

DESCRIPTION

Title of Invention: COMPOSITION CONTAINING HEXAFLUOROPROPENE

Technical Field

[0001]

5 The present disclosure relates to a hexafluoropropene-containing composition.

Background Art

[0002]

10 PLT 1 discloses a method of etching a silicon-based material such as a silicon oxide film and/or a low-dielectric constant film containing silicon by using hexafluoropropene.

Citation List

15 Patent Literature

[0003]

 PTL 1: WO0221586-A1

Summary of Invention

20 Technical Problem

[0004]

 The present disclosure aims to provide a novel hexafluoropropene-containing composition.

25 Solution to Problem

[0005]

 The present disclosure encompasses the following features.

[0006]

30 Item 1.

 A hexafluoropropene-containing composition comprising:

 (1) hexafluoropropene;

 (2) hexafluorocyclopropane; and

 (3) at least one component selected from the group

35 consisting of oxygen, hydrogen fluoride, and hydrogen chloride,

wherein, based on the total amount of the composition,

when the composition contains oxygen as (3), the amount of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by volume or less,

5 when the composition contains hydrogen fluoride as (3), the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less, or

when the composition contains hydrogen chloride as (3), the amount of the hydrogen chloride (3) is 0.05 ppm by volume or
10 more and 5 ppm by volume or less.

[0007]

Item 2.

The composition according to Item 1,
wherein, based on the total amount of the composition,

15 the hexafluoropropene (1) is present at 95 vol% to 99.99999 vol%, and

the hexafluorocyclopropane (2) is present at 1 ppm by volume to 1,000 ppm by volume.

[0008]

20 Item 3.

A method for storing a hexafluoropropene-containing composition,

the composition comprising:

(1) hexafluoropropene,
25 (2) hexafluorocyclopropane, and
(3) at least one component selected from the group consisting of oxygen, hydrogen fluoride, and hydrogen chloride, wherein, based on the total amount of the composition,

when the composition contains oxygen as (3), the amount
30 of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by volume or less,

when the composition contains hydrogen fluoride as (3), the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less, or

35 when the composition contains hydrogen chloride as (3),

the amount of the hydrogen chloride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less.

[0009]

Item 4.

5 The method according to Item 3,

wherein, based on the total amount of the composition,

the hexafluoropropene (1) is present at 95 vol% to 99.99999 vol%, and

10 the hexafluorocyclopropane (2) is present at 1 ppm by volume to 1,000 ppm by volume.

[0010]

Item 5.

A method for suppressing a decrease in the purity of hexafluoropropene in a hexafluoropropene-containing composition,
15 the composition comprising:

(1) hexafluoropropene;

(2) hexafluorocyclopropane; and

(3) at least one component selected from the group consisting of oxygen, hydrogen fluoride, and hydrogen chloride,
20 wherein, based on the total amount of the composition, when the composition contains oxygen as (3), the amount of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by volume or less,

25 when the composition contains hydrogen fluoride as (3), the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less, or

when the composition contains hydrogen chloride as (3), the amount of the hydrogen chloride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less.

30 [0011]

Item 6.

The method according to Item 5,

wherein, based on the total amount of the composition,

35 the hexafluoropropene (1) is present at 95 vol% to 99.99999 vol%, and

the hexafluorocyclopropane (2) is present at 1 ppm by volume to 1,000 ppm by volume.

[0012]

The present disclosure discloses a method for stably
5 transporting a composition containing hexafluoropropene (HFP) and
hexafluorocyclopropane ($c\text{-C}_3\text{F}_6$). According to the present
disclosure, the oxygen concentration, hydrogen fluoride
concentration, or hydrogen chloride concentration in the HFP-
containing composition is adjusted, which makes it possible to
10 suppress the decomposition of HFP or $c\text{-C}_3\text{F}_6$ and suppress a
decrease in the purity of HFP or $c\text{-C}_3\text{F}_6$ during transportation of
the HFP-containing composition in cylinders or the like, whereby
storage stability of the HFP-containing composition can be
maintained.

15 [0013]

The HFP-containing composition of the present
disclosure contains $c\text{-C}_3\text{F}_6$ and has improved storage stability. The
composition can be stably transported by suppressing the
decomposition of HFP or $c\text{-C}_3\text{F}_6$ even under harsh marine
20 transportation conditions in containers or the like (e.g.,
conditions such as temperature increase during transportation).

