fine particles.

[0083] The ions as conductive substances include all forms of ions possible for the type of the metal. The ions as conductive substances may be present in the dissolved state as ions with certain valence, or may be present within the substance as ions such as, for example, in the coordinated state.

[0084] The metal oxide as a conductive substance may be an oxide of one type of metal, or may be an oxide of a plurality of types of metals (namely, a composite oxide).

[0085] The metal nitride as a conductive substance may be a nitride of one type of metal, or may be a nitride of a plurality of types of metals (namely, a composite nitride).

[0086] The carbon as a conductive substance may be carbon black, for example.

[0087] The conductive polymer as a conductive substance may be polyacetylene and/or polythiophene, for example.

[0088] The content of conductive substances in the pre-purification composition is not

limited, and, for example, when it is 150 ppm by mass or more (or even 500 ppm by mass or more), the content of conductive substances can be effectively reduced by the production method according to the present disclosure. The content of conductive substances can be measured as described in Examples with the use of an inductively coupled plasma mass spectrometry instrument (ICP-MS).

[0089] The pre-purification composition may contain insoluble matter including solid matter such as resin fragments of the container and dirt and dust from the air, both conductive and not conductive; for example, when the content of insoluble matter of 5 μm or more is 50 pieces/mL or more (or even 100 pieces/mL or more), by performing the purification treatment according to the present disclosure capable of reducing the content of conductive substances, it is also possible to effectively reduce the content of insoluble matter. The number of insoluble matter (fine particles) of conductive substances contained in the composition can be measured as described in Examples with the use of a liquid particle counter.

[0090] (1-7-5) Physical Properties of Pre-Purification Composition

The pre-purification composition is a composition containing both a compound represented by C_9F_{18} and conductive substances, and in the production method according to the present disclosure, it is subjected to purification treatment performed with the use of at least one selected from the group consisting of filtration filter, ion-exchange resin, metal-ion removal filter, metal-ion removing agent, distillation, fractionation, centrifugation, and electrostatic attraction.

[0091] In view of performing the above-mentioned purification treatment to reduce the content of conductive substances, from the viewpoint of purification efficiency, it is preferable that the pre-purification composition have a low kinematic viscosity, a low coagulation point, and a high boiling point. Containing the compound represented by C_9F_{18} as the main component (preferably in 80 mass% or more), the pre-purification composition tends to have such physical properties as a low kinematic viscosity, a low coagulation point, and a high boiling point.

[0092] The kinematic viscosity of the pre-purification composition at -40°C is

preferably 15 cSt or less, more preferably 10 cSt or less, further preferably 7 cSt or less. When the kinematic viscosity is as low as 15 cSt or less, ease of filtration is enhanced, and thereby the efficiency of purification treatment with the use of a filtration filter, a metal-ion removal filter, and/or the like, in particular, can be enhanced. The method
5 for measuring the kinematic viscosity at -40°C according to the present disclosure is described in Examples.

[0093] The coagulation point of the pre-purification composition is preferably -35°C or less, more preferably -50°C or less, further preferably -70°C or less, most preferably -100°C or less. When the coagulation point is as low as -35°C or less, the purification
10 treatment can be carried out at low temperatures and, therefore, loss of the compound represented by C₉F₁₈ due to evaporation and/or the like can be reduced and the efficiency of purification treatment can be enhanced. The method for measuring the coagulation point according to the present disclosure is described in Examples.

[0094] The boiling point of the pre-purification composition is 90°C or more, more preferably 95°C or more, further preferably 105°C or more. When the boiling point is as high as 90°C or more, for the same reason as mentioned above with respect to the low coagulation point, loss of the compound represented by C₉F₁₈ due to evaporation and/or the like can be reduced and the efficiency of purification treatment can be enhanced. The method for measuring the boiling point according to the present
15 disclosure is described in Examples.

[0095] (1-7-6) Purification Treatment of Pre-Purification Composition

In the production method according to the present disclosure, the pre-purification composition is subjected to purification treatment performed with the use of at least one selected from the group consisting of filtration filter, ion-exchange resin,
25 metal-ion removal filter, metal-ion removing agent, distillation, fractionation, centrifugation, and electrostatic attraction.

[0096] These purification means can be used or carried out according to the conventional procedure, and in the production method according to the present disclosure, purification treatment with a filtration filter is particularly preferable.

When a filtration filter is used, the upper limit and the lower limit to the filter pore size are not limited, and the upper limit can be set at 5 μm or less, or 1 μm or less, or 0.5 μm or less, or 0.1 μm or less, for example. Moreover, the lower limit can be set at 1.0 nm or more, or 0.5 nm or more, or 0.2 nm or more, or 0.1 nm or more, for example.

5 [0097] The ion-exchange resin may be either a cation-exchange resin or an anion-exchange resin. The anion-exchange resin can be an ion-exchange resin having amino groups and/or quaternary ammonium groups as functional groups, for example. Preferably, the ion-exchange resin is a strongly basic anion-exchange resin. The basicity of the anion-exchange resin can be adjusted by selecting the type of the
10 polymer backbone and/or the functional group. The anion-exchange resin may be a commercially available product, and, for example, "Diaion (registered trademark) SA" products and the like manufactured by Mitsubishi Chemical, "A200" and the like manufactured by Purolite, and "Amberlite (registered trademark)" products and the like manufactured by Organo can be used. The cation-exchange resin can be an ion-
15 exchange resin having carboxyl groups and/or sulfonic groups as functional groups, for example. The acidity of the cation-exchange resin can be adjusted by selecting the type of the polymer backbone and/or the functional group. The cation-exchange resin may be a commercially available product, and, for example, "Diaion (registered trademark) SK" products and the like manufactured by Mitsubishi Chemical, "C100"
20 and the like manufactured by Purolite, and "Amberlite (registered trademark)" products and the like manufactured by Organo can be used.

