

[DESCRIPTION]

[Title of Invention]

FLUORINE-CONTAINING ALKYL AMMONIUM BORATE COMPOUND AND METHOD
FOR PRODUCING SAME

5 [Technical Field]

[0001]

The present invention relates to a fluorine-containing
alkylammonium borate compound useful as a co-catalyst for the
polymerization of olefin, diene, and acetylene, a composition
10 containing same, and a production method thereof.

[Background Art]

[0002]

Many reports have been conventionally made on the use of
metallocene compound and non-metallocene type metal complex
15 catalysts such as diimine complex, phenoxy complex, and the
like as catalysts for the polymerization of olefins, dienes,
and acetylenes. As co-catalysts used for stabilizing the
cationic active species of these metal complex catalysts,
aluminoxanes such as alkylaluminum, methylaluminoxane (MAO) and
20 the like, Broensted acid salts such as ammonium borate and the
like, and Lewis acid salts such as triphenylcarbenium borate
and the like are used (Non-Patent Document 1).

[0003]

In the catalyst activation reaction by the aforementioned
25 Broensted acid salt, the leaving group on the metal complex
catalyst is protonated and eliminated from the metal complex
catalyst to generate a cationic active species of the metal
complex catalyst, whereby non-coordinating anions derived from
Broensted acid salt stabilize the active species. As the
30 Broensted base constituting the Broensted acid salt, various
borate compounds such as tetrakis(pentafluorophenyl)borate,
which is a non-coordinating anion, and the like have been
reported (Non-Patent Document 1) and, as the Broensted acid,
Broensted acids containing nitrogen, phosphorus, oxygen, and/or
35 sulfur are known (Patent Document 1).

[0004]

As the aforementioned Broensted acid salts, Broensted acid salts (ammonium borates) containing nitrogen such as dimethylanilinium tetrakis(pentafluorophenyl)borate, tri(n-
5 butyl)ammonium tetrakis(pentafluorophenyl)borate, methylpyrrolidinium tetrakis(pentafluorophenyl)borate, and the like are known (Patent Document 2). In the catalyst activation reactions by these ammonium borates, neutral amine compounds are produced due to the loss of proton in the protonation stage.
10 Such neutral amine compound may interact with the cationic active species of the metal complex catalyst, in which case the compound is feared to adversely affect the polymerization reaction.

[0005]

15 In order to reduce the basicity of neutral amine compounds generated in the catalyst activation reaction, N-(pentafluorophenyl)pyrrolidinium tetrakis(pentafluorophenyl)borate and the like have been proposed as co-catalysts (Patent Document 3).

20 [Document List]

[Non-Patent Document]

[0006]

[Non-Patent Document 1]

Chem. Rev. 2000, 100, 1391-1434

25 [Patent Document]

[0007]

[Patent Document 1]

US Patent No. 5132380

[Patent Document 2]

30 WO 2010/014344

[Patent Document 3]

WO 2001/042249

[Summary of Invention]

[Problems to be Solved by the Invention]

35 [0008]

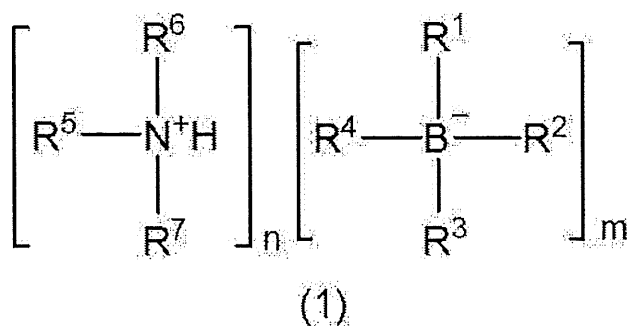
In view of these conventional techniques, the present inventors provide a fluorine-containing alkylammonium borate compound showing, as compared with the prior art, high polymerization activity as a co-catalyst used for the polymerization reactions of olefin, diene, and acetylene by a metal complex catalyst, a composition containing the compound, and an industrial production method thereof.

[Means of Solving the Problems]

[0009]

The present inventors conducted intensive studies and found for the first time that a compound represented by the following formula (1):

[0010]



15

[0011]

wherein

R^1 , R^2 , R^3 , and R^4 are each independently a C_{6-14} aryl group substituted by one or more fluorine atoms or one or more fluoro C_{1-4} alkyl groups,

R^5 is a C_{1-30} alkyl group substituted by one or more substituents selected from the group consisting of a C_{6-14} aryl group substituted by one or more fluorine atoms, and a fluorine atom,

R^6 and R^7 are each independently an optionally substituted C_{1-30} alkyl group, an optionally substituted C_{3-15} cycloalkyl group, or an optionally substituted C_{6-14} aryl group, or

R^6 and R^7 are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group,

n is 1, and

m is 1 or 2

(hereinafter to be also referred to as "the compound of the present invention") shows a high metal complex catalyst

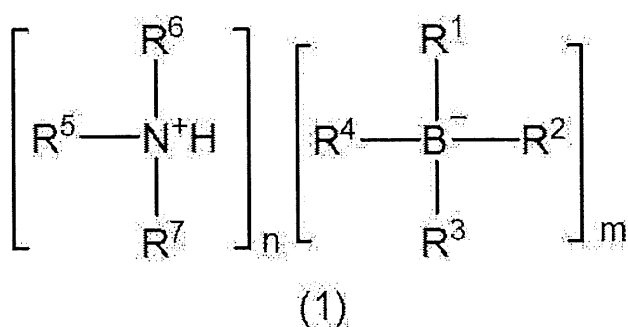
5 activation ability and is useful as a co-catalyst in polymerization reactions of olefin, diene, and acetylene, which resulted in the completion of the present invention.

[0012]

Accordingly, the present invention provides the following.

10 [1] A compound represented by the following formula (1):

[0013]



[0014]

15 wherein

R^1 , R^2 , R^3 , and R^4 are each independently a C_{6-14} aryl group substituted by one or more fluorine atoms or one or more fluoro C_{1-4} alkyl groups,

R^5 is a C_{1-30} alkyl group substituted by one or more

20 substituents selected from the group consisting of a C_{6-14} aryl group substituted by one or more fluorine atoms, and a fluorine atom,

R^6 and R^7 are each independently an optionally substituted C_{1-30} alkyl group, an optionally substituted C_{3-15} cycloalkyl group,

25 or an optionally substituted C_{6-14} aryl group, or

R^6 and R^7 are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group,

n is 1, and

30 m is 1 or 2.

[2] The compound of the aforementioned [1], wherein R^1 , R^2 , R^3 , and R^4 are each independently a phenyl group, a 1-naphthyl group, a 2-naphthyl group, a 2-biphenyl group, a 3-biphenyl group, a 4-biphenyl group, a 1-anthryl group, a 2-anthryl group, a 9-anthryl group, a 9-phenanthryl group, or a 3-phenanthryl group, each of which is substituted by one or more fluorine atoms or one or more fluoro C_{1-4} alkyl groups.

[3] The compound of the aforementioned [1], wherein all of R^1 , R^2 , R^3 , and R^4 are pentafluorophenyl groups, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenyl) groups, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl groups, or 1,3,4,5,6,7,8-heptafluoro-2-naphthyl groups.

[4] The compound of any of the aforementioned [1] to [3], wherein R^5 is a C_{1-6} alkyl group substituted by a phenyl group or a naphthyl group, each of which is substituted by one or more fluorine atoms, or a fluoro C_{1-6} alkyl group.

[5] The compound of any of the aforementioned [1] to [4], wherein R^6 and R^7 are each independently a C_{1-30} alkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a C_{6-14} aryl group optionally substituted by halogen atom(s),
- (2) a halogen atom, and
- (3) a C_{1-30} alkoxy group; or

a C_{3-8} cycloalkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a halogen atom,
- (2) a C_{1-30} alkyl group,
- (3) a C_{1-30} alkoxy group,
- (4) a halo C_{1-30} alkyl group, and
- (5) a halo C_{1-30} alkoxy group.

[6] The compound of any of the aforementioned [1] to [4], wherein R^6 and R^7 are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group derived from a 3- to 8-membered monocyclic nitrogen-containing non-aromatic heterocyclic group.

[7] The compound of any of the aforementioned [1] to [4], wherein R^5 is a fluoro C_{1-6} alkyl group, R^6 and R^7 are each independently a C_{1-30} alkyl group optionally substituted by substituent(s) selected from the group

5 consisting of

- (1) a C_{6-14} aryl group optionally substituted by halogen atom(s),
- (2) a halogen atom, and
- (3) a C_{1-30} alkoxy group.

[8] The compound of any of the aforementioned [1] to [7],
10 wherein n and m are each 1.

[9] The compound of any of the aforementioned [1] to [8], wherein a total carbon number of R^5 , R^6 , and R^7 is not less than 25.

[10] A composition comprising the compound of any of the
15 aforementioned [1] to [9], and a compound of the following formula (2):

[0015]



20 [0016]

wherein

R and R' are each independently an optionally substituted C_{1-30} alkyl group, an optionally substituted C_{3-15} cycloalkyl group, or an optionally substituted C_{6-14} aryl group

25 (hereinafter to be also referred to as "the composition of the present invention").

[11] The composition of the aforementioned [10], wherein R and R' are each independently an optionally substituted C_{1-30} alkyl group.

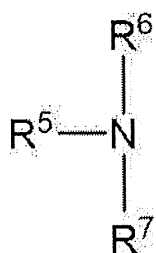
30 [12] The composition of the aforementioned [10] or [11], wherein a content of the compound represented by the aforementioned formula (2) with respect to 1 mol of the compound represented by the aforementioned formula (1) is 0.01 to 10 mol.

[13] The composition of the aforementioned [10] or [11], wherein a content of the compound represented by the aforementioned formula (2) with respect to 1 mol of the compound represented by the aforementioned formula (1) is 0.1 to 3 mol.

[14] A composition comprising the compound of any of the aforementioned [1] to [9], and a compound of the following formula (3):

[0017]

10



(3)

[0018]

wherein R^5 , R^6 , and R^7 are as defined above

(in the composition, the compound represented by formula (3) is an amine compound deprotonated from the cation constituting the formula (1) described in the aforementioned [1]).

[15] The composition of the aforementioned [14], wherein a content of the compound represented by the aforementioned formula (3) with respect to 1 mol of the compound represented by the aforementioned formula (1) is 0.01 to 10 mol.

[16] The composition of the aforementioned [14], wherein a content of the compound represented by the aforementioned formula (3) with respect to 1 mol of the compound represented by the aforementioned formula (1) is 0.5 to 3 mol.