Advantageous Effects of Invention

[0014]

25 The present disclosure can provide a novel
hexafluoropropene-containing composition.

Description of Embodiments

[0015]

30 In the present specification, the terms "include,"
"contain," and variations thereof express concepts that encompass
all of "comprise," "consist essentially of," and "consist of." In
the present specification, when a numerical range is expressed as
"A to B," the expression means A or more and B or less. In the
35 present specification, when expressions such as "part(s)" and "%"

are used, these mean parts by mass, parts by weight, mass%, or wt%.

[0016]

[1] Hexafluoropropene-containing composition

5 The hexafluoropropene (HFP)-containing composition of the present disclosure comprises:

- (1) hexafluoropropene (HFP);
- (2) hexafluorocyclopropane ($c\text{-C}_3\text{F}_6$); and
- (3) at least one component selected from the group

10 consisting of oxygen (O_2), hydrogen fluoride (HF), and hydrogen chloride (HCl)

wherein, based on the total amount of the composition,

 when the composition contains oxygen as (3), the amount of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by
15 volume or less,

 when the composition contains hydrogen fluoride as (3), the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less, or

 when the composition contains hydrogen chloride as (3),
20 the amount of the hydrogen chloride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less.

[0017]

 Preferably, in the HFP-containing composition of the present disclosure,

25 based on the total amount of the composition,
 the HFP (1) is present at 95 vol% to 99.99999 vol%, and
 the $c\text{-C}_3\text{F}_6$ (2) is present at 1 ppm by volume to 1,000 ppm by volume.

[0018]

30 The present disclosure discloses a method for stably transporting a composition containing HFP and $c\text{-C}_3\text{F}_6$. According to the present disclosure, the oxygen concentration, hydrogen fluoride concentration, or hydrogen chloride concentration in the HFP-containing composition is adjusted, which makes it possible
35 to suppress the decomposition of HFP or $c\text{-C}_3\text{F}_6$ and suppress a

decrease in the purity of HFP or $c\text{-C}_3\text{F}_6$ during transportation of the HFP-containing composition in cylinders or the like, whereby the storage stability of the HFP-containing composition can be maintained.

5 [0019]

The HFP-containing composition of the present disclosure contains $c\text{-C}_3\text{F}_6$, has improved storage stability, and can be stably transported by suppressing the decomposition of HFP or $c\text{-C}_3\text{F}_6$ even under harsh marine transportation conditions in
10 containers or the like (e.g., conditions such as temperature increase during transportation).

[0020]

(1) Hexafluoropropene

The HFP-containing composition of the present
15 disclosure contains (1) hexafluoropropene ($\text{CF}_3\text{-CF=CF}_2$, also known as hexafluoropropylene, HFP) as a first component.

[0021]

The HFP-containing composition of the present disclosure contains the first component "(1) HFP" as a main
20 component. For improved storage stability and stable transportation by suppressing the decomposition of HFP or $c\text{-C}_3\text{F}_6$ even under harsh marine transportation conditions in containers or the like (e.g., conditions such as temperature increase during transportation), the amount of HFP in the HFP-containing
25 composition based on the total amount (gas volume) of the HFP-containing composition is preferably 95 vol% to 99.99999 vol%, more preferably 95 vol% to 99.999 vol% or 96 vol% to 99.99 vol%, even more preferably 97 vol% to 99.95 vol%, and particularly preferably 98 vol% to 99.9 vol%.

30 [0022]

(2) Hexafluorocyclopropane

The HFP-containing composition of the present disclosure contains (2) hexafluorocyclopropane ($c\text{-C}_3\text{F}_6$) as a second component.

35 [0023]

The HFP-containing composition of the present disclosure contains hexafluorocyclopropane ($\text{c-C}_3\text{F}_6$) as a second component. Thus, the composition has improved storage stability and can be stably transported by suppressing the decomposition of HFP or its dimer and/or trimer even under harsh marine transportation conditions in containers or the like (e.g., conditions such as temperature increase during transportation).
[0024]

For improved storage stability and stable transportation by suppressing the decomposition of HFP or $\text{c-C}_3\text{F}_6$ even under harsh marine transportation conditions in containers or the like (e.g., conditions such as temperature increase during transportation), the amount of $\text{c-C}_3\text{F}_6$ in the HFP-containing composition based on the total amount (gas volume) is preferably 1 ppm by volume (ppm volume) to 1,000 ppm by volume (ppm volume), more preferably 5 ppm by volume to 500 ppm by volume, even more preferably 5 ppm by volume to 300 ppm by volume, and particularly preferably 5 ppm by volume to 200 ppm by volume.
[0025]

(3) Oxygen, hydrogen fluoride, and hydrogen chloride

The HFP-containing composition of the present disclosure contains (3), as a third component, at least one component selected from the group consisting of oxygen (O_2), hydrogen fluoride (HF), and hydrogen chloride (HCl).