[0098] The metal-ion removing agent may be a chelating agent, activated carbon, and/or the like. For example, the chelating agent can be CRB03, CRB05, and CR20 (all manufactured by Mitsubishi Chemical), Si-Thiol, Si-Thiourea, Si-TMT, Si-DMT,
25 Si-SCX-2, Si-Amine, Si-Trisamine, Si-Imidazole, Si-TBD, and Si-PHI (all manufactured by SiliCycle), Muromac XMS-5418 (manufactured by Muromachi Chemicals), IRC76-HG, IRC748, and IRC747UPS (all manufactured by Organo), S910 (manufactured by Purolite), MPA (manufactured by Reaxa QuadraPure), and the like. For example, the activated carbon may be "SHIRASAGI (registered trademark)" and

the like manufactured by Osaka Gas Chemicals, "Filtrisorb (registered trademark) CAL", "Diahope (registered trademark)", "Diasorb (registered trademark)", and the like manufactured by Calgon Carbon Japan, "Evadia (registered trademark)" products and the like manufactured by Swing, and the like.

5 [0099] By carrying out the above-mentioned purification treatment, it is possible to obtain a composition (a post-purification composition) in which the content of conductive substances is reduced.

[0100] The above-described production method has the above-described purification treatment step, and it may also have other steps such as a step to mix an additive such
10 as a stabilizer to the composition.

[0101] (2. Use of Composition)

The composition according to the present disclosure can be used for various purposes of use. For example, the composition according to the present disclosure can be used as heat transfer fluid, synthetic solvent, and the like.

15 [0102] Thus far, embodiments of the present invention are described, but it is obvious that the present invention is by no means limited to those examples and can be implemented in various configurations without departing from the gist of the present invention.

Examples

20 [0103] In the following, a more detailed description will be given of embodiments of the present invention by way of Examples, but these Examples are not intended to limit the scope of the present invention.

[0104] (Production Example 1)

25 Based on a method described in Chem Bar (1973), vol. 106, pp 2950-2959, an HFP trimer was obtained. The HFP trimer thus obtained was purified by distillation for removal of impurities such as hexafluoropropene dimers and tetramers. The purified HFP trimer was distilled and thereby separated into compounds represented by the formulae (I), (II), and (III). Each HFP trimer thus separated was dehydrated on silica gel.

[0105] The compounds represented by the formulae (I), (II), and (III) thus obtained were mixed in such a manner that the ratio between the compounds represented by the formulae (I), (II), and (III) became as specified in the table below, and thereby trimer mixtures 1 to 4 were obtained.

5 [0106] [Table 1]

Trimer mixture	Formula (I)	Formula (II)	Formula (III)
1	1.5 mass%	35.5 mass%	63.0 mass%
2	52.0 mass%	16.0 mass%	32.0 mass%
3	77.0 mass%	8.0 mass%	15.0 mass%
4	90.0 mass%	5.0 mass%	5.0 mass%

[0107] (Production Example 2)

Based on a method described in Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1981), vol. 4, pp 1064-1067, C_9F_{16} was synthesized.

10 [0108] (Production Example 3)

As C_8F_{16} , a reagent manufactured by Wako Pure Chemical Industries was purchased.

[0109] (Examples 1 to 43)

15 The trimer mixtures 1 to 4 and an additive, which was C_9F_{16} of Production Example 2 or C_8F_{16} of Production Example 3, were mixed in the proportion specified in the table below, and thereby compositions for heat conduction fluid of Examples 1 to 43 were obtained.

[0110] (Stability Test)

20 The composition for heat conduction fluid thus obtained was placed and hermetically sealed in an autoclave made of SUS, and then heated and maintained under the conditions specified in the table below.

[0111] (Measurement of Concentration of Fluoride Ions)

25 To a specimen, an equal amount (weight) of distilled water was added, and the resultant was shaken for about 20 seconds to extract F ions into the aqueous layer. Then, 2.5 to 3.0 mL of the aqueous layer was taken out with a pipette and mixed with twice the amount of TISAB solution (total ionic strength adjustment buffer,

manufactured by HORIBA), and the resulting liquid was used as a sample for measurement with a fluoride ion meter (manufactured by HORIBA). In consideration of the dilution with the TISAB solution, three times the measured value is the concentration of fluoride ions. Results are given in the table below.

[0112] [Table 2]