[17] The composition of any of the aforementioned [14] to [16], further comprising a compound represented by the following formula (2):

[0019]



[0020]

wherein

R and R' are each independently an optionally substituted C₁₋₃₀ alkyl group, an optionally substituted C₃₋₁₅ cycloalkyl group, or an optionally substituted C₆₋₁₄ aryl group.

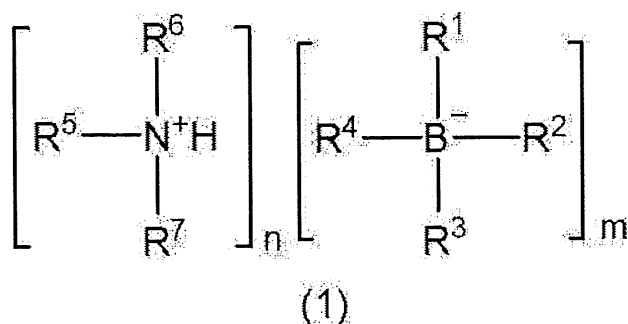
[18] A co-catalyst for polymerizing at least one monomer selected from the group consisting of olefin, diene, and acetylene, comprising the compound of any of the aforementioned [1] to [9], or the composition of any of the aforementioned [10] to [17].

[19] A method for producing a polymer, comprising polymerizing at least one kind of monomer selected from the group consisting of olefin, diene and acetylene by using the compound of any of the aforementioned [1] to [9] or the composition of any of the aforementioned [10] to [17] as a co-catalyst.

[20] A method for producing a compound represented by the following formula (1):

[0021]

20



[0022]

wherein

R¹, R², R³, and R⁴ are each independently a C₆₋₁₄ aryl group substituted by one or more fluorine atoms or one or more fluoro C₁₋₄ alkyl groups,

R⁵ is a C₁₋₃₀ alkyl group substituted by one or more substituents selected from the group consisting of

(1) a C₆₋₁₄ aryl group substituted by one or more fluorine atoms

and

(2) a fluorine atom,

R⁶ and R⁷ are each independently an optionally substituted C₁₋₃₀ alkyl group, an optionally substituted C₃₋₁₅ cycloalkyl group,

5 or an optionally substituted C₆₋₁₄ aryl group, or

R⁶ and R⁷ are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group,

n is 1, and

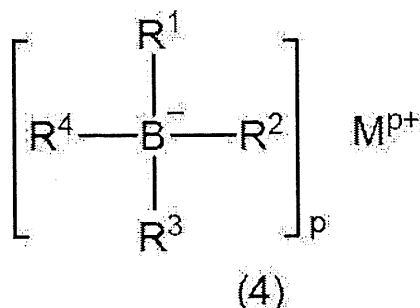
10 m is 1 or 2,

the method comprising a step of reacting, in the presence of a protic acid, a compound represented by the following formula

(4):

[0023]

15



[0024]

wherein

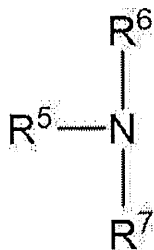
R¹, R², R³, and R⁴ are as defined above,

20 M^{p+} is an alkali metal ion or an alkaline earth metal ion, and

p is 1 or 2,

with a compound represented by the following formula (3):

[0025]



(3)

[0026]

wherein R⁵, R⁶ and R⁷ are as defined above.

[Effect of the Invention]

5 [0027]

According to the present invention, a fluorine-containing alkylammonium borate compound showing a high metal complex catalyst activation ability and useful as a co-catalyst in the polymerization reactions of olefin, diene, and acetylene, a
10 composition containing same, and an industrial production method thereof can be provided.

[Description of Embodiments]

[0028]

The definitions of the terms and respective symbols used
15 in the present specification are explained below.

[0029]

In the present specification, the "halogen atom" means a fluorine atom, a chlorine atom, a bromine atom, or an iodine atom.

20 [0030]

In the present specification, the "alkyl (group)" means a linear or branched chain alkyl group having a carbon number of not less than 1.

[0031]

25 In the present specification, the "C₁₋₃₀ alkyl (group)" means a linear or branched chain alkyl group having a carbon number of 1 to 30. Examples thereof include methyl, ethyl, propyl, isopropyl, butyl, isobutyl, sec-butyl, tert-butyl, pentyl, isopentyl, neopentyl, 1-ethylpropyl, hexyl, isohexyl,

1,1-dimethylbutyl, 2,2-dimethylbutyl, 3,3-dimethylbutyl, 2-ethylbutyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, hexadecyl, octadecyl, nonadecyl, eicosyl, docosyl, tricosyl, tetracosyl, pentacosyl, hexacosyl,
5 heptacosyl, octacosyl, nonacosyl, triacontyl, and the like.
[0032]

In the present specification, the "C₁₋₁₈ alkyl (group)" means a linear or branched chain alkyl group having a carbon number of 1 to 18. Examples thereof include methyl, ethyl,
10 propyl, isopropyl, butyl, isobutyl, sec-butyl, tert-butyl, pentyl, isopentyl, neopentyl, 1-ethylpropyl, hexyl, isohexyl, 1,1-dimethylbutyl, 2,2-dimethylbutyl, 3,3-dimethylbutyl, 2-ethylbutyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, hexadecyl, octadecyl, and the like.
15 [0033]

In the present specification, the "C₁₋₆ alkyl (group)" means a linear or branched chain alkyl group having a carbon number of 1 to 6. Examples thereof include methyl, ethyl, propyl, isopropyl, butyl, isobutyl, sec-butyl, tert-butyl,
20 pentyl, isopentyl, neopentyl, 1-ethylpropyl, hexyl, isohexyl, 1,1-dimethylbutyl, 2,2-dimethylbutyl, 3,3-dimethylbutyl, 2-ethylbutyl, and the like. Among them, C₁₋₄ alkyl group is preferred.
[0034]

In the present specification, the "halo C₁₋₃₀ alkyl (group)" means the aforementioned "C₁₋₃₀ alkyl" group in which one or more hydrogen atoms are substituted by halogen atom(s). Specific examples thereof include fluoromethyl, difluoromethyl, trifluoromethyl, 2-chloroethyl, 2-bromoethyl, 2-iodoethyl, 2-
30 fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, heptafluoropropyl, 2,2-difluoropropyl, 1,1,2,2-tetrafluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 4,4,4-trifluorobutyl, 2,2-difluoropentyl, 5,5,5-trifluoropentyl,
35 2,2-difluorohexyl, 6,6,6-trifluorohexyl, and the like. Among

them, a "fluoro C₁₋₃₀ alkyl (group)" which is a C₁₋₃₀ alkyl group in which the halogen atom in the aforementioned "halo C₁₋₃₀ alkyl (group)" is a fluorine atom, and substituted by one or more fluorine atoms is preferred.

5 [0035]

In the present specification, the "halo C₁₋₆ alkyl (group)" means the aforementioned "C₁₋₆ alkyl" group in which one or more hydrogen atoms are substituted by halogen atom(s). Specific examples thereof include fluoromethyl, difluoromethyl, 10 trifluoromethyl, 2-chloroethyl, 2-bromoethyl, 2-iodoethyl, 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, heptafluoropropyl, 2,2-difluoropropyl, 1,1,2,2-tetrafluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 15 4,4,4-trifluorobutyl, 2,2-difluoropentyl, 5,5,5-trifluoropentyl, 2,2-difluorohexyl, 6,6,6-trifluorohexyl and the like. Among them, a "fluoro C₁₋₆ alkyl (group)" which is the aforementioned "halo C₁₋₆ alkyl" group in which the halogen atom is a fluorine atom is preferred.

20 [0036]

Specific examples of the "fluoro C₁₋₆ alkyl (group)" include fluoromethyl, difluoromethyl, trifluoromethyl, 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, heptafluoropropyl, 2,2-difluoropropyl, 25 1,1,2,2-tetrafluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 4,4,4-trifluorobutyl, 2,2-difluoropentyl, 5,5,5-trifluoropentyl, 2,2-difluorohexyl, 6,6,6-trifluorohexyl, and the like. As specific examples of the fluoro C₁₋₆ alkyl (group) in the 30 definition of R⁵, fluoro C₁₋₆ alkyl (groups) having fluorine atom(s) at the β -position and/or γ -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 2,2-difluoropentyl, 2,2-difluorohexyl and the 35

like are preferred, fluoro C₁₋₄ alkyl (groups) having fluorine atom(s) at the β -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, and the like are more preferred, and fluoro C₂₋₄ alkyl (groups) having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl, and the like are further preferred.

10 [0037]

Specific examples of the "fluoro C₁₋₄ alkyl (group)" as the substituent of the C₆₋₁₄ aryl group for R¹, R², R³, or R⁴, or the substituent of the cyclic group formed by R⁶ and R⁷ bonded to each other together with the nitrogen atom bonded thereto include fluoromethyl, difluoromethyl, trifluoromethyl, 1,1-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, heptafluoropropyl, 1,1,2,2-tetrafluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 1,1-difluoropropyl, 2,2-difluoropropyl, nonafluorobutyl, 1,1,2,2-tetrafluorobutyl, 2,2-difluorobutyl, 1,1-difluorobutyl, 4,4,4-trifluorobutyl, and the like. Among them, trifluoromethyl, 1,1-difluoroethyl, pentafluoroethyl, 1,1-difluoropropyl, heptafluoropropyl, 1,1-difluorobutyl, and nonafluorobutyl are preferred, trifluoromethyl, 1,1-difluoroethyl, and pentafluoroethyl are more preferred.

25 [0038]

In the present specification, the "cycloalkyl (group)" means a cyclic alkyl group. Unless the carbon number range is particularly limited, it is preferably a C₃₋₁₅ cycloalkyl group, more preferably a C₃₋₈ cycloalkyl group.