[0026]

(a) Oxygen (O_2)

The HFP-containing composition of the present disclosure contains (3) oxygen (O_2) as a third component.
[0027]

The amount of oxygen (O_2) in the HFP-containing composition based on the total amount (gas volume) of the HFP-containing composition is 0.05 ppm by volume or more and 10 ppm by volume or less. The O_2 concentration in the HFP-containing composition is adjusted within the above range, which makes it possible to suppress the decomposition of HFP or $\text{c-C}_3\text{F}_6$ and

suppress a decrease in the purity of HFP or $\text{c-C}_3\text{F}_6$ in the HFP-containing composition during transportation of the HFP-containing composition in cylinders or the like, whereby the storage stability of the HFP-containing composition can be well maintained. The HFP-containing composition can be stably transported by suppressing the decomposition of HFP or $\text{c-C}_3\text{F}_6$ even under harsh marine transportation conditions in containers or the like (e.g., conditions such as temperature increase during transportation).

10 [0028]

The amount of oxygen (O_2) in the HFP-containing composition based on the total amount (gas volume) of the HFP-containing composition is preferably 0.1 ppm by volume (ppm volume) to 10 ppm by volume (ppm volume), and more preferably 0.1 ppm by volume to 5 ppm by volume.

15 [0029]

(b) Hydrogen fluoride (HF)

The HFP-containing composition of the present disclosure contains (3) hydrogen fluoride (HF) as a third component.

20 [0030]

The amount of hydrogen fluoride (HF) in the HFP-containing composition based on the total amount (gas volume) of the HFP-containing composition is 0.05 ppm by volume or more and 5 ppm by volume or less. The HF concentration in the HFP-containing composition is adjusted within the above range, which makes it possible to suppress the decomposition of HFP or $\text{c-C}_3\text{F}_6$ and suppress a decrease in the purity of HFP or $\text{c-C}_3\text{F}_6$ in the HFP-containing composition during transportation of the HFP-containing composition in cylinders or the like, whereby the storage stability of the HFP-containing composition can be well maintained. The HFP-containing composition can be stably transported by suppressing the decomposition of HFP or $\text{c-C}_3\text{F}_6$ even under harsh marine transportation conditions in containers or the like (e.g., conditions such as temperature increase during

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transportation).

[0031]

The amount of hydrogen fluoride (HF) in the HFP-containing composition based on the total amount (gas volume) of the HFP-containing composition is preferably 0.1 ppm by volume (ppm volume) to 5 ppm by volume (ppm volume), more preferably 0.1 ppm by volume to 3 ppm by volume, and even more preferably 0.1 ppm by volume to 1 ppm by volume.

[0032]

10 (c) Hydrogen chloride (HCl)

The HFP-containing composition of the present disclosure contains (3) hydrogen chloride (HCl) as a third component.

[0033]

15 The amount of hydrogen chloride (HCl) in the HFP-containing composition based on the total amount (gas volume) of the HFP-containing composition is 0.05 ppm by volume or more and 5 ppm by volume or less. The HCl concentration in the HFP-containing composition is adjusted within the above range, which makes it possible to suppress the decomposition of HFP or $\text{c-C}_3\text{F}_6$ and suppress a decrease in the purity of HFP or $\text{c-C}_3\text{F}_6$ in the HFP-containing composition during transportation of the HFP-containing composition in cylinders or the like, whereby the storage stability of the HFP-containing composition can be well maintained. The HFP-containing composition can be stably transported by suppressing the decomposition of HFP or $\text{c-C}_3\text{F}_6$ even under harsh marine transportation conditions in containers or the like (e.g., conditions such as temperature increase during transportation).

30 [0034]

The amount of hydrogen chloride (HCl) in the HFP-containing composition based on the total amount (gas volume) of the HFP-containing composition is preferably 0.1 ppm by volume (ppm volume) to 5 ppm by volume (ppm volume), more preferably 0.1 ppm by volume to 3 ppm by volume, and even more preferably 0.1

ppm by volume to 1 ppm by volume.

[0035]

[2] Method for storing a hexafluoropropene-containing composition

The present disclosure encompasses a method for storing
5 a hexafluoropropene (HFP)-containing composition and a method for
suppressing a decrease in the purity of HFP in an HFP-containing
composition.