	Trimer mixture No.	Trimer content (g)	Additive	Additive content (mg)	wt%
Ex. 1	1	10	C ₉ F ₁₆	140	1.39
Ex. 2	2	10	C ₉ F ₁₆	0.01	0.0001
Ex. 3	2	10	C ₉ F ₁₆	1	0.01
Ex. 4	2	10	C ₉ F ₁₆	10	0.1
Ex. 5	2	10	C ₉ F ₁₆	0.01	0.0001
Ex. 6	2	10	C ₉ F ₁₆	1	0.01
Ex. 7	2	10	C ₉ F ₁₆	10	0.1
Ex. 8	3	10	C ₉ F ₁₆	0.01	0.0001
Ex. 9	3	10	C ₉ F ₁₆	1	0.01
Ex. 10	3	10	C ₉ F ₁₆	10	0.1
Ex. 11	3	10	C ₉ F ₁₆	0.01	0.0001
Ex. 12	3	10	C ₉ F ₁₆	1	0.01
Ex. 13	3	10	C ₉ F ₁₆	10	0.1
Ex. 14	4	10	C ₉ F ₁₆	0.01	0.0001
Ex. 15	4	10	C ₉ F ₁₆	1	0.01
Ex. 16	4	10	C ₉ F ₁₆	10	0.1
Ex. 17	4	10	C ₉ F ₁₆	0.01	0.0001
Ex. 18	4	10	C ₉ F ₁₆	1	0.01
Ex. 19	4	10	C ₉ F ₁₆	10	0.1
Ex. 20	2	10	C ₈ F ₁₆	0.01	0.0001
Ex. 21	2	10	C ₈ F ₁₆	1	0.01
Ex. 22	2	10	C ₈ F ₁₆	10	0.1
Ex. 23	2	10	C ₈ F ₁₆	100	1.0
Ex. 24	2	10	C ₈ F ₁₆	0.01	0.0001
Ex. 25	2	10	C ₈ F ₁₆	1	0.01
Ex. 26	2	10	C ₈ F ₁₆	10	0.1
Ex. 27	2	10	C ₈ F ₁₆	100	1.0
Ex. 28	3	10	C ₈ F ₁₆	0.01	0.0001
Ex. 29	3	10	C ₈ F ₁₆	1	0.01
Ex. 30	3	10	C ₈ F ₁₆	10	0.1
Ex. 31	3	10	C ₈ F ₁₆	100	1.0
Ex. 32	3	10	C ₈ F ₁₆	0.01	0.0001
Ex. 33	3	10	C ₈ F ₁₆	1	0.01
Ex. 34	3	10	C ₈ F ₁₆	10	0.1
Ex. 35	3	10	C ₈ F ₁₆	100	1.0
Ex. 36	4	10	C ₈ F ₁₆	0.01	0.0001
Ex. 37	4	10	C ₈ F ₁₆	1	0.01
Ex. 38	4	10	C ₈ F ₁₆	10	0.1
Ex. 39	4	10	C ₈ F ₁₆	100	1.0
Ex. 40	4	10	C ₈ F ₁₆	0.01	0.0001
Ex. 41	4	10	C ₈ F ₁₆	1	0.01
Ex. 42	4	10	C ₈ F ₁₆	10	0.1
Ex. 43	4	10	C ₈ F ₁₆	100	1.0
Comp. Ex. 1	2	10	-	-	-
Comp. Ex. 2	2	10	-	-	-

[0113] [Table 3]

	Initial concentration of F ions (ppm by mass)	Stability test		After-test concentration of F ions (ppm by mass)
		Temperature (°C)	Time (hour)	
Ex. 1	0.6	100	168	1.2
Ex. 2	0.6	100	168	3.3
Ex. 3	0.6	100	168	2.1
Ex. 4	0.6	100	168	1.8
Ex. 5	0.6	200	168	3.6
Ex. 6	0.6	200	168	2.7
Ex. 7	0.6	200	168	2.1
Ex. 8	0.6	100	168	3.6
Ex. 9	0.6	100	168	2.4
Ex. 10	0.6	100	168	1.8
Ex. 11	0.6	200	168	4.2
Ex. 12	0.6	200	168	3.6
Ex. 13	0.6	200	168	2.7
Ex. 14	0.6	100	168	3.9
Ex. 15	0.6	100	168	2.7
Ex. 16	0.6	100	168	2.1
Ex. 17	0.6	200	168	4.5
Ex. 18	0.6	200	168	3.9
Ex. 19	0.6	200	168	3.3
Ex. 20	0.6	100	168	1.2
Ex. 21	0.6	100	168	0.9
Ex. 22	0.6	100	168	0.3
Ex. 23	0.6	100	168	0.3
Ex. 24	0.6	200	168	1.5
Ex. 25	0.6	200	168	1.2
Ex. 26	0.6	200	168	0.9
Ex. 27	0.6	200	168	0.3
Ex. 28	0.6	100	168	1.5
Ex. 29	0.6	100	168	1.2
Ex. 30	0.6	100	168	0.9
Ex. 31	0.6	100	168	0.6
Ex. 32	0.6	200	168	1.8
Ex. 33	0.6	200	168	1.5
Ex. 34	0.6	200	168	1.2
Ex. 35	0.6	200	168	0.6
Ex. 36	0.6	100	168	2.1
Ex. 37	0.6	100	168	1.5
Ex. 38	0.6	100	168	0.9
Ex. 39	0.6	100	168	0.6
Ex. 40	0.6	200	168	1.8
Ex. 41	0.6	200	168	1.8
Ex. 42	0.6	200	168	1.2
Ex. 43	0.6	200	168	0.9
Comp. Ex. 1	0.6	100	168	45
Comp. Ex. 2	0.6	200	168	99

[0114] From the above results, it is demonstrated that Examples 1 to 43 containing C₉F₁₆ or C₈F₁₆ did not show an increase of fluorine concentrations even after long-term storage at high temperatures and therefore they were stable.

[0115] <Measurement of Boiling Point, Pour Point, and Dielectric Constant>

5 The boiling point of the trimer mixture was measured by DSC (differential scanning calorimetry), as a temperature at which an endothermic peak was observed while the temperature was being raised from 25°C at 5°C/min. The pour point was measured by DSC, as a temperature at which an endothermic peak was observed while the temperature was being raised at 5°C/min after lowered with liquid nitrogen to the
10 coagulation point or less. The dielectric constant was a value at a frequency of 1 kHz measured by a capacitance method in an environment at a temperature of 25°C and a humidity of 60%.