30 [0039]

In the present specification, the "C₃₋₁₅ cycloalkyl (group)" means a cyclic alkyl group having a carbon number of 3 to 15. Examples thereof include cyclopropyl, cyclobutyl, cyclopentyl, cyclohexyl, cycloheptyl, cyclooctyl, cyclononyl,

cyclodecyl, cycloundecyl, cyclododecyl, cyclotridecyl, cyclotetradecyl, cyclopentadecyl, and the like. The "C₃₋₈ cycloalkyl (group)" means a cyclic alkyl group having a carbon number of 3 to 8. Examples thereof include cyclopropyl, cyclobutyl, cyclopentyl, cyclohexyl, cycloheptyl, cyclooctyl, and the like. Among them, a C₃₋₆ cycloalkyl group is preferred.
[0040]

In the present specification, the "alkoxy (group)" means a group in which a linear or branched chain alkyl group is bonded to an oxygen atom.
[0041]

In the present specification, the "C₁₋₃₀ alkoxy (group)" means a linear or branched chain alkoxy group having a carbon number of 1 to 30. Examples thereof include methoxy, ethoxy, propoxy, isopropoxy, butoxy, isobutoxy, sec-butoxy, tert-butoxy, pentyloxy, isopentyloxy, neopentyloxy, hexyloxy, isohexyloxy, 1,1-dimethylbutoxy, 2,2-dimethylbutoxy, 3,3-dimethylbutoxy, 2-ethylbutoxy, heptyloxy, octyloxy, nonyloxy, decyloxy, undecyloxy, dodecyloxy, tridecyloxy, tetradecyloxy, hexadecyloxy, octadecyloxy, eicosyloxy, docosyloxy, tricosyloxy, tetracosyloxy, pentacosyloxy, hexacosyloxy, heptacosyloxy, octacosyloxy, nonacosyloxy, triacontyloxy, and the like.
[0042]

In the present specification, the "C₁₋₆ alkoxy (group)" means a linear or branched chain alkoxy group having a carbon number of 1 to 6. Examples thereof include methoxy, ethoxy, propoxy, isopropoxy, butoxy, isobutoxy, sec-butoxy, tert-butoxy, pentyloxy, isopentyloxy, neopentyloxy, hexyloxy, and the like. Among them, a C₁₋₄ alkoxy group is preferred.

[0043]

In the present specification, the "halo C₁₋₃₀ alkoxy (group)" means the aforementioned "C₁₋₃₀ alkoxy" group in which one or more hydrogen atoms are substituted by halogen atom(s). Specific examples thereof include difluoromethoxy, trifluoromethoxy, 2-chloroethoxy, 2-bromoethoxy, 2-iodoethoxy,

2-fluoroethoxy, 2,2-difluoroethoxy, 2,2,2-trifluoroethoxy,
pentafluoroethoxy, heptafluoropropoxy, 2,2-difluoropropoxy,
1,1,2,2-tetrafluoropropoxy, 2,2,3,3-tetrafluoropropoxy, 3,3,3-
trifluoropropoxy, 2,2,3,3,3-pentafluoropropoxy, 2,2-
5 difluorobutoxy, 2,2,3,3,3-pentafluoropropoxy, 4,4,4-
trifluorobutoxy, 2,2-difluoropentyloxy, 5,5,5-
trifluoropentyloxy, 2,2-difluorohexyloxy, 6,6,6-
trifluorohexyloxy, and the like. Among them, "halo C₁₋₆ alkoxy"
in which one or more hydrogen atoms in the aforementioned "C₁₋₆
10 alkoxy" group are substituted by halogen atom(s) is preferred.
[0044]

In the present specification, the "fluoro C₁₋₆ alkoxy
(group)" means the aforementioned "halo C₁₋₆ alkoxy" group in
which the halogen atom is a fluorine atom. Specific examples
15 thereof include difluoromethoxy, trifluoromethoxy, 2-
fluoroethoxy, 2,2-difluoroethoxy, 2,2,2-trifluoroethoxy,
pentafluoroethoxy, heptafluoropropoxy, 2,2-difluoropropoxy,
1,1,2,2-tetrafluoropropoxy, 2,2,3,3-tetrafluoropropoxy, 3,3,3-
trifluoropropoxy, 2,2,3,3,3-pentafluoropropoxy, 2,2-
20 difluorobutoxy, 2,2,3,3,3-pentafluoropropoxy, 4,4,4-
trifluorobutoxy, 2,2-difluoropentyloxy, 5,5,5-
trifluoropentyloxy, 2,2-difluorohexyloxy, 6,6,6-
trifluorohexyloxy, and the like. Among them, "fluoro C₁₋₄
alkoxy (groups)" such as difluoromethoxy, trifluoromethoxy,
25 2,2,2-trifluoroethoxy, pentafluoroethoxy, pentafluoroethoxy,
2,2,3,3-tetrafluoropropoxy, 3,3,3-trifluoropropoxy, 4,4,4-
trifluorobutoxy, and the like are preferred, difluoromethoxy,
trifluoromethoxy, 2,2,2-trifluoroethoxy, pentafluoroethoxy, and
pentafluoroethoxy are more preferred, and trifluoromethoxy is
30 particularly preferred.

[0045]

In the present specification, the "aryl (group)" means a
monocyclic or polycyclic (fused) hydrocarbon group showing
aromaticity. Specific examples thereof include C₆₋₁₄ aryl
35 groups such as phenyl, 1-naphthyl, 2-naphthyl, 2-biphenyl, 3-

biphenylyl, 4-biphenylyl, 1-anthryl, 2-anthryl, 9-anthryl, 3-phenanthryl, 9-phenanthryl, and the like. Among them, phenyl, 4-biphenylyl, 1-naphthyl, and 2-naphthyl are preferred.

[0046]

5 In the present specification, the "C₆₋₁₄ aryl (group) substituted by one or more fluorine atoms or one or more fluoro C₁₋₄ alkyl groups" means the aforementioned C₆₋₁₄ aryl group in which one or more hydrogen atoms are substituted by a fluorine atom or a fluoro C₁₋₄ alkyl group, Specifically, for example,
10 pentafluorophenyl group, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenylyl) group, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl group, 1,3,4,5,6,7,8-heptafluoro-2-naphthyl group, 4-trifluoromethylphenyl group, 3,4-bis(trifluoromethyl)phenyl group, and the like can be mentioned. Among them,
15 pentafluorophenyl group, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenylyl) group, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl group, and 1,3,4,5,6,7,8-heptafluoro-2-naphthyl group are preferred.

[0047]

20 In the present specification, the "C₁₋₃₀ alkyl (group) substituted by a C₆₋₁₄ aryl group substituted by one or more fluorine atoms" means a C₁₋₃₀ alkyl (group) substituted by the aforementioned C₆₋₁₄ aryl group in which one or more hydrogen atoms are substituted by a fluorine atom, and a C₁₋₆ alkyl group
25 substituted by a phenyl group or a naphthyl group, each substituted by one or more fluorine atoms, is preferred. Specifically, for example, pentafluorophenylmethyl group, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenylyl)methyl group, 2,3,4,5,6,7,8-heptafluoro-1-naphthylmethyl group,
30 1,3,4,5,6,7,8-heptafluoro-2-naphthylmethyl group and the like can be mentioned. Among them, pentafluorophenylmethyl group, 2,3,4,5,6,7,8-heptafluoro-1-naphthylmethyl group, or 1,3,4,5,6,7,8-heptafluoro-2-naphthylmethyl group is preferred, and pentafluorophenylmethyl group is more preferred.

35 [0048]

In the present specification, the "cyclic group formed by R^6 and R^7 bonded to each other together with the nitrogen atom bonded thereto" means a cyclic ammonio group derived from a saturated nitrogen-containing non-aromatic heterocyclic group (in the case of the compound of the present invention (compound (1))) or a saturated nitrogen-containing non-aromatic heterocycle (in the case of below-mentioned compound (3)). As used herein, the "cyclic ammonio group derived from a saturated nitrogen-containing non-aromatic heterocyclic group" means a cyclic ammonio group formed by the binding of R^6 and R^7 to each other and the binding of a hydrogen atom to the nitrogen atom to which R^6 and R^7 are bonded. Such "cyclic group" may have, besides carbon atom, a hetero atom selected from an oxygen atom, a sulfur atom, and a nitrogen atom as other ring-constituting atom of the nitrogen atom of the amino group, and a cyclic ammonio group derived from a 3- to 8-membered (preferably 4- to 6-membered) monocyclic nitrogen-containing non-aromatic heterocyclic group can be mentioned. Specific examples of the "cyclic group" include cyclic ammonio groups derived from 3- to 8-membered monocyclic nitrogen-containing non-aromatic heterocyclic groups, such as aziridinyl group, azetidiny group, pyrrolidinyl group, pyrrolinyl group, piperidyl group, azepanyl group, morpholinyl group, thiomorpholinyl group, piperazinyl group, oxazolidinyl group, thiazolidinyl group, imidazolidinyl group, oxazolinyl group, thiazolinyl group, imidazolinyl group, pyrazolidinyl group, pyrazolinyl group, tetrahydropyridyl group, tetrahydropyrimidinyl group, tetrahydrotriazolyl group, and the like. Among them, a cyclic ammonio group derived from azetidiny group, pyrrolidinyl group, piperidyl group, piperazinyl group, or morpholinyl group is preferred, and a cyclic ammonio group derived from piperidyl group or piperazinyl group is more preferred. As the "saturated nitrogen-containing non-aromatic heterocycle", a 3- to 8-membered (preferably 4- to 6-membered) monocyclic nitrogen-containing non-aromatic heterocycle that may have, besides

carbon atom, a hetero atom selected from an oxygen atom, a sulfur atom, and a nitrogen atom as other ring-constituting atom of the nitrogen atom of the amino group can be mentioned. Specific examples of the "saturated nitrogen-containing non-
5 aromatic heterocycle" include 3- to 8-membered monocyclic nitrogen-containing non-aromatic heterocycles such as aziridine, azetidine, pyrrolidine, pyrroline, piperidine, azepane, morpholine, thiomorpholine, piperazine, oxazolidine, thiazolidine, imidazolidine, oxazoline, thiazoline, imidazoline,
10 pyrazolidine, pyrazoline, tetrahydropyridine, tetrahydropyrimidine, tetrahydrotriazoline, and the like. Among them, azetidine, pyrrolidine, piperidine, piperazine, or morpholine is preferred, and piperidine or piperazine is more preferred.

15 [0049]

In the present specification, the "optionally substituted" means unsubstituted or having one or more substituents. Unless otherwise particularly indicated, (1) a halogen atom, (2) a nitro group, (3) a cyano group, (4) a C₁₋₃₀
20 alkyl group, (5) a halo C₁₋₃₀ alkyl group, (6) a C₃₋₈ cycloalkyl group, (7) a C₁₋₃₀ alkoxy group, (8) a halo C₁₋₃₀ alkoxy group, (9) a C₆₋₁₄ aryl group, and the like can be mentioned as the "substituent". Among them, a halogen atom, a cyano group, a C₁₋₆ alkyl group, a halo C₁₋₆ alkyl group, a C₁₋₆ alkoxy group, a
25 halo C₁₋₆ alkoxy group, and a phenyl group are preferred, and a halogen atom (e.g., fluorine atom), a C₁₋₄ alkyl group (e.g., methyl, ethyl), a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy), a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl and the like), or a halo
30 C₁₋₄ alkoxy group (e.g., fluoro C₁₋₄ alkoxy group such as trifluoromethoxy, 2,2,2-trifluoroethoxy, and the like) is more preferred. When plural substituents are present, respective substituents may be the same or different. The above-mentioned substituents may also be further substituted by one or more of
35 a C₁₋₆ alkyl group, a C₁₋₆ alkoxy group, a halogen atom, a phenyl

group, and the like.