[0036]

In the method for storing an HFP-containing composition
10 or the method for suppressing a decrease in the purity of HFP,
the HFP-containing composition comprises:

- (1) hexafluoropropene (HFP);
- (2) hexafluorocyclopropane ($\text{c-C}_3\text{F}_6$); and
- (3) at least one component selected from the group
15 consisting of oxygen, hydrogen fluoride, and hydrogen chloride,
wherein, based on the total amount of the composition,

when the composition contains oxygen as (3), the amount
of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by
volume or less,

20 when the composition contains hydrogen fluoride as (3),
the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or
more and 5 ppm by volume or less, or

when the composition contains hydrogen chloride as (3),
the amount of the hydrogen chloride (3) is 0.05 ppm by volume or
25 more and 5 ppm by volume or less.

[0037]

Preferably, in the HFP-containing composition,
based on the total amount of the composition,
the HFP (1) is present at 95 vol% to 99.99999 vol%, and
30 the $\text{c-C}_3\text{F}_6$ (2) is present at 1 ppm by volume to 1,000
ppm by volume.

[0038]

The details of the HFP-containing composition are as
described in section [1] Hexafluoropropene-containing
35 composition.

[0039]

The present disclosure discloses a method for stably transporting a composition containing HFP and $c\text{-C}_3\text{F}_6$. According to the present disclosure, the oxygen (O_2) concentration, hydrogen fluoride (HF) concentration, or hydrogen chloride (HCl) concentration in the HFP-containing composition is adjusted, which makes it possible to suppress the decomposition of HFP or $c\text{-C}_3\text{F}_6$ and suppress a decrease in the purity of HFP or $c\text{-C}_3\text{F}_6$ in the HFP-containing composition during transportation of the HFP-containing composition in cylinders or the like, whereby the storage stability of the HFP-containing composition can be well maintained.

[0040]

The HFP-containing composition of the present disclosure contains $c\text{-C}_3\text{F}_6$ and suppresses the generation of an HFP-derived decomposed product or a $c\text{-C}_3\text{F}_6$ -derived decomposed product. Thus, the composition has improved storage stability and can be stably transported by suppressing the decomposition of HFP or $c\text{-C}_3\text{F}_6$ even under harsh marine transportation conditions in containers or the like (e.g., conditions such as temperature increase during transportation).

[0041]

[3] Application of hexafluoropropene-containing composition

The present disclosure encompasses a method for etching a semiconductor manufacturing substrate using, as an etching gas, the hexafluoropropene (HFP)-containing composition of the present disclosure, which is supplied as a plasma-generating gas.

[0042]

A silicon oxide film (SiO_2 film) or a silicon-based material having a silicon nitride film (SiN film) formed thereon can be etched using a gas plasma of the HFP-containing composition (etching gas). The silicon-based material to be etched is preferably a semiconductor manufacturing substrate.

[0043]

When hole etching is performed in a silicon oxide film

(SiO₂ film) or a silicon-based material having a silicon nitride film (SiN film) formed thereon using a gas plasma generated from the HFP-containing composition, good etching can be achieved in which the processed hole shape is well maintained. The conditions for the etching method (in particular, the dry etching method) are the same as those for the conventional method.

Examples

[0044]

The present invention is specifically described below with reference to Examples and Comparative Examples, which do not limit the present invention.

[0045]

[1-1] Storage stability of hexafluoropropene-containing composition

Example 1-1

Hexafluoropropene: HFP

Hexafluorocyclopropane: c-C₃F₆

Oxygen: O₂

According to a conventional method, HFP was purified, the c-C₃F₆ concentration was adjusted to predetermined levels (150 ppm by volume, 50 ppm by volume, and 5 ppm by volume), and the O₂ concentration was adjusted to predetermined levels (100 ppm by volume, 10 ppm by volume, 5 ppm by volume, and 1 ppm by volume), whereby HFP-containing compositions were prepared.

[0046]

The c-C₃F₆ concentration (ppm by volume) in each HFP-containing composition was analyzed by gas chromatography (GC analysis).

[0047]

The O₂ concentration (ppm by volume) in each HFP-containing composition was analyzed by GC.

[0048]

The HFP-containing compositions were stored at a constant temperature (20°C or 50°C). After fixed periods (30

days, 90 days, and 180 days), the gas phase was extracted, and a hexafluoropropene (HFP)-derived decomposed product was analyzed by GC analysis (ppm by volume).