[0116] <Measurement of Kinematic Viscosity and Density>

15 The kinematic viscosity and the density of the trimer mixture were measured with a kinematic viscometer, SVM3001, manufactured by Anton Paar.

[0117] <Measurement of Specific Heat>

 The specific heat of the trimer mixture was measured by DSC. The conditions of the measurement were as described below.

 Measurement apparatus: Differential scanning calorimeter DSC8500

20 manufactured by Perkin-Elmer

 Temperature raising rate: 10°C/minute

 Reference specimen: Sapphire (-Al₂O₃)

 Atmosphere: In dry nitrogen stream

 Specimen container: Hermetically sealed aluminum container

25 [0118] <Measurement of Thermal Conductivity>

 The thermal conductivity of the trimer mixture was measured by a transient hot-wire method.

[0119] <Compatibility>

 The compatibility of the trimer mixture was evaluated by using a mixture of

equal amounts of the below-listed three types of solvents and each trimer mixture.

Galden HT135 (manufactured by Solvay)

SF-10 (manufactured by Chemours)

FC3283 (manufactured by 3M)

5 [0120] [Table 4]

	Mixture 1	Mixture 2	Mixture 3	Mixture 4
Boiling point (°C)	112	109	109	108.5
Pour point (°C)	-110	<-130	<-130	<-130
Dielectric constant (1 kHz)	1.97	2.00	2.00	2.00
Kinematic viscosity (cSt) @-20°C	4.8	2.6	2.5	2.4
Dielectric strength (kV)	>40	>40	>40	>40
Specific heat (J/kg·K) @30°C	1100	1060	1060	1060
Thermal conductivity (W/mK) @30°C	0.074	0.064	0.062	0.060
Compatibility	Compatible with three types	Compatible with three types	Compatible with three types	Compatible with three types

[0121] (Examples 44 to 59)

The trimer mixtures 1 to 4 and water were mixed in the amounts specified in the table below, and thereby compositions of Examples 44 to 59 were obtained.

[0122] [Table 5]

	Trimer mixture	Amount of added trimer (g)	Amount of added water (mg)
Ex. 44	1	26	1.82
Ex. 45	2	10	0.01
Ex. 46	2	10	1
Ex. 47	2	10	10
Ex. 48	2	10	0.01
Ex. 49	2	10	1
Ex. 50	2	10	10
Ex. 51	3	10	0.01
Ex. 52	3	10	1
Ex. 53	3	10	10
Ex. 54	3	10	0.01
Ex. 55	3	10	1
Ex. 56	3	10	10
Ex. 57	4	10	0.01
Ex. 58	4	10	1
Ex. 59	4	10	10
Comp. Ex. 3	2	10	-
Comp. Ex. 4	2	10	-
Comp. Ex. 5	2	10	150
Comp. Ex. 6	2	10	150

[0123] (Stability Test)

Each of the compositions of Examples 44 to 59 and Comparative Examples 3 to 6 was placed and hermetically sealed in an autoclave made of SUS, and then heated and maintained under the conditions specified in the table below.

[0124] (Measurement of Dielectric Strength)

A liquid specimen was made to enter into the gaps between spherical electrodes aligned at regular intervals, followed by raising the voltage at a steady rate, and the breakdown voltage was measured and regarded as the dielectric strength. Specific conditions of the measurement were as described below.

Electrode shape: Spherical (ϕ 12.5 mm)

Intervals between electrodes: 2.5 mm

Pressure-raising rate: 2 kV/second

Atmosphere of measurement: Air (22°C, 57%RH)

[0125] [Table 6]

	Concentration of F ions (initial) (ppm by mass)	Stability test		Concentration of F ions (after test) (ppm by mass)	Dielectric strength (kV)
		Temperature (°C)	Time (hour)		
Ex. 44	0.6	150	168	0.356	64
Ex. 45	0.6	100	168	2.1	68
Ex. 46	0.6	100	168	3.6	60
Ex. 47	0.6	100	168	4.8	61
Ex. 48	0.6	200	168	3.0	68
Ex. 49	0.6	200	168	5.4	63
Ex. 50	0.6	200	168	6.6	61
Ex. 51	0.6	100	168	3.3	66
Ex. 52	0.6	100	168	4.8	54
Ex. 53	0.6	100	168	6.3	52
Ex. 54	0.6	200	168	4.8	68
Ex. 55	0.6	200	168	7.8	63
Ex. 56	0.6	200	168	12.0	58
Ex. 57	0.6	100	168	3.6	48
Ex. 58	0.6	100	168	5.4	44
Ex. 59	0.6	100	168	7.5	46
Comp. Ex. 3	0.6	100	168	102	39
Comp. Ex. 4	0.6	200	168	303	64
Comp. Ex. 5	0.6	100	168	210	68
Comp. Ex. 6	0.6	200	168	492	60

[0126] From the above results, it is demonstrated that Examples 44 to 59 containing certain amounts of water did not show an increase of fluorine concentrations even after long-term storage at high temperatures and therefore they were stable. Moreover, it is also demonstrated that Examples 44 to 59 had high dielectric strength.