[0050]

In the present specification, specific examples of the "alkali metal ion" include lithium ion, potassium ion, sodium ion, cesium ion, and the like.

[0051]

In the present specification, specific examples of the "alkaline earth metal ion" include magnesium ion, calcium ion, and the like.

10 [0052]

In the present specification, the "hydrocarbon solvent" means solvents including aromatic hydrocarbon solvents and/or aliphatic hydrocarbon solvents. Among them, aliphatic hydrocarbon solvents are preferable from the aspects of odor and toxicity.

[0053]

In the present specification, examples of the "aromatic hydrocarbon solvent" include benzene, toluene, xylene, and the like.

20 [0054]

In the present specification, examples of the "aliphatic hydrocarbon solvent" include n-hexane, isohexane, n-heptane, n-octane, cyclohexane, methylcyclohexane, a mixed solvent thereof, and the like.

25 [0055]

In the present specification, the "soluble in hydrocarbon solvent (or aliphatic hydrocarbon solvent)" means that the compound (or composition) of the present invention is dissolved in a solution of a hydrocarbon solvent (or aliphatic hydrocarbon solvent) and the compound (or composition) of the present invention at 25°C at a concentration of not less than 5 wt% to form a clear homogeneous solution. In addition, the "easily soluble in hydrocarbon solvent (or aliphatic hydrocarbon solvent)" means that the compound (or composition) of the present invention is dissolved in a solution of a

hydrocarbon solvent (or aliphatic hydrocarbon solvent) and the compound (or composition) of the present invention at 25°C at a concentration of not less than 20 wt% (preferably not less than 30 wt%) to form a clear homogeneous solution.

5 [0056]

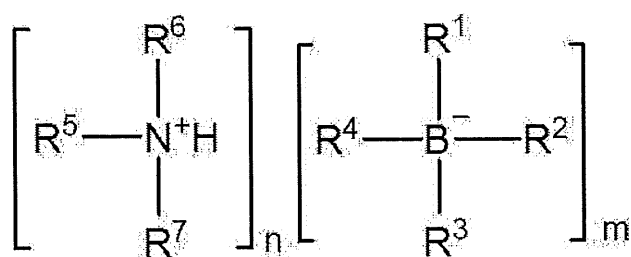
(Compound of the present invention)

The compound of the present invention is explained below.

[0057]

The compound of the present invention is a compound
10 represented by the following formula (1):

[0058]



(1)

[0059]

15 wherein

R^1 , R^2 , R^3 , and R^4 are each independently a C_{6-14} aryl group substituted by one or more fluorine atoms or one or more fluoro C_{1-4} alkyl groups,

R^5 is a C_{1-30} alkyl group substituted by one or more
20 substituents selected from the group consisting of a C_{6-14} aryl group substituted by one or more fluorine atoms, and a fluorine atom,

R^6 and R^7 are each independently an optionally substituted C_{1-30} alkyl group, an optionally substituted C_{3-15} cycloalkyl group,
25 or an optionally substituted C_{6-14} aryl group, or

R^6 and R^7 are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group,

n is 1, and

30 m is 1 or 2.

[0060]

Preferred embodiments of the compound represented by the formula (1) (hereinafter to be also referred to as "compound (1)") are explained below.

5 [0061]

Each group of compound (1) is explained below.

[0062]

R^1 , R^2 , R^3 , and R^4 are preferably each independently a phenyl group, a 1-naphthyl group, a 2-naphthyl group, a 2-biphenylyl group, a 3-biphenylyl group, a 4-biphenylyl group, a 1-anthryl group, a 2-anthryl group, a 9-anthryl group, a 3-phenanthryl group, or a 9-phenanthryl group, each substituted by one or more fluorine atoms or one or more fluoro C_{1-4} alkyl groups (e.g., trifluoromethyl group), more preferably each
15 independently a phenyl group, a 1-naphthyl group, a 2-naphthyl group, or a 4-biphenylyl group, each substituted by one or more fluorine atoms or one or more trifluoromethyl groups, particularly preferably a pentafluorophenyl group, a 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenylyl) group, a
20 2,3,4,5,6,7,8-heptafluoro-1-naphthyl group, or a 1,3,4,5,6,7,8-heptafluoro-2-naphthyl group, wherein all of R^1 , R^2 , R^3 , and R^4 are the same.

[0063]

R^5 is preferably a C_{1-6} alkyl group substituted by a
25 phenyl group or a naphthyl group, each of which is substituted by one or more fluorine atoms, or a fluoro C_{1-6} alkyl group, more preferably, a phenylmethyl group substituted by one or more fluorine atoms (e.g., pentafluorophenylmethyl group) or a fluoro C_{1-6} alkyl group, further preferably, a fluoro C_{1-6} alkyl
30 group (e.g., fluoro C_{1-6} alkyl group having fluorine atom(s) at the β -position and/or γ -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl,
35 2,2-difluoropentyl, 2,2-difluorohexyl, and the like),

particularly preferably, a fluoro C₁₋₄ alkyl group (e.g., a fluoro C₁₋₄ alkyl group having fluorine atom(s) at the β -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, and the like).

[0064]

R⁶ and R⁷ are preferably each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s),
- (2) a halogen atom, and
- (3) a C₁₋₃₀ alkoxy group; or

a C₃₋₈ cycloalkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a halogen atom,
- (2) a C₁₋₃₀ alkyl group,
- (3) a C₁₋₃₀ alkoxy group,
- (4) a halo C₁₋₃₀ alkyl group, and
- (5) a halo C₁₋₃₀ alkoxy group,

more preferably, each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s) (e.g., fluorine atom),
- (2) a halogen atom, and
- (3) a C₁₋₃₀ alkoxy group.

[0065]

In another embodiment of R⁶ and R⁷, R⁶ and R⁷ are preferably bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group derived from a 3- to 8-membered monocyclic nitrogen-containing non-aromatic heterocyclic group, R⁶ and R⁷ are more preferably bonded to each other to form, together with the nitrogen atom bonded thereto, a cyclic group derived from an

azetidiny1 group, a pyrrolidinyl group, a piperidyl group, a piperazinyl group, or a morpholinyl group, each of which is optionally substituted by substituent(s) selected from the group consisting of,

- 5 (1) a halogen atom (e.g., fluorine atom),
(2) a C₁₋₄ alkyl group (e.g., methyl, ethyl),
(3) a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy),
(4) a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl, and the like), and
10 (5) a halo C₁₋₄ alkoxy group, or R⁶ and R⁷ are particularly preferably bonded to each other to form, together with the nitrogen atom bonded thereto, a cyclic group derived from a piperidyl group or a piperazinyl group, each of which is optionally substituted by a halogen atom (e.g., fluorine atom),
15 a C₁₋₄ alkyl group (e.g., methyl, ethyl), a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy), a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl, and the like), or a halo C₁₋₄ alkoxy group.

[0066]

- 20 The total carbon number of R⁵, R⁶, and R⁷ is preferably not less than 25, more preferably not less than 35.

[0067]

n is preferably 1.

[0068]

- 25 m is preferably 1 or 2, more preferably 1.

[0069]

As preferred compound (1), the following compounds can be mentioned.

[0070]

- 30 [Compound (1-1)]

Compound (1) of the aforementioned formula (1), wherein R¹, R², R³, and R⁴ are each independently a phenyl group, a 1-naphthyl group, a 2-naphthyl group, a 2-biphenyl group, a 3-biphenyl group, a 4-biphenyl group, a 1-anthryl group, a 2-anthryl group, a 9-anthryl group, a 3-phenanthryl group, or a

35

9-phenanthryl group, each of which is substituted by one or more fluorine atoms or one or more fluoro C₁₋₄ alkyl groups (e.g., trifluoromethyl group),

R⁵ is a C₁₋₆ alkyl group substituted by a phenyl group or a
5 naphthyl group, each of which is substituted by one or more fluorine atoms, or a fluoro C₁₋₆ alkyl group (preferably, fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and the like.),

10 R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s),
- (2) a halogen atom, and

15 (3) a C₁₋₃₀ alkoxy group; or

a C₃₋₈ cycloalkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a halogen atom,
- (2) a C₁₋₃₀ alkyl group,
- 20 (3) a C₁₋₃₀ alkoxy group,
- (4) a halo C₁₋₃₀ alkyl group, and
- (5) a halo C₁₋₃₀ alkoxy group,

n is 1, and

m is 1.

25 [0071]

[Compound (1-2)]

Compound (1) of the aforementioned formula (1), wherein R¹, R², R³, and R⁴ are each independently a phenyl group, a 1-naphthyl group, a 2-naphthyl group or a 4-biphenyl group,
30 each of which is substituted by one or more fluorine atoms or one or more trifluoromethyl groups,
R⁵ is a phenylmethyl group (e.g., pentafluorophenylmethyl group) substituted by one or more fluorine atoms, or a fluoro C₁₋₆ alkyl group, further preferably, a fluoro C₁₋₆ alkyl group
35 (e.g., fluoro C₁₋₆ alkyl group having fluorine atom(s) at the β -

position and/or γ -position, such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl,
5 2,2-difluoropentyl, 2,2-difluorohexyl, and the like), particularly preferably, a fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms, such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and the like,
10 R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group consisting of
(1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s) (e.g., fluorine atom),
15 (2) a halogen atom, and
(3) a C₁₋₃₀ alkoxy group,
n is 1, and
m is 1.

[0072]

20 [Compound (1-3)]

Compound (1) of the aforementioned formula (1), wherein R¹, R², R³, and R⁴ are all the same and are pentafluorophenyl groups, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenyl) groups, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl groups, or
25 1,3,4,5,6,7,8-heptafluoro-2-naphthyl groups,
R⁵ is a fluoro C₁₋₆ alkyl group (e.g., fluoro C₁₋₆ alkyl group having fluorine atom(s) at the β -position and/or γ -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl,
30 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 2,2-difluoropentyl, 2,2-difluorohexyl, and the like),
R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group
35 consisting of

(1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s)
(e.g., fluorine atom),

(2) a halogen atom, and

(3) a C₁₋₃₀ alkoxy group,

5 n is 1, and

m is 1.