[0049]

5 [Table 1]

Table 1: Effect to suppress generation of decomposed product					(4) HFP, c-C ₃ F ₆ , O ₂				
c-C ₃ F ₆ 150 ppm by volume					c-C ₃ F ₆ 150 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	Amount of decomposed product (ppm by volume)			Temperature	50°C	Amount of decomposed product (ppm by volume)		
Oxygen concentration	100	12	18	32	Oxygen concentration	100	21	43	87
(ppm by volume)	10	N.D.	5	8	(ppm by volume)	10	4	10	22
	5	N.D.	N.D.	N.D.		5	N.D.	N.D.	N.D.
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
c-C ₃ F ₆ 50 ppm by volume					c-C ₃ F ₆ 50 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	Amount of decomposed product (ppm by volume)			Temperature	50°C	Amount of decomposed product (ppm by volume)		
Oxygen concentration	100	8	12	28	Oxygen concentration	100	18	32	66
(ppm by volume)	10	N.D.	4	6	(ppm by volume)	10	3	6	18
	5	N.D.	N.D.	N.D.		5	N.D.	N.D.	N.D.
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
c-C ₃ F ₆ 5 ppm by volume					c-C ₃ F ₆ 5 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	Amount of decomposed product (ppm by volume)			Temperature	50°C	Amount of decomposed product (ppm by volume)		
Oxygen concentration	100	6	8	13	Oxygen concentration	100	9	12	23
(ppm by volume)	10	N.D.	ND	3	(ppm by volume)	10	N.D.	2	8
	5	N.D.	N.D.	N.D.		5	N.D.	N.D.	N.D.
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.

[0050]

The compositions containing HFP and c-C₃F₆(150 ppm by volume, 50 ppm by volume, and 5 ppm by volume) of the Examples in which the O₂ concentration was adjusted (10 ppm by volume or less) successfully suppressed the generation an HFP-derived decomposed product or a c-C₃F₆-derived decomposed product even after the

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fixed periods (30 days, 90 days, and 180 days) of storage at a constant temperature (20°C or 50°C).

[0051]

[1-2] Study of lower limit amount of oxygen (O₂) in composition

5 **Example 1-2**

According to a conventional method, a composition containing HFP and c-C₃F₆ (5 ppm by volume) was prepared, and the O₂ concentration was adjusted to predetermined levels (0.1 ppm by volume, 0.01 ppm by volume), whereby compositions containing O₂,
10 HFP, and c-C₃F₆ were prepared.

[0052]

The O₂ concentration (ppm by volume) in each composition containing HFP and c-C₃F₆ (5 ppm by volume) was analyzed by GC.

15 [0053]

The compositions containing HFP and c-C₃F₆ (5 ppm by volume) were stored at a constant temperature (20°C, 50°C). After fixed periods (30 days, 90 days, and 180 days), the gas phase was extracted and analyzed by GC (ppm by volume).

20 [0054]

[Table 2]

Table 2: Study of lower limit amount of oxygen				
O ₂ concentration (ppm by volume)		Decomposed product (ppm by volume)		
20°C		30 days	90 days	180 days
Example 1-2-1	0.1	N.D.	N.D.	N.D.
Comparative Example 1-1-1	0.01	0.01	0.02	0.05
O ₂ concentration (ppm by volume)		Decomposed product (ppm by volume)		
50°C		30 days	90 days	180 days
Example 1-2-2	0.1	N.D.	N.D.	N.D.
Comparative Example 1-1-2	0.01	0.01	0.03	0.06

[0055]

In the composition in which the O₂ concentration was
25 0.1 ppm by volume, no decomposed product was observed. In the composition in which the O₂ concentration was 0.01 ppm by volume, a decomposed product was detected.

[0056]

[2-1] Storage stability of hexafluoropropene-containing composition

Example 2-1

5 Hexafluoropropene: HFP

Hexafluorocyclopropane: $c\text{-C}_3\text{F}_6$

Hydrogen fluoride: HF

According to a conventional method, HFP was purified, the $c\text{-C}_3\text{F}_6$ concentration was adjusted to predetermined levels (150
10 ppm by volume, 50 ppm by volume, and 5 ppm by volume), and the HF concentration was adjusted to predetermined levels (10 ppm by volume, 5 ppm by volume, 1 ppm by volume, and 0.1 ppm by volume), whereby HFP-containing compositions were prepared.

[0057]

15 The $c\text{-C}_3\text{F}_6$ concentration (ppm by volume) in each HFP-containing composition was analyzed by gas chromatography (GC analysis).

[0058]

The HF concentration (ppm by volume) in each HFP-
20 containing composition was analyzed by a Fourier transform infrared spectrophotometer (FT-IR) to measure the initial concentration.