[0127] (Examples 60 to 83)

Anhydrous hydrofluoric acid was added to the trimer mixtures 1 to 4 to prepare base liquids, and the resultants were diluted with the corresponding trimer mixtures, respectively, to form compositions of Examples 60 to 83 having concentrations of fluoride ions as specified in the table below.

[0128] (Stability Test)

A specimen was placed and hermetically sealed in an autoclave made of SUS, and then heated and maintained under the conditions specified in the table below.

[0129] (Measurement of Purity of Hexafluoropropene Trimer)

The purity of the hexafluoropropene trimer before and after the stability test was measured by gas chromatography.

[0130] [Table 7]

	Trimer mixture	Trimer content (g)	F ion amount (initial) (ppm by mass)
Ex. 60	1	26	<0.001
Ex. 61	1	9.2	<0.001
Ex. 62	1	8.8	<0.001
Ex. 63	2	10	0.6
Ex. 64	2	10	0.6
Ex. 65	2	10	0.6
Ex. 66	2	10	0.6
Ex. 67	2	10	0.6
Ex. 68	2	10	0.6
Ex. 69	2	10	0.6
Ex. 70	3	10	0.6
Ex. 71	3	10	0.6
Ex. 72	3	10	0.6
Ex. 73	3	10	0.6
Ex. 74	3	10	0.6
Ex. 75	3	10	0.6
Ex. 76	3	10	0.6
Ex. 77	4	10	0.6
Ex. 78	4	10	0.6
Ex. 79	4	10	0.6
Ex. 80	4	10	0.6
Ex. 81	4	10	0.6
Ex. 82	4	10	0.6
Ex. 83	4	10	0.6
Comp. Ex. 7	2	10	0.6
Comp. Ex. 8	2	10	0.6
Comp. Ex. 9	2	10	0.6
Comp. Ex. 10	2	10	0.6

[0131] [Table 8]

	Stability test		Initial purity (wt%)	After-test purity (wt%)
	Temperature (°C)	Time (hour)		
Ex. 60	150	24	99.9	99.9
Ex. 61	150	24	99.9	99.9
Ex. 62	150	24	99.9	99.9
Ex. 63	100	168	99.9	99.9
Ex. 64	100	168	99.9	99.9
Ex. 65	100	168	99.9	99.9
Ex. 66	100	168	99.9	99.9
Ex. 67	200	168	99.9	99.9
Ex. 68	200	168	99.9	99.9
Ex. 69	200	168	99.9	99.8
Ex. 70	100	168	99.9	99.9
Ex. 71	100	168	99.9	99.8
Ex. 72	100	168	99.9	99.8
Ex. 73	100	168	99.9	99.7
Ex. 74	200	168	99.9	99.7
Ex. 75	200	168	99.9	99.7
Ex. 76	200	168	99.9	99.8
Ex. 77	100	168	99.9	99.7
Ex. 78	100	168	99.9	99.7
Ex. 79	100	168	99.9	99.7
Ex. 80	100	168	99.9	99.6
Ex. 81	200	168	99.9	99.6
Ex. 82	200	168	99.9	99.5
Ex. 83	200	168	99.9	99.5
Comp. Ex. 7	100	168	99.9	98.4
Comp. Ex. 8	200	168	99.9	98.5
Comp. Ex. 9	200	168	99.9	99.2
Comp. Ex. 10	200	336	99.9	99.0

[0132] From the above results, it is demonstrated that in Examples 60 to 83 containing certain amounts of fluoride ions, the purity of the hexafluoropropene trimer remained high even after long-term storage at high temperatures.

- 5 [0133] The compounds represented by the formulae (I), (II), and (III) thus obtained were mixed in such a manner that the ratio between the compounds represented by the formulae (I), (II), and (III) became as specified in the table below, and thereby trimer mixtures 5 to 6 were obtained.

[0134] [Table 9]

Trimer mixture	Formula (I)	Formula (II)	Formula (III)
5	3.0 mass%	33.0 mass%	64.0 mass%
6	99.5 mass%	0.2 mass%	0.3 mass%

[0135] (Production of Hexafluoropropene Tetramer)

(Production Example 4)

Based on a method described in Tetrahedron Lett. 1974, 24, 2129-2132, a
5 hexafluoropropene tetramer was obtained.

[0136] By mixing the trimer mixture 5 or 6 and the hexafluoropropene tetramer
obtained in Production Example 4, it is possible to obtain the composition according to
the present disclosure.

[0137] <Evaluation Method>

10 (Boiling Point)

The boiling point was measured by DSC, as a temperature at which an
endothermic peak was observed while the temperature was being raised from 25°C at
5°C/minute.

[0138] (Kinematic Viscosity)

15 The kinematic viscosity was a value at 25°C measured in accordance with JIS K
2283 with an Ubbelohde viscometer.

[0139] (Dielectric Constant)

The dielectric constant was a value at a frequency of 1 kHz measured by a
capacitance method in an environment at a temperature of 25°C and a humidity of 60%.

20 [0140] (Examples 84 to 85)

The trimer mixture 5 and the hexafluoropropene tetramer were mixed in a
weight ratio of 91:9 (Example 84), and the trimer mixture 6 and the hexafluoropropene
tetramer were mixed in a weight ratio of 91:9 (Example 85). Physical properties were
measured and results are given in Table 2.