[0073]

[Compound (1-4)]

Compound (1) of the aforementioned formula (1), wherein
10 R¹, R², R³, and R⁴ are all the same and are pentafluorophenyl
groups, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenyl)
groups, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl groups, or
1,3,4,5,6,7,8-heptafluoro-2-naphthyl groups,
R⁵ is a fluoro C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl groups
15 having fluorine atom(s) at the β -position such as 2-fluoroethyl,
2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-
difluoropropyl, 2,2,3,3-tetrafluoropropyl, 2,2,3,3,3-
pentafluoropropyl, 2,2-difluorobutyl, and the like, among which
a fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -
20 position and having 3 or more fluorine atoms such as 2,2,2-
trifluoroethyl, 2,2,3,3,3-pentafluoropropyl, and the like is
preferred),

R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally
substituted by substituent(s) selected from the group

25 consisting of

(1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s)
(e.g., fluorine atom),

(2) a halogen atom, and

(3) a C₁₋₃₀ alkoxy group,

30 n is 1, and

m is 1.

[0074]

[Compound (1-5)]

Compound (1) of the aforementioned formula (1), wherein
35 R¹, R², R³, and R⁴ are each independently a phenyl group, a 1-

naphthyl group, a 2-naphthyl group, a 2-biphenylyl group, a 3-biphenylyl group, a 4-biphenylyl group, a 1-anthryl group, a 2-anthryl group, a 9-anthryl group, a 3-phenanthryl group, or a 9-phenanthryl group, substituted by one or more fluorine atoms
5 or one or more fluoro C₁₋₄ alkyl groups (e.g., trifluoromethyl group),

R⁵ is a C₁₋₆ alkyl group substituted by a phenyl group or a naphthyl group, each of which is substituted by one or more fluorine atoms, or a fluoro C₁₋₆ alkyl group (preferably, fluoro
10 C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl, and the like),

R⁶ and R⁷ are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic
15 group derived from a 3- to 8-membered monocyclic nitrogen-containing non-aromatic heterocyclic group,

n is 1, and

m is 1 or 2.

[0075]

20 [Compound (1-6)]

Compound (1) of the aforementioned formula (1), wherein R¹, R², R³, and R⁴ are each independently a phenyl group, a 1-naphthyl group, a 2-naphthyl group, or a 4-biphenylyl group substituted by one or more fluorine atoms or one or more
25 trifluoromethyl groups,

R⁵ is a phenylmethyl group substituted by one or more fluorine atoms (e.g., pentafluorophenylmethyl group) or a fluoro C₁₋₆ alkyl group, further preferably, a fluoro C₁₋₆ alkyl group (e.g., fluoro C₁₋₆ alkyl group having fluorine atom(s) at the β -
30 position and/or γ -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 2,2-difluoropentyl, 2,2-difluorohexyl, and the like, among
35 which a fluoro C₂₋₄ alkyl group having fluorine atom(s) at the

β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and the like is preferred),

R⁶ and R⁷ are bonded to each other to form, together with the
5 nitrogen atom bonded thereto, a cyclic group derived from an azetidiny1 group, a pyrrolidinyl group, a piperidyl group, a piperazinyl group, or a morpholinyl group, each of which is optionally substituted by substituent(s) selected from the group consisting of

- 10 (1) a halogen atom (e.g., fluorine atom),
(2) a C₁₋₄ alkyl group (e.g., methyl, ethyl),
(3) a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy),
(4) a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl, and the like), and
15 (5) a halo C₁₋₄ alkoxy group,

n is 1, and

m is 1 or 2.

[0076]

[Compound (1-7)]

20 Compound (1) of the aforementioned formula (1), wherein R¹, R², R³, and R⁴ are all the same and are pentafluorophenyl groups, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenyl) groups, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl groups, or 1,3,4,5,6,7,8-heptafluoro-2-naphthyl groups,
25 R⁵ is a fluoro C₁₋₆ alkyl group (e.g., fluoro C₁₋₆ alkyl group having fluorine atom(s) at the β -position and/or γ -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-
30 difluorobutyl, 2,2-difluoropentyl, 2,2-difluorohexyl, and the like, among which fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl, and the like are preferred),

35 R⁶ and R⁷ are bonded to each other to form, together with the

nitrogen atom bonded thereto, a cyclic group derived from a piperidyl group or a piperazinyl group, each of which is optionally substituted by substituent(s) selected from the group consisting of,

- 5 (1) a halogen atom (e.g., fluorine atom),
(2) a C₁₋₄ alkyl group (e.g., methyl, ethyl),
(3) a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy),
(4) a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl, and the like), and
10 (5) a halo C₁₋₄ alkoxy group,
n is 1, and
m is 1 or 2.

[0077]

[Compound (1-8)]

- 15 Compound (1) of the aforementioned formula (1), wherein R¹, R², R³, and R⁴ are all the same and are pentafluorophenyl groups, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenyl) groups, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl groups, or 1,3,4,5,6,7,8-heptafluoro-2-naphthyl groups,
20 R⁵ is a fluoro C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group having fluorine atom(s) at the β -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, and the like, among which
25 fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl, and the like are preferred),

R⁶ and R⁷ are bonded to each other to form, together with the
30 nitrogen atom bonded thereto, a cyclic group derived from a piperidyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a halogen atom (e.g., fluorine atom),
(2) a C₁₋₄ alkyl group (e.g., methyl, ethyl),
35 (3) a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy),

(4) a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl, and the like), and
(5) a halo C₁₋₄ alkoxy group,
n is 1, and

5 m is 1.

[0078]

Specific preferable examples of compound (1) include, for example, the compounds of the below-mentioned Examples 1, 4 to 21, 23, and 25, and the like.

10 [0079]

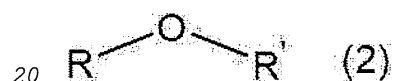
(Composition of the present invention)

The composition of the present invention is explained below.

[0080]

15 The composition of the present invention is a composition containing the aforementioned compound (1) (the compound of the present invention), and a compound represented by the following formula (2):

[0081]



[0082]

wherein

R and R' are each independently an optionally substituted C₁₋₃₀ alkyl group, an optionally substituted C₃₋₁₅ cycloalkyl group,
25 or an optionally substituted C₆₋₁₄ aryl group
(hereinafter to be also referred to as "compound (2)").

[0083]

The composition containing compound (1) and compound (2) is not particularly limited as long as it contains the both
30 compounds, and may include a compound in which compound (2) is coordinated to compound (1) to form a complex. The composition of the present invention is preferably a composition including a complex formed by compound (1) and compound (2).

[0084]

Preferred embodiments of compound (2) are explained below.

Each group of compound (2) is explained below.

[0085]

R and R' are each independently an optionally substituted
5 C₁₋₃₀ alkyl group, an optionally substituted C₃₋₁₅ cycloalkyl
group, or an optionally substituted C₆₋₁₄ aryl group, preferably,
each independently a C₁₋₃₀ alkyl group optionally substituted by
substituent(s) selected from the group consisting of
(1) a halogen atom,
10 (2) a C₁₋₃₀ alkoxy group, and
(3) a halo C₁₋₃₀ alkoxy group;
a C₃₋₁₅ cycloalkyl group optionally substituted by
substituent(s) selected from the group consisting of
(1) a halogen atom,
15 (2) a C₁₋₃₀ alkyl group,
(3) a C₁₋₃₀ alkoxy group,
(4) a halo C₁₋₃₀ alkyl group, and
(5) a halo C₁₋₃₀ alkoxy group; or
a C₆₋₁₄ aryl group optionally substituted by substituent(s)
20 selected from the group consisting of
(1) a halogen atom,
(2) a C₁₋₃₀ alkyl group,
(3) a C₁₋₃₀ alkoxy group,
(4) a halo C₁₋₃₀ alkyl group, and
25 (5) a halo C₁₋₃₀ alkoxy group,
more preferably, each independently a C₁₋₃₀ alkyl group;
a C₃₋₈ cycloalkyl group (e.g., cyclopentyl, cyclohexyl, etc.);
or a phenyl group optionally substituted by substituent(s)
selected from the group consisting of
30 (1) a halogen atom,
(2) a C₁₋₆ alkyl group,
(3) a C₁₋₆ alkoxy group,
(4) a halo C₁₋₆ alkyl group, and
(5) a halo C₁₋₆ alkoxy group,
35 further preferably, each independently a C₁₋₃₀ alkyl group

(preferably, C₁₋₁₈ alkyl group such as methyl, butyl, hexyl, octyl, decyl, dodecyl, tetradecyl, hexadecyl, octadecyl, and the like).

[0086]

5 Specific preferable examples of compound (2) include dibutyl ether, dihexyl ether, dioctyl ether, dodecyl ether, didodecyl ether, ditetradecyl ether, dihexadecyl ether, dioctadecyl ether, cyclopentyl methyl ether, diphenyl ether, phenyl octadecyl ether, and the like.

10 [0087]

 In the composition of the present invention, the content of compound (2) with respect to 1 mol of compound (1) is preferably 0.01 to 10 mol, more preferably 0.1 to 3 mol.

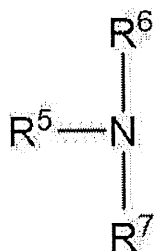
[0088]

15 Specific preferable examples of the composition of the present invention include the compositions of the below-mentioned Examples 2, 3, 24, 26 to 34, and the like.

[0089]

 Another embodiment of the composition of the present
20 invention is a composition containing the aforementioned compound (1) (the compound of the present invention), and a compound represented by the following formula (3):

[0090]



(3)

25 [0091]

 wherein R⁵, R⁶, and R⁷ are as defined above
(hereinafter to be also referred to as "compound (3)")
(in this composition, compound (3) is an amine compound
deprotonated from the cation constituting the aforementioned

compound (1)).

[0092]

The preferred embodiment of compound (3) is the same as the preferred embodiment of each group (R^5 , R^6 , and R^7) in
5 compound (1), except that R^6 and R^7 are deprotonated when they bond with each other to form a cyclic group together with the nitrogen atom bonded thereto.

[0093]

As preferred compound (3), the following compounds can be
10 mentioned.