[0059]

The HFP-containing compositions were stored at a
25 constant temperature (20°C or 50°C). After fixed periods (30 days, 90 days, and 180 days), the gas phase was extracted, and a hexafluoropropene (HFP)-derived decomposed product was analyzed by GC analysis (ppm by volume).

30

35

[0060]

[Table 3]

Table 3: Effect to suppress generation of decomposed product					(5) HFP, c-C ₃ F ₆ , HF				
c-C ₃ F ₆ 150 ppm by volume					c-C ₃ F ₆ 150 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	Amount of decomposed product (ppm by volume)			Temperature	50°C	Amount of decomposed product (ppm by volume)		
HF concentration (ppm by volume)	10	11	32	67	HF concentration (ppm by volume)	10	20	56	76
	5	N.D.	14	44		5	11	32	45
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
	0.1	N.D.	N.D.	N.D.		0.1	N.D.	N.D.	N.D.
c-C ₃ F ₆ 50 ppm by volume					c-C ₃ F ₆ 50 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	Amount of decomposed product (ppm by volume)			Temperature	50°C	Amount of decomposed product (ppm by volume)		
HF concentration (ppm by volume)	10	3	21	33	HF concentration (ppm by volume)	10	15	29	47
	5	N.D.	10	24		5	7	18	32
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
	0.1	N.D.	N.D.	N.D.		0.1	N.D.	N.D.	N.D.
c-C ₃ F ₆ 5 ppm by volume					c-C ₃ F ₆ 5 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	Amount of decomposed product (ppm by volume)			Temperature	50°C	Amount of decomposed product (ppm by volume)		
HF concentration (ppm by volume)	10	4	7	9	HF concentration (ppm by volume)	10	11	16	29
	5	N.D.	ND	3		5	N.D.	6	11
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
	0.1	N.D.	N.D.	N.D.		0.1	N.D.	N.D.	N.D.

[0061]

- 5 The compositions containing HFP and c-C₃F₆ (150 ppm by volume, 50 ppm by volume, and 5 ppm by volume) of the Example in which the HF concentration was adjusted (5 ppm by volume or less) successfully suppressed the generation of an HFP-derived decomposed product or a c-C₃F₆-derived decomposed product even
- 10 after the fixed periods (30 days, 90 days, and 180 days) of storage at a constant temperature (20°C or 50°C).

[0062]

[2-2] Study of lower limit amount of hydrogen fluoride (HF) in composition

Example 2-2

5 According to a conventional method, a composition containing HFP and $c\text{-C}_3\text{F}_6$ (5 ppm by volume) was prepared, and the HF concentration was adjusted to predetermined levels (0.1 ppm by volume, 0.01 ppm by volume), whereby compositions containing HF, HFP, and $c\text{-C}_3\text{F}_6$ were prepared.

10 [0063]

The HF concentration (ppm by volume) in each composition containing HFP and $c\text{-C}_3\text{F}_6$ (5 ppm by volume) was analyzed by GC.

[0064]

15 The compositions containing HFP and $c\text{-C}_3\text{F}_6$ (5 ppm by volume) were stored at a constant temperature (20°C, 50°C). After fixed periods (30 days, 90 days, and 180 days), the gas phase was extracted and analyzed by GC (ppm by volume).

[0065]

20 [Table 4]

Table 4: Study of lower limit amount of hydrogen fluoride				
HF concentration (ppm by volume)		Decomposed product (ppm by volume)		
20°C		30 days	90 days	180 days
Example 2-2-1	0.1	N.D.	N.D.	N.D.
Comparative Example 2-1-1	0.01	0.1	0.2	0.5
HF concentration (ppm by volume)		Decomposed product (ppm by volume)		
50°C		30 days	90 days	180 days
Example 2-2-2	0.1	N.D.	N.D.	N.D.
Comparative Example 2-1-2	0.01	0.2	0.4	0.6

[0066]

In the composition in which the HF concentration was 0.1 ppm by volume, no decomposed product was observed. In the composition in which the HF concentration was 0.01 ppm by volume, a decomposed product was detected.

25 [0067]

[3-1] Storage stability of hexafluoropropene-containing composition

Example 3-1

Hexafluoropropene: HFP

5 Hexafluorocyclopropane: $c\text{-C}_3\text{F}_6$

Hydrogen chloride: HCl

According to a conventional method, HFP was purified, the $c\text{-C}_3\text{F}_6$ concentration was adjusted to predetermined levels (150 ppm by volume, 50 ppm by volume, and 5 ppm by volume), and the
10 HCl concentration was adjusted to predetermined levels (10 ppm by volume, 5 ppm by volume, 1 ppm by volume, and 0.1 ppm by volume), whereby HFP-containing compositions were prepared.