[0141] [Table 10]

	Boiling point (°C)	Dielectric constant	Kinematic viscosity (cSt)
Trimer mixture 5	115	2.0	1.4
Trimer mixture 6	107	2.0	0.9

[0142] <Examples 86 to 87>

In Examples 86 to 87, a pre-purification composition to be subjected to purification procedure (Composition 1 containing both an HFP trimer and conductive substances) was prepared. Specifically, 750 g of DMF and 7.2 g of cesium fluoride were placed and hermetically sealed in an autoclave made of SUS. After air was evacuated from the autoclave, 2268 g of hexafluoropropylene was added over 4.5 hours while the temperature inside the autoclave was maintained at 70 to 110°C. From the reaction liquid thus obtained, the lower layer was separated and rinsed with ultrapure water, and thereby 2219 g of a composition containing an HFP trimer (Composition 1, a pre-purification composition) was obtained. GCFID and GC-MS analyses were carried out, and by an area normalization method, it was checked that the content of the HFP trimer was 87 mass% in the total amount of the composition regarded as 100 mass%, and in the total amount of the HFP trimer regarded as 100 mass%, the contents of the compounds represented by the formulae (I), (II), and (III) according to the present specification were 78 mass%, 9 mass%, and 13 mass%, respectively.

[0143] <Example 88>

In the same manner as in Examples 86 to 87, the compounds represented by the formulae (I), (II), and (III) were isolated and mixed in such a manner that the contents of the compounds represented by the formulae (I), (II), and (III) became 55 mass%, 15 mass%, and 30 mass%, respectively, and thereby 2200 g of a composition was prepared.

[0144] <Example 89>

In the same manner as in Examples 86 to 87, the compounds represented by the formulae (I), (II), and (III) were isolated and mixed in such a manner that the contents of the compounds represented by the formulae (I), (II), and (III) became 3 mass%, 33 mass%, and 64 mass%, respectively, and thereby 2200 g of a composition was

prepared.

[0145] <Examples 90>

In the same manner as in Examples 86 to 87, the compounds represented by the formulae (I), (II), and (III) were isolated and mixed in such a manner that the contents of the compounds represented by the formulae (I), (II), and (III) became 90 mass%, 5 mass%, and 5 mass%, respectively, and thereby 2200 g of a composition was prepared.

[0146] (Measurement of Contents of Metals and Metal Ions in Pre-Purification Composition)

The content of conductive substances (specifically, in the present examples, the content of metals and metal ions) in the pre-purification composition was determined by the procedure described below with the use of an inductively coupled plasma mass spectrometry instrument (ICP-MS) (The same applies to the post-purification composition). The measured values are given in Table 1 below.

(1) The pre-purification composition in an amount of 2000 g was poured into a polytetrafluoroethylene (PTFE) beaker, and the beaker was placed on a hot plate at 250°C to let volatile components be volatilized.

(2) Nitric acid for trace amount analysis (manufactured by Wako Pure Chemical Industries) having a concentration of 70% was diluted with ultrapure water to form nitric acid having a concentration of about 4 mass%, and about 50 mL of the resultant was placed in the PTFE beaker.

(3) The PTFE beaker was placed on a hot plate at 150°C for 1 hour to let the metal remaining in the beaker dissolve.

(4) The amount of metal ions dissolved in nitric acid was measured on an ICP-MS. The amount of metal ions in nitric acid, the amount of nitric acid, and the amount of volatilized solution were used to calculate the content of metals and metal ions in the solution.

(5) All the procedures related to the above-mentioned measurement were carried out inside a Class 10000 clean room in which a KOACH table manufactured by Koken Ltd. was installed for ensuring a work environment equivalent to Class 1.

[0147] (Measurement of Number of Insoluble Matter (Fine Particles) in Pre-Purification Composition)

5 The number of particles having particle sizes of 5.0 μm or more, the number of particles having particle sizes of not less than 1.0 μm and less than 5.0 μm , the number of particles having particle sizes of not less than 0.5 μm and less than 1.0 μm , and the number of particles having particle sizes of less than 0.3 μm and less than 0.5 μm in the pre-purification composition were measured with a liquid particle counter ("KL-22" manufactured by RION) at a temperature of 23°C (The same applies to the post-purification composition). All the procedures related to the above-mentioned measurement were carried out inside a Class 10000 clean room in which a KOACH table manufactured by Koken Ltd. was installed for ensuring a work environment equivalent to Class 1.

[0148] (Measurement of Boiling Point, Coagulation Point, and Kinematic Viscosity of Pre-Purification Composition)

15 The boiling point was measured by DSC, as a temperature at which an endothermic peak was observed while the temperature was being raised from 25°C at 5°C/min. The coagulation point was measured by DSC, as a temperature at which an endothermic peak was observed while the temperature was being raised at 5°C/min after lowered with liquid nitrogen to -150°C or less (the temperature at which solidification was observed). The kinematic viscosity was measured in accordance with JIS K 2283 with an Ubbelohde viscometer. The measured values are given in Table 1 below.

[0149] (Purification Treatment)

25 As the filtration apparatus, a multi-stage filtration apparatus was prepared that consisted of a required number of required units connected in series where each unit was formed of one filter contained in one vessel.

[0150] The pre-purification composition was placed in a pressurized vessel and cooled to -5°C or less, and then in a highly pure argon atmosphere, with the use of the above-mentioned filtration apparatus, pressure filtration was carried out to 0.02 MPa to form a

post-purification composition. The filters used here in the units of the filtration apparatus were:

Filter A ("IonKleen SL" manufactured by Nihon Pall, with filtration area of 0.58 m²),

5 Filter B ("Ultiplex P-Nylon" manufactured by Nihon Pall, with pore size of 0.15 µm and filtration area of 1.2 m²), and

Filter C ("Ultiplex P-Nylon" manufactured by Nihon Pall, with pore size of 40 nm and filtration area of 1.2 m²)

and these were used as specified in Table 1. Results are given in Table 1.