[0094]

[Compound (3-1)]

Compound (3) wherein, in the aforementioned formula (3), R^5 is a C_{1-6} alkyl group substituted by a phenyl group or a
15 naphthyl group, each of which is substituted by one or more fluorine atoms, or a fluoro C_{1-6} alkyl group (preferably, fluoro C_{2-4} alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and the like.), and
20 R^6 and R^7 are each independently a C_{1-30} alkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a C_{6-14} aryl group optionally substituted by halogen atom(s),
 - (2) a halogen atom, and
 - 25 (3) a C_{1-30} alkoxy group; or
- a C_{3-8} cycloalkyl group optionally substituted by substituent(s) selected from the group consisting of
- (1) a halogen atom,
 - (2) a C_{1-30} alkyl group,
 - 30 (3) a C_{1-30} alkoxy group,
 - (4) a halo C_{1-30} alkyl group, and
 - (5) a halo C_{1-30} alkoxy group.

[0095]

[Compound (3-2)]

35 Compound (3) wherein, in the aforementioned formula (3),

R⁵ is a phenylmethyl group (e.g., pentafluorophenylmethyl group) or a fluoro C₁₋₆ alkyl group, each of which is substituted by one or more fluorine atoms, further preferably, a fluoro C₁₋₆ alkyl group (e.g., fluoro C₁₋₆ alkyl group having
5 fluorine atom(s) at the β -position and/or γ -position such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 2,2-difluoropentyl, 2,2-difluorohexyl, and the
10 like, among which, preferably a fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and the like), and
R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally
15 substituted by substituent(s) selected from the group consisting of
(1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s),
(2) a halogen atom, and
(3) a C₁₋₃₀ alkoxy group.

20 [0096]

[Compound (3-3)]

Compound (3) wherein, in the aforementioned formula (3),
R⁵ is a fluoro C₁₋₆ alkyl group (e.g., fluoro C₁₋₆ alkyl group having fluorine atom(s) at the β -position and/or γ -position
25 such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, 2,2-difluoropentyl, 2,2-difluorohexyl, and the like), among which, preferably a fluoro C₂₋₄ alkyl group having
30 fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and the like), and
R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group
35 consisting of

- (1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s) (e.g., fluorine atom),
- (2) a halogen atom, and
- (3) a C₁₋₃₀ alkoxy group.

5 [0097]

[Compound (3-4)]

Compound (3) wherein, in the aforementioned formula (3), R⁵ is a fluoro C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group having fluorine atom(s) at the β -position such as 2-fluoroethyl, 10 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, and the like, among which a fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2- 15 trifluoroethyl, 2,2,3,3,3-pentafluoropropyl, and the like is preferred), and

R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group consisting of a C₆₋₁₄ aryl group optionally substituted by 20 halogen atom(s) (e.g., fluorine atom), a halogen atom, and a C₁₋₃₀ alkoxy group.

[0098]

[Compound (3-5)]

Compound (3) wherein, in the aforementioned formula (3), 25 R⁵ is a C₁₋₆ alkyl group substituted by a phenyl group or a naphthyl group, each of which is substituted by one or more fluorine atoms, or a fluoro C₁₋₆ alkyl group (preferably, fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 30 2,2,3,3,3-pentafluoropropyl and the like.), and R⁶ and R⁷ are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted 3 to 8-membered monocyclic nitrogen-containing non-aromatic heterocycle.

35 [0099]

[Compound (3-6)]

Compound (3) wherein, in the aforementioned formula (3),
R⁵ is a phenylmethyl group substituted by one or more fluorine
atoms (e.g., pentafluorophenylmethyl group) or a fluoro C₁₋₆
5 alkyl group, further preferably, a fluoro C₁₋₆ alkyl group (e.g.,
fluoro C₁₋₆ alkyl group having fluorine atom(s) at the β -
position and/or γ -position such as 2-fluoroethyl, 2,2-
difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-
difluoropropyl, 2,2,3,3-tetrafluoropropyl, 3,3,3-
10 trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl,
2,2-difluoropentyl, 2,2-difluorohexyl, and the like), among
which, preferably a fluoro C₂₋₄ alkyl group having fluorine
atom(s) at the β -position and having 3 or more fluorine atoms
such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and
15 the like), and

R⁶ and R⁷ are bonded to each other to form, together with the
nitrogen atom bonded thereto, azetidine, pyrrolidine,
piperidine, piperazine, or morpholine, each of which is
optionally substituted by substituent(s) selected from the
20 group consisting of

- (1) a halogen atom (e.g., fluorine atom),
- (2) a C₁₋₄ alkyl group (e.g., methyl, ethyl),
- (3) a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy),
- (4) a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such
25 as trifluoromethyl, 2,2,2-trifluoroethyl, and the like), and
- (5) a halo C₁₋₄ alkoxy group.

[0100]

[Compound (3-7)]

Compound (3) wherein, in the aforementioned formula (3),
30 R⁵ is a fluoro C₁₋₆ alkyl group (e.g., fluoro C₁₋₆ alkyl group
having fluorine atom(s) at the β -position and/or γ -position
such as 2-fluoroethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl,
pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl,
3,3,3-trifluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-
35 difluorobutyl, 2,2-difluoropentyl, 2,2-difluorohexyl, and the

like, among which, preferably a fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl and the like), and

5 R⁶ and R⁷ are bonded to each other to form, together with the nitrogen atom bonded thereto, piperidine or piperazine, each of which is optionally substituted by substituent(s) selected from the group consisting of

- (1) a halogen atom (e.g., fluorine atom),
- 10 (2) a C₁₋₄ alkyl group (e.g., methyl, ethyl),
- (3) a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy),
- (4) a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl, and the like), and
- (5) a halo C₁₋₄ alkoxy group.

15 [0101]

[Compound (3-8)]

Compound (3) wherein, in the aforementioned formula (3), R⁵ is a fluoro C₁₋₄ alkyl group (e.g., a fluoro C₁₋₄ alkyl group having fluorine atom(s) at the β -position such as 2-fluoroethyl, 20 2,2-difluoroethyl, 2,2,2-trifluoroethyl, pentafluoroethyl, 2,2-difluoropropyl, 2,2,3,3-tetrafluoropropyl, 2,2,3,3,3-pentafluoropropyl, 2,2-difluorobutyl, and the like, among which, preferably a fluoro C₂₋₄ alkyl group having fluorine atom(s) at the β -position and having 3 or more fluorine atoms such as 25 2,2,2-trifluoroethyl, 2,2,3,3,3-pentafluoropropyl, and the like), and

R⁶ and R⁷ are bonded to each other to form, together with the nitrogen atom bonded thereto, piperidine optionally substituted by substituent(s) selected from the group consisting of

- 30 (1) a halogen atom (e.g., fluorine atom),
- (2) a C₁₋₄ alkyl group (e.g., methyl, ethyl),
- (3) a C₁₋₄ alkoxy group (e.g., methoxy, ethoxy),
- (4) a halo C₁₋₄ alkyl group (e.g., fluoro C₁₋₄ alkyl group such as trifluoromethyl, 2,2,2-trifluoroethyl, and the like) and
- 35 (5) a halo C₁₋₄ alkoxy group.

[0102]

Specific preferable examples of compound (3) include fluorine-containing alkylamine, fluorine-containing alkylcyclic amine, and the like used for synthesizing the compounds of the
5 below-mentioned Examples 1, 4 to 21.

[0103]

In the composition of the present invention including compound (1) and compound (3), the content of compound (3) with respect to 1 mol of compound (1) is preferably 0.01 to 10 mol,
10 more preferably 0.5 to 3 mol.

[0104]

Specific preferable examples of the composition of the present invention including compound (1) and compound (3) include the composition of the below-mentioned Example 22 and
15 the like.

[0105]

The composition of the present invention containing compound (1) and compound (3) may further contain the aforementioned compound (2) in addition to compound (1) and
20 compound (3).

[0106]

A preferred embodiment of compound (2) that may be further contained is the same as the preferred embodiment of each group (R and R') in the aforementioned compound (2) and
25 specific preferable examples thereof. In the composition of the present invention containing compound (1), compound (2), and compound (3), the content of each of compound (2) and compound (3) with respect to 1 mol of compound (1) is the same as that mentioned above.

30 [0107]

In the compound or composition of the present invention, when the total carbon number of R⁵, R⁶, and R⁷ in the aforementioned formula (1) is not less than 25, it is soluble in aliphatic hydrocarbon solvents at room temperature (15 to
35 30°C). In contrast, conventionally-known borate-type compounds

(e.g., hydrogenated tetrakis(pentafluorophenyl)borate, hydrogenated tetrakis(pentafluorophenyl)borate diethyl ether complex, lithium tetrakis(pentafluorophenyl)borate, etc.) are insoluble in aliphatic hydrocarbon solvents such as n-hexane and the like. Thus, known borate compounds are generally used for polymerization reactions as a solution of aromatic hydrocarbon solvents. As a result, removal of the contaminating aromatic hydrocarbon solvent becomes necessary, and there are problems such as a large commercial cost associated with an increase in the number of steps. In some cases, it is used in the polymerization reaction as a suspension of an aliphatic hydrocarbon solvent. However, there are problems such as poor operability due to being suspended, the need for an excessive amount, and the like. Therefore, the compound or composition of the present invention that is soluble in aliphatic hydrocarbon solvents is particularly useful as a co-catalyst for homogeneous polymerization reactions of olefin, diene, and acetylene, using hydrocarbon solvents (particularly, aliphatic hydrocarbon solvents).

[0108]

(Production method of the compound of the present invention)

The production method of the compound (or composition) of the present invention (hereinafter to be also referred to as "the production method of the present invention") is explained below.