[0068]

The $c\text{-C}_3\text{F}_6$ concentration (ppm by volume) in each HFP-
15 containing composition was analyzed by gas chromatography (GC analysis).

[0069]

The HCL concentration (ppm by volume) in each HFP-containing composition was analyzed by a Fourier transform
20 infrared spectrophotometer (FT-IR) to measure the initial concentration.

[0070]

The HFP-containing compositions were stored at a constant temperature (20°C or 50°C). After fixed periods (30
25 days, 90 days, and 180 days), the gas phase was extracted, and a hexafluoropropene (HFP)-derived decomposed product was analyzed by GC analysis (ppm by volume).

30

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[0071]

[Table 5]

Table 5: Effect to suppress generation of decomposed product					(6) HFP, c-C ₃ F ₆ , HCL				
c-C ₃ F ₆ 150 ppm by volume					c-C ₃ F ₆ 150 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	<u>Amount of</u> <u>decomposed product</u> <u>(ppm by volume)</u>			Temperature	50°C	<u>Amount of</u> <u>decomposed product</u> <u>(ppm by volume)</u>		
HCl concentration (ppm by volume)	10	14	29	55	HCl concentration (ppm by volume)	10	22	60	86
	5	N.D.	18	49		5	14	39	50
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
	0.1	N.D.	N.D.	N.D.		0.1	N.D.	N.D.	N.D.
c-C ₃ F ₆ 50 ppm by volume					c-C ₃ F ₆ 50 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	<u>Amount of</u> <u>decomposed product</u> <u>(ppm by volume)</u>			Temperature	50°C	<u>Amount of</u> <u>decomposed product</u> <u>(ppm by volume)</u>		
HCl concentration (ppm by volume)	10	5	26	41	HCl concentration (ppm by volume)	10	16	31	58
	5	N.D.	11	29		5	8	20	37
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
	0.1	N.D.	N.D.	N.D.		0.1	N.D.	N.D.	N.D.
c-C ₃ F ₆ 5 ppm by volume					c-C ₃ F ₆ 5 ppm by volume				
Period		30 days	90 days	180 days	Period		30 days	90 days	180 days
Temperature	20°C	<u>Amount of</u> <u>decomposed product</u> <u>(ppm by volume)</u>			Temperature	50°C	<u>Amount of</u> <u>decomposed product</u> <u>(ppm by volume)</u>		
HCl concentration (ppm by volume)	10	6	9	11	HCl concentration (ppm by volume)	10	18	21	33
	5	N.D.	ND	4		5	N.D.	9	15
	1	N.D.	N.D.	N.D.		1	N.D.	N.D.	N.D.
	0.1	N.D.	N.D.	N.D.		0.1	N.D.	N.D.	N.D.

[0072]

5 The compositions containing HFP and c-C₃F₆ (150 ppm by volume, 50 ppm by volume, and 5 ppm by volume) of the Example in which the HCl concentration was adjusted (5 ppm by volume or less) successfully suppressed the generation of an HFP-derived decomposed product or a c-C₃F₆-derived decomposed product even
10 after the fixed periods (30 days, 90 days, and 180 days) of storage at a constant temperature (20°C or 50°C).

[0073]

[3-2] Study of lower limit amount of hydrogen chloride (HCl) in composition

Example 3-2

According to a conventional method, a composition containing HFP and $c\text{-C}_3\text{F}_6$ (5 ppm by volume) was prepared, and the HCL concentration was adjusted to predetermined levels (0.1 ppm by volume, 0.01 ppm by volume), whereby compositions containing HCL, HFP, and $c\text{-C}_3\text{F}_6$ were prepared.

[0074]

The HCL concentration (ppm by volume) in each composition containing HFP and $c\text{-C}_3\text{F}_6$ (5 ppm by volume) was analyzed by GC.

[0075]

The compositions containing HFP and $c\text{-C}_3\text{F}_6$ (5 ppm by volume) were stored at a constant temperature (20°C, 50°C). After fixed periods (30 days, 90 days, and 180 days), the gas phase was extracted and analyzed by GC (ppm by volume).