10 [0151] From the results in Table 1, it is indicated that by subjecting the pre-purification composition to a certain purification treatment, it is possible to reduce the content of conductive substances (metals and metal ions, in particular).

[0152] [Table 11]

Item				Ex. 86	Ex. 87	Ex. 88	Ex. 89	Ex. 90
Raw material	Filtration raw material		-	6FT	6FT	6FT	6FT	6FT
			T-1	78	78	52	8	90
			T-2	9	9	16	43	5
			T-3	13	13	32	49	5
	Pre-purification raw material		-	Comp. 1	Comp. 1	Comp. 2	Comp. 3	Comp. 4
	Boiling point		°C	105	105	110	115	105
	Coagulation point		°C	-90	-90	-90	-90	-90
	Kinematic viscosity	25°C	cSt	0.6	0.6	1.0	1.4	0.4
-40°C		cSt	4.8	4.8	5.2	7.5	3.7	
Filter	Filter type 1		-	Filter A	Filter A	Filter A	Filter A	Filter A
	Filter type 2		-	Filter B	Filter C	Filter C	Filter C	Filter C
Before filtration	Metal amount	Al	ppm	5.1	20.9	15.2	11.2	17
		Ba	ppm	5.1	10.7	7.7	17.3	14.5
		Be	ppm	8.9	5.7	4.8	BDL	BDL
		Bi	ppm	9.4	8.4	6.7	BDL	BDL
		Ca	ppm	5.7	8.3	10.2	7.7	6.2
		Co	ppm	9.6	8.7	10.5	9.9	22
		Cr	ppm	35.2	26	15	20	11.2
		Cu	ppm	11.2	7.2	7.2	8.8	6.3
		Fe	ppm	42.9	53.7	55.2	60.1	47.5
		Ga	ppm	BDL	2.3	BDL	BDL	BDL
		K	ppm	9.3	7.1	6.2	9.9	22.2
		Li	ppm	6.3	BDL	2.2	BDL	2.2
		Mg	ppm	6.3	6.6	2.5	7.1	10.2
		Mn	ppm	6	10.9	6.5	7.9	12.8
		Na	ppm	9.6	9.1	8.2	10.5	15.2
		Ni	ppm	9	10.1	15.5	11.1	7.7
		Pb	ppm	6.3	10.2	BDL	BDL	BDL
		Sr	ppm	9.4	10	BDL	BDL	BDL
		V	ppm	9.9	9.2	10.3	12.1	16.1
		Zn	ppm	14.6	5.1	6.6	8.8	5.3
	Total	ppm	219.8	230.2	190.5	202.4	216.4	
	Particle amount	≥5.0 μm	pieces/mL	8.9	8.4	15.0	18.8	10.1
		5.0-1.0 μm	pieces/mL	20.7	35.8	37.7	40.3	20.0
1.0-0.5 μm		pieces/mL	58.2	84.2	67.8	70.5	83.5	
0.5-0.3 μm		pieces/mL	194.4	236.5	229.9	250.2	210.5	
Dielectric strength		kV	40	41	42	40	40	

[0153] [Table 12]

Item				Ex. 86	Ex. 87	Ex. 88	Ex. 89	Ex. 90
After filtration	Metal amount	Al	ppm	3.61	5.6	6.6	2.3	5.8
		Ba	ppm	BDL	BDL	BDL	BDL	BDL
		Be	ppm	BDL	BDL	BDL	BDL	BDL
		Bi	ppm	0.67	BDL	BDL	BDL	BDL
		Ca	ppm	29.61	3.54	2.26	1.1	4
		Co	ppm	0.01	BDL	BDL	BDL	BDL
		Cr	ppm	BDL	BDL	BDL	BDL	BDL
		Cu	ppm	4.66	0.37	0.5	1.1	0.5
		Fe	ppm	1.65	0.1	0.3	0.5	0.1
		Ga	ppm	BDL	BDL	BDL	BDL	BDL
		K	ppm	BDL	BDL	BDL	BDL	BDL
		Li	ppm	BDL	0.01	BDL	BDL	BDL
		Mg	ppm	6.11	0.19	0.3	0.22	0.2
		Mn	ppm	0.07	BDL	BDL	BDL	BDL
		Na	ppm	BDL	5.19	2.2	1.5	0.1
		Ni	ppm	0.11	BDL	BDL	BDL	BDL
		Pb	ppm	2.39	BDL	BDL	BDL	BDL
		Sr	ppm	0.02	BDL	BDL	BDL	BDL
		V	ppm	BDL	BDL	BDL	BDL	BDL
		Zn	ppm	1.23	0.61	0.7	2.5	2.5
		Total	ppm	50.14	15.61	12.86	9.22	13.2
	Particle amount	≥5.0 μm	pieces/mL	1.9	0.2	0.1	0.7	0.8
		5.0-1.0 μm	pieces/mL	13.0	2.4	2.7	1.1	2.1
		1.0-0.5 μm	pieces/mL	43.8	5.4	5.9	7.9	5.2
		0.5-0.3 μm	pieces/mL	85.5	3.0	4.2	5.5	2.1
	Dielectric strength		kV	60	67	65	70	69

*In Table, BDL represents below detection limits.