[0109]

The production method of the compound (or composition) of the present invention is not particularly limited, and it can be produced according to the methods described in, for example, the following Production Methods 1 to 3, or the below-mentioned Production Examples or Examples. Particularly, it is possible to obtain the compound (or composition) of the present invention in a simple operation at a low cost and in a high yield as compared with the conventional methods, by producing a fluorine-containing alkylamine by the following production

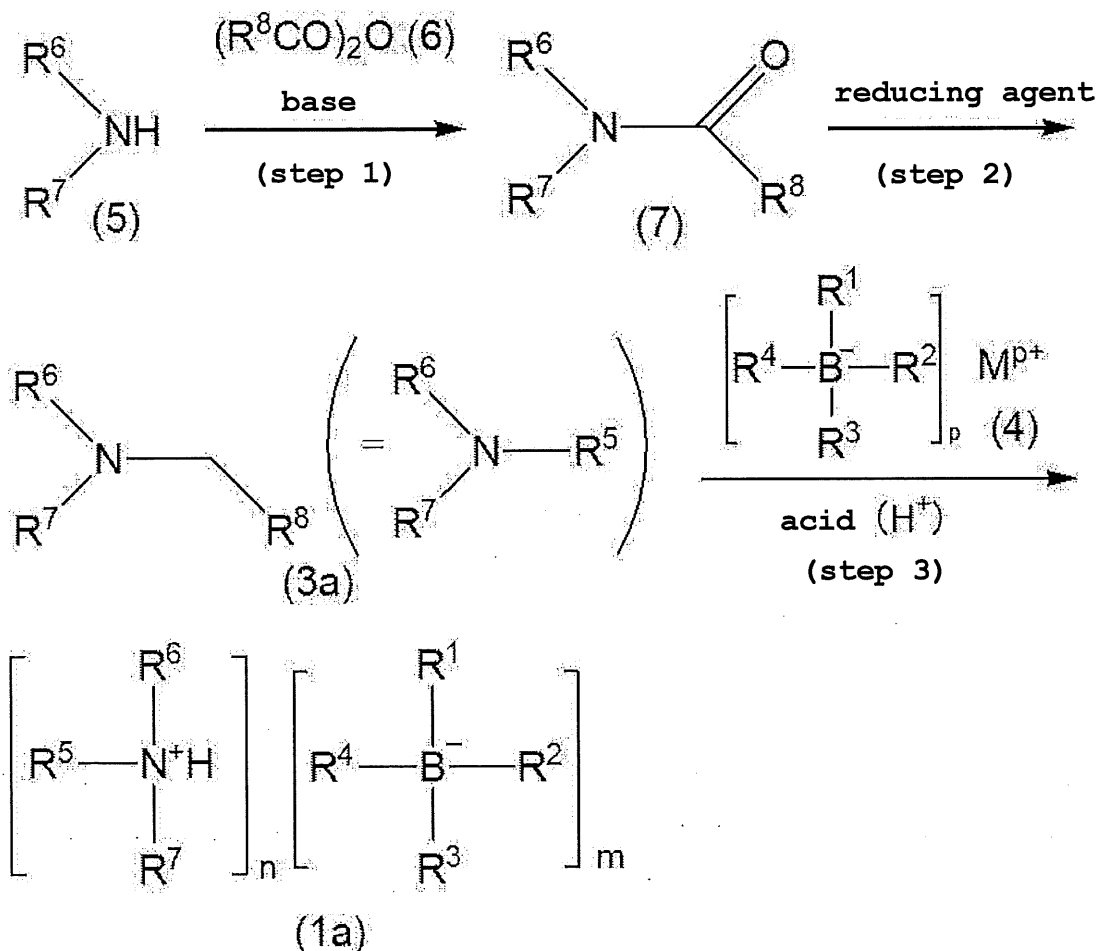
method. Thus, the production method can be an industrial production method.

[0110]

(Production Method 1)

5 Production Method 1 includes a step of obtaining compound (7) by reacting dialkylamine compound (5) with fluoroalkylcarboxylic anhydride (6) in the presence of a base in a solvent that does not affect the reaction (step 1), a step of converting compound (7) to compound (3a) by reducing with a
10 reducing agent in a solvent that does not affect the reaction (step 2), and a step of obtaining compound (1a) (the compound of the present invention) by reacting compound (3a) with compound (4) in a solvent that does not affect the reaction and in the presence of an acid (step 3).

15 [0111]



[0112]

wherein R⁸ is a C₁₋₂₉ alkyl group substituted by one or more

substituents selected from the group consisting of a C₆₋₁₄ aryl group substituted by one or more fluorine atoms and a fluorine atom, and other each symbol is as defined above.

[0113]

5 (step 1)

The solvent used in this step includes, for example, hydrocarbon solvents such as toluene, n-hexane, isohexane, n-heptane, n-octane, cyclohexane, methylcyclohexane, and the like; ethers such as diethyl ether, tetrahydrofuran, and the
10 like; halogen solvents such as chloroform, dichloromethane, and the like; and a mixed solvent thereof, and a toluene-tetrahydrofuran mixed solvent is particularly preferred.

[0114]

The amount of compound (6) to be used is generally 1 to 3
15 mol, preferably 1 to 2 mol, more preferably 1.2 mol, per 1 mol of compound (5).

[0115]

The base used in this step includes, for example, organic bases such as triethylamine, N,N-diisopropylethylamine,
20 pyridine, 2,6-lutidine, and the like, and among them, triethylamine is preferred.

The amount of the base to be used is generally 1 to 3 mol, preferably 1 to 2 mol, more preferably 1.2 mol, per 1 mol of compound (5).

25 [0116]

The reaction temperature is generally 0°C to 40°C, preferably 10°C to 35°C, more preferably room temperature (15°C to 30°C), and the reaction time is generally about 10 min to 10 hr, preferably about 30 min to 2 hr.

30 [0117]

In Production Method 1, compound (7) prepared as mentioned above can be directly used in step 2.

[0118]

(step 2)

35 The solvent used in this step is not particularly limited

and includes, for example, ethers such as triglyme, tetrahydrofuran, tetrahydropyran, dioxane, and the like, and among them, tetrahydrofuran is preferred.

[0119]

5 The reducing agent used in this step includes, for example, boron hydride, aluminum hydride, and the like. Specific examples of the reducing agent include, but are not particularly limited to, sodium borohydride, lithium borohydride, lithium aluminum hydride, borane-tetrahydrofuran
10 complex, borane-dimethylsulfide complex, sodium borohydride/iodine, sodium borohydride/trifluoroacetic acid, and the like, and among them, boron hydrides such as borane-tetrahydrofuran complex, borane-dimethylsulfide complex, sodium borohydride/iodine, sodium borohydride/trifluoroacetic acid,
15 and the like are preferred.

 The amount of the reducing agent such as borane-tetrahydrofuran complex, borane-dimethylsulfide complex, sodium borohydride/iodine, sodium borohydride/trifluoroacetic acid, and the like to be used is generally 2 to 10 mol, preferably 2
20 to 3 mol, per 1 mol of compound (7).

[0120]

 The reaction temperature is generally the refluxing temperature of the solvent to be used, preferably 40°C to 80°C.

 The reaction time is generally about 30 min to 10 hr,
25 preferably about 1 to 4 hr.

[0121]

(step 3)

 The solvent used in this step is not particularly limited and includes, for example, halogenated hydrocarbons such as
30 dichloromethane, chloroform, dichloroethane, carbon tetrachloride, trichloroethylene, and the like and hydrocarbon solvents such as toluene, n-hexane, isohexane, n-heptane, n-octane, cyclohexane, methylcyclohexane, and the like, and dichloromethane, chloroform, n-hexane, isohexane, n-heptane,
35 cyclohexane, methylcyclohexane, and the like are preferred, and

among them, dichloroethane, chloroform, n-hexane, isohexane, n-heptane, or methylcyclohexane is particularly preferred.

[0122]

Examples of the acid used in this step include protic
5 acids such as hydrogen bromide, hydrogen chloride, hydrogen iodide, and the like, and among them, hydrogen chloride is preferred. As hydrogen chloride, a commercially available product (1.0 M hydrogen chloride-diethyl ether solution or hydrochloric acid) can be directly used.

10 The amount of the acid to be used is generally 1 to 5 mol, preferably 1 to 2 mol, per 1 mol of compound (3a).

[0123]

Compound (4) used in this step is not particularly limited, and a commercially available product or purified
15 product may also be used. In addition, one prepared by a method known per se may also be used. Specific examples of compound (4) include lithium tetrakis(pentafluorophenyl)borate, potassium tetrakis(pentafluorophenyl)borate, lithium tetrakis(heptafluoronaphthyl)borate, potassium
20 tetrakis(heptafluoronaphthyl)borate, chloro magnesium tetrakis(pentafluorophenyl)borate, chloro magnesium tetrakis(heptafluoronaphthyl)borate, bromo magnesium tetrakis(pentafluorophenyl)borate, bromo magnesium tetrakis(heptafluoronaphthyl)borate, lithium
25 tetrakis(nonafluorobiphenyl)borate, potassium tetrakis(nonafluorobiphenyl)borate, chloro magnesium tetrakis(nonafluorobiphenyl)borate, bromo magnesium tetrakis(nonafluorobiphenyl)borate, tri-diethyl ether complex of lithium tetrakis(pentafluorophenyl)borate, mono-diethyl
30 ether complex of lithium tetrakis(pentafluorophenyl)borate, and the like.

The amount of compound (4) to be used is generally 1 to 1.5 mol, preferably 1 mol, per 1 mol of compound (3a).

[0124]

35 The reaction temperature is generally 0°C to 80°C,

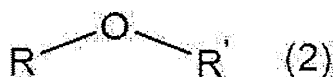
preferably 15°C to 60°C, and the reaction time is generally about 10 min to 10 hr, preferably about 1 to 3 hr.

[0125]

(Production Method 2)

5 Production Method 2 is a method of obtaining a composition containing compound (1a) and compound (2) (the composition of the present invention) by performing the reaction of the aforementioned Production Method 1, step 3, in the presence of a compound represented by the formula (2):

10 [0126]



[0127]

wherein each symbol is as defined above (compound (2)). The method can be performed in the same manner as in the aforementioned Production Method 1, step 3, except
15 that compound (2) is added.

[0128]

The amount of compound (2) to be used is generally 0.01 to 10 mol, preferably 0.1 to 3 mol, per 1 mol of compound (4).

20 [0129]

(Production Method 3)

Production Method 3 is a method of obtaining a composition containing compound (1a) and compound (3a) (the composition of the present invention) by using an excess amount
25 of compound (3a) in the reaction of the aforementioned Production Method 1, step 3. The method can be performed in the same manner as in the aforementioned Production Method 1, step 3, except that an excess amount of compound (3a) is used.

[0130]

30 The amount of compound (3a) to be used is generally 1.01 to 11 mol, preferably 1.1 to 4 mol, per 1 mol of compound (4).

[0131]

The compound of the present invention (compound (1)) or the composition of the present invention is a compound or

composition derived from fluorine-containing alkylamine, and is substantially free of compounds that can be catalyst poison, such as amine compounds with high basicity and high nucleophilicity. Therefore, it is useful as a co-catalyst for the polymerization of olefin, diene, and acetylene.

[0132]

The present invention encompasses production methods of polymers, including polymerizing at least one kind of monomer selected from the group consisting of olefin, diene, and acetylene by using the compound of the present invention (compound (1)) or the composition of the present invention as a co-catalyst.

[0133]

Production of a polymer by using the compound of the present invention (compound (1)) (or composition) as a co-catalyst can be specifically performed according to, for example, the method described in the below-mentioned Experimental Example.

[Example]

[0134]

The present invention is specifically explained in detail in the following by referring to Examples, Production Examples, and Experimental Example; however, the present invention is not limited to those Examples and the like. % means mol/mol% for yield and wt% for others unless particularly indicated. The room temperature refers to a temperature of from 15°C to 30°C unless particularly indicated.