[0076]

[Table 6]

Table 6: Study of lower limit amount of hydrogen chloride				
HCl concentration (ppm by volume)		Decomposed product (ppm by volume)		
20°C		30 days	90 days	180 days
Example 3-2-1	0.1	N.D.	N.D.	N.D.
Comparative Example 3-1-1	0.01	0.1	0.3	0.5
HCl concentration (ppm by volume)		Decomposed product (ppm by volume)		
50°C		30 days	90 days	180 days
Example 3-2-2	0.1	N.D.	N.D.	N.D.
Comparative Example 3-1-2	0.01	0.2	0.5	0.6

[0077]

In the composition in which the HCl concentration was 0.1 ppm by volume, no decomposed product was observed. In the composition in which the HCl concentration was 0.01 ppm by volume, a decomposed product was detected.

[0078]

[4] Industrial Applicability

The present disclosure discloses a method for stably transporting a composition containing HFP and $c\text{-C}_3\text{F}_6$. According to the present disclosure, the oxygen (O_2) concentration, hydrogen fluoride (HF) concentration, or hydrogen chloride (HCl)

5 concentration in the HFP-containing composition is adjusted, which makes it possible to suppress the decomposition of HFP or $c\text{-C}_3\text{F}_6$ and suppress a decrease in the purity of HFP or $c\text{-C}_3\text{F}_6$ in the HFP-containing composition during transportation of the HFP-containing composition in cylinders or the like, whereby the
10 storage stability of the HFP-containing composition can be well maintained.

[0079]

The HFP-containing composition of the present disclosure contains hexafluorocyclopropane ($c\text{-C}_3\text{F}_6$) and suppresses
15 the generation of an HFP-derived decomposed product or a $c\text{-C}_3\text{F}_6$ -derived decomposed product. The composition has improved storage stability and can be stably transported by suppressing the decomposition of HFP or $c\text{-C}_3\text{F}_6$ even under harsh marine transportation conditions in containers or the like (e.g.,
20 conditions such as temperature increase during transportation).

CLAIMS

[Claim 1]

A hexafluoropropene-containing composition comprising:

(1) hexafluoropropene;

5 (2) hexafluorocyclopropane; and

(3) at least one component selected from the group consisting of oxygen, hydrogen fluoride, and hydrogen chloride, wherein, based on the total amount of the composition,

when the composition contains oxygen as (3), the amount of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by volume or less,

when the composition contains hydrogen fluoride as (3), the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less, or

15 when the composition contains hydrogen chloride as (3), the amount of the hydrogen chloride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less.

[Claim 2]

20 The composition according to claim 1, wherein, based on the total amount of the composition,

the hexafluoropropene (1) is present at 95 vol% to 99.99999 vol%, and

25 the hexafluorocyclopropane (2) is present at 1 ppm by volume to 1,000 ppm by volume.

[Claim 3]

A method for storing a hexafluoropropene-containing composition,

30 the composition comprising:

(1) hexafluoropropene;

(2) hexafluorocyclopropane; and

(3) at least one component selected from the group consisting of oxygen, hydrogen fluoride, and hydrogen chloride, 35 wherein, based on the total amount of the composition,

when the composition contains oxygen as (3), the amount of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by volume or less,

when the composition contains hydrogen fluoride as (3),
5 the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less, or

when the composition contains hydrogen chloride as (3), the amount of the hydrogen chloride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less.

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[Claim 4]

The method according to claim 3,
wherein, based on the total amount of the composition,

the hexafluoropropene (1) is present at 95 vol% to
15 99.99999 vol%, and

the hexafluorocyclopropane (2) is present at 1 ppm by volume to 1,000 ppm by volume.

[Claim 5]

20 A method for suppressing a decrease in the purity of hexafluoropropene in a hexafluoropropene-containing composition, the composition comprising:

(1) hexafluoropropene;

(2) hexafluorocyclopropane; and

25 (3) at least one component selected from the group consisting of oxygen, hydrogen fluoride, and hydrogen chloride, wherein, based on the total amount of the composition,

when the composition contains oxygen as (3), the amount of the oxygen (3) is 0.05 ppm by volume or more and 10 ppm by
30 volume or less,

when the composition contains hydrogen fluoride as (3), the amount of the hydrogen fluoride (3) is 0.05 ppm by volume or more and 5 ppm by volume or less, or

when the composition contains hydrogen chloride as (3),
35 the amount of the hydrogen chloride (3) is 0.05 ppm by volume or

more and 5 ppm by volume or less.

[Claim 6]

The method according to claim 5,

5 wherein, based on the total amount of the composition,

the hexafluoropropene (1) is present at 95 vol% to
99.99999 vol%, and

the hexafluorocyclopropane (2) is present at 1 ppm by
volume to 1,000 ppm by volume.

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