CLAIMS

[Claim 1] A composition comprising:

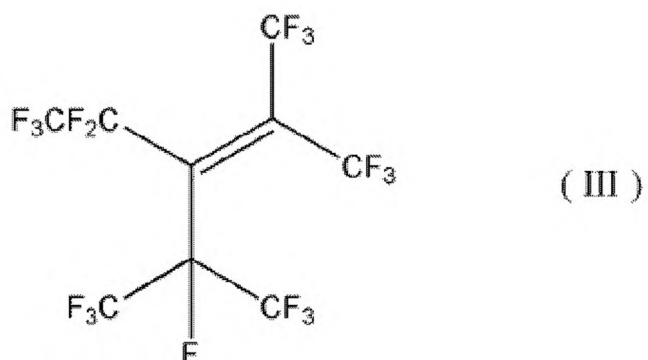
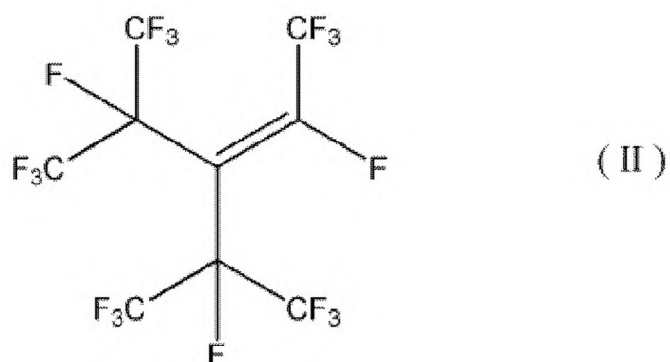
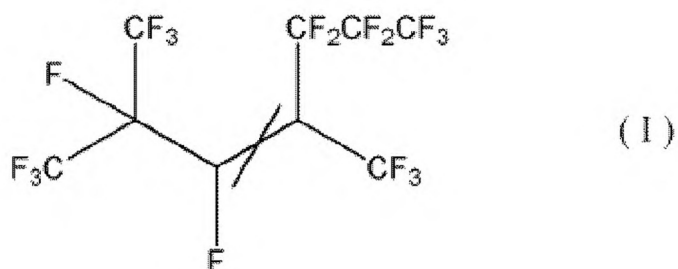
a hexafluoropropene trimer represented by C_9F_{18} ; and

- 5 (i) C_mF_{2m} and/or $C_nF_{(2n-2)}$ where m is an integer of 4 to 12 except 9 and n is an integer of 4 to 12, (ii) water where a content of the water relative to a total amount of the hexafluoropropene trimer represented by C_9F_{18} regarded as 100 parts by mass is from 0.0001 to 0.1 parts by mass, and/or (iii) fluoride ions where a content of the fluoride ions relative to the total amount of the hexafluoropropene trimer represented
10 by C_9F_{18} regarded as 100 parts by mass is from 0.0000001 to 5 parts by mass.

[Claim 2] The composition according to claim 1, wherein the

hexafluoropropene trimer includes at least one selected from the group consisting of compounds represented by formulae (I) to (III) below.

[Chem. 1]



[Claim 3] The composition according to claim 2, wherein a content of the
5 compound represented by the formula (I) relative to the total amount of the
hexafluoropropene trimer is 85 mass% or more.

[Claim 4] The composition according to claim 2, wherein a content of the
compound represented by the formula (I) relative to the total amount of the

hexafluoropropene trimer is less than 85 mass%.

[Claim 5] The composition according to claim 2, wherein a content of the compound represented by the formula (I) relative to the total amount of the
5 hexafluoropropene trimer is not less than 50 mass% and less than 85 mass%.

[Claim 6] The composition according to claim 1, comprising the hexafluoropropene trimer represented by C_9F_{18} and (i) C_mF_{2m} and/or $C_nF_{(2n-2)}$ where m is an integer of 4 to 12 except 9 and n is an integer of 4 to 12.
10

[Claim 7] The composition according to claim 1, wherein m is an integer of 6 to 11 except 9 and n is an integer of 6 to 11.

[Claim 8] The composition according to claim 1, wherein a content of C_mF_{2m} and/or $C_nF_{(2n-2)}$ relative to the total amount of the hexafluoropropene trimer represented
15 by C_9F_{18} regarded as 100 parts by mass is from 0.0001 to 10 parts by mass.

[Claim 9] The composition according to claim 1, comprising the hexafluoropropene trimer represented by C_9F_{18} and (ii) water.
20

[Claim 10] The composition according to claim 1, comprising the hexafluoropropene trimer represented by C_9F_{18} and (iii) fluoride ions.

[Claim 11] The composition according to claim 1, further comprising a
25 hexafluoropropene tetramer represented by $C_{12}F_{24}$, wherein a content of the hexafluoropropene trimer relative to a total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer is 80 mass% or more.

[Claim 12] The composition according to claim 11, wherein the content of the

hexafluoropropene trimer relative to the total amount of the hexafluoropropene trimer and the hexafluoropropene tetramer is from 90 mass% to 99.99 mass%.

5 [Claim 13] The composition according to claim 11, wherein the hexafluoropropene tetramer includes 1,1,1,2,5,6,6,6-octafluoro-2,3,5-tris(trifluoromethyl)-4-(perfluoropropyl-2-yl)-3-hexene.

10 [Claim 14] The composition according to claim 1, further comprising a conductive substance, wherein a content of the conductive substance is 100 ppm by mass or less.

15 [Claim 15] The composition according to claim 1, further comprising a conductive substance, wherein a content of insoluble matter of 5 μm or more is 10 pieces/mL or less.