[0135]

Unless otherwise specified, the solvents and reagents used in the following Examples were purchased from vendors such as Tokyo Chemical Industry Co., Ltd., FUJIFILM Wako Pure Chemical Corporation, Junsei Chemical Co., Ltd., KANTO CHEMICAL CO., INC., Combi-Blocks, Inc, and the like. In addition, the deuterated solvents used for NMR measurement were purchased from Cambridge Isotope Laboratories, Inc.

For the analysis, the following instrument was used.

^1H -NMR and ^{19}F -NMR: 400YH manufactured by JEOL Ltd. (JEOL).

[0136]

[Production Example 1]

5 Synthesis of N,N-dioctadecyl-2,2,2-trifluoroacetamide

N,N-Dioctadecylamine (2.0 g, 3.8 mmol) and triethylamine (0.5 g, 5.0 mmol) were dissolved in tetrahydrofuran (10 mL), and trifluoroacetic anhydride (1.0 g, 4.8 mmol) was added at room temperature. The mixture was stirred at room temperature
10 for 1 hr, water was added, and the mixture was extracted with ethyl acetate. The organic layer was washed with 1 M hydrochloric acid and saturated brine, dried over anhydrous magnesium sulfate, and concentrated under reduced pressure. The residue was purified by column chromatography (n-
15 hexane/ethyl acetate=100/0-95/5) to give the title compound (1.87 g, 79%).

^1H NMR (CDCl_3) δ : 0.89 (6H, t), 1.26-1.43 (60H, m), 1.56-1.59 (4H, m), 3.30-3.37 (4H, m);

^{19}F NMR (CDCl_3) δ : -70.1 (3F, s).

20 [0137]

[Production Example 2]

Synthesis of N,N-dioctadecyl-2,2,2-trifluoroethylamine

N,N-Dioctadecyl-2,2,2-trifluoroacetamide (1.0 g, 1.6 mmol) obtained in Production Example 1 was dissolved in
25 tetrahydrofuran (10 mL), 1 M borane-tetrahydrofuran complex solution in tetrahydrofuran (5 mL) was added, and the mixture was refluxed for 3 hr. The mixture was ice-cooled, water was carefully added dropwise thereto, and the mixture was extracted with ethyl acetate. The organic layer was washed with
30 saturated brine, dried over anhydrous magnesium sulfate, and concentrated under reduced pressure to give the title compound (0.87 g, 88%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.25-1.40 (60H, m), 1.42-1.44 (4H, m), 2.56 (4H, t), 3.00 (2H, q);

35 ^{19}F NMR (CDCl_3) δ : -71.3 (3F, t).

[0138]

[Production Example 3]

Synthesis of N,N-dioctadecyl-2,2,2-trifluoroethylamine hydrochloride

5 N,N-Dioctadecyl-2,2,2-trifluoroethylamine (1.0 g, 1.7 mmol) obtained in Production Example 2 was dissolved in n-hexane (10 mL), 1.0 M hydrogen chloride-diethyl ether solution (10 mL) was added, and the mixture was stirred at room temperature for 3 hr. The precipitate was collected by
10 filtration, washed with n-hexane, and dried under reduced pressure to give the title compound (0.987 g, 93%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.40 (60H, m), 1.93 (4H, br s), 3.15 (4H, br s), 3.77 (2H, q);

^{19}F NMR (CDCl_3) δ : -63.4 (3F, t).

15 [0139]

[Example 1]

Synthesis of N,N-dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate

N,N-Dioctadecyl-2,2,2-trifluoroethylamine hydrochloride
20 (0.32 g, 0.5 mmol) obtained in Production Example 3 was dissolved in chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.45 g, 0.5 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by
25 filtration, and the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 45°C to give the title compound (0.62 g, 97%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.40 (60H, m), 1.94 (4H, br s), 3.15 (4H, br s), 3.77 (2H, q);

30 ^{19}F NMR (CDCl_3) δ : -66.7 (3F, t), -134.1 (8H, m), -163.2 (4H, m), -1675.5 (8H, m).

[0140]

[Example 2]

Synthesis of composition containing N,N-dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and
35

ditetradecyl ether

N,N-Dioctadecyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate (0.30 g, 0.22 mmol) obtained
in Example 1 was mixed with ditetradecyl ether (0.09 g, 0.22
5 mmol) to give the title composition.

^1H NMR (CDCl_3) δ : 0.85-0.90 (12H, m), 1.20-1.33 (104H, m),
1.52-1.68 (8H, m), 3.14-3.18 (4H, m), 3.40 (4H, t), 3.62 (2H,
q);

^{19}F NMR (CDCl_3) δ : -66.8 (3F, br s), -134.0 (8F, m), -163.2 (4F,
10 t), -167.5 (8F, m).

[0141]

n-Hexane was added to the composition obtained in Example
2 to prepare a 20 wt% n-hexane solution thereof, and the
solution was confirmed to be a homogeneous solution.

15 [0142]

Isohexane was added to the composition obtained in
Example 2 to prepare a 20 wt% isohexane solution thereof, and
the solution was confirmed to be a homogeneous solution.

[0143]

20 n-Heptane was added to the composition obtained in
Example 2 to prepare a 20 wt% n-heptane solution thereof, and
the solution was confirmed to be a homogeneous solution.

[0144]

ISOPAR E (registered trade mark) was added to the
25 composition obtained in Example 2 to prepare a 20 wt% ISOPAR E
(registered trade mark) solution thereof, and the solution was
confirmed to be a homogeneous solution.

[0145]

Cyclohexane was added to the composition obtained in
30 Example 2 to prepare a 20 wt% cyclohexane solution thereof, and
the solution was confirmed to be a homogeneous solution.

[0146]

Methylcyclohexane was added to the composition obtained
in Example 2 to prepare a 20 wt% methylcyclohexane solution
35 thereof, and the solution was confirmed to be a homogeneous

solution.

[0147]

[Example 3]

Synthesis of composition containing N,N-dioctadecyl-2,2,2-
5 trifluoroethylammonium tetrakis(pentafluorophenyl)borate and
dibutyl ether

N,N-Dioctadecyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate (300 mg, 0.23 mmol) obtained
in Example 1 was mixed with dibutyl ether (62 mg, 0.46 mmol) to
10 give the title composition.

^1H NMR (CDCl_3) δ : 0.87 (6H, t), 0.92 (12H, t), 1.20-1.68 (78H,
m), 3.14-3.18 (4H, m), 3.41 (8H, t), 3.62 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.7 (3F, s), -134.0 (8F, s), -163.3 (4F, t),
-167.5 (8H, m).

15 [0148]

It was confirmed that the composition obtained in Example
3 was dissolved in n-hexane at 30 wt%.

[0149]

[Example 4]

20 Synthesis of N,N-dioctadecyl-N-(2,2,2-trifluoroethyl)ammonium
tetrakis(2-heptafluoronaphthyl)borate

A diethyl ether solution (46.9 wt%) (1.0 g, 0.46 mmol) of
lithium tetrakis(2-heptafluoronaphthyl)borate produced by a
method known per se (e.g., WO 2007/070770) and dioctadecyl-N-
25 (2,2,2-trifluoroethyl)ammonium hydrochloride (0.292 g, 0.46
mmol) were added to dichloromethane (10 mL), and the mixture
was stirred at room temperature and partitioned by adding water.
The organic layer was dried over anhydrous sodium sulfate and
concentrated to give the title compound (0.78 g) as a yellow
30 solid.

^1H NMR (CDCl_3) δ : 0.87 (6H, t), 1.24 (60H, m), 1.62 (4H, m),
3.20 (4H, t), 3.69 (2H, q);

^{19}F NMR (CDCl_3) δ : -63.3 (3F, t), -106.7 (4F, m), -123.4 (4F, m),
-143.3 (4F, m), -146.8 (4F, m), -152.5 (4F, m), -155.8 (4F, m),
35 -157.3 (4F, m).

[0150]

[Production Example 4]

Synthesis of N-benzyl-N-methyl-2,2,2-trifluoroethylamine

N-Methylbenzylamine (manufactured by KANTO CHEMICAL CO.,
5 INC.) (1.1 g, 9.1 mmol) and triethylamine (1.5 g, 15 mmol) were
mixed, and 2,2,2-trifluoroethyl trifluoromethanesulfonate (2.2
g, 9.5 mmol) was added at room temperature. The mixture was
stirred overnight, 1 M hydrochloric acid was added thereto, and
the mixture was extracted with ethyl acetate. The organic
10 layer was washed with water, dried over anhydrous magnesium
sulfate, and concentrated under reduced pressure. The residue
was purified by column chromatography (n-hexane/ethyl
acetate=98/2-90/10) to give the title compound (1.65 g, 86%).
¹H NMR (CDCl₃) δ: 2.43 (3H, s), 3.03 (2H, q), 3.71 (2H, s),
15 7.25-7.34 (5H, m); ¹⁹F NMR (CDCl₃) δ: -69.9 (3F, t).

[0151]

[Production Example 5]

Synthesis of N-benzyl-N-methyl-2,2,2-trifluoroethylamine
hydrochloride

20 To N-benzyl-N-methyl-2,2,2-trifluoroethylamine (1.65 g,
8.1 mmol) obtained in Production Example 4 was added 1 M
hydrogen chloride-diethyl ether solution (20 mL). The mixture
was stirred at room temperature for 1 hr, and the solvent was
evaporated under reduced pressure to give the title compound
25 (1.97 g, 100%).
¹H NMR (CDCl₃) δ: 2.96 (3H, s), 3.70-3.81 (2H, m), 4.46 (2H, s),
7.41-7.52 (3H, m), 7.59-7.67 (2H, m);
¹⁹F NMR (CDCl₃) δ: -63.2 (3F, br s).

[0152]

30 [Example 5]

Synthesis of N-benzyl-N-methyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate

N-Benzyl-N-methyl-2,2,2-trifluoroethylamine hydrochloride
(0.30 g, 1.25 mmol) obtained in Production Example 5 was
35 dissolved in chloroform (30 mL), lithium

tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (1.10 g, 1.21 mol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced
5 pressure. The residue was dried under reduced pressure at 45°C to give the title compound (1.07 g, 93%).

^1H NMR (CDCl_3) δ : 2.97 (3H, s), 3.60 (2H, q), 4.33 (2H, s), 7.41-7.57 (5H, m);

^{19}F NMR (CDCl_3) δ : -65.3 (3F, t), -134.0 (8H, m), -163.4 (4F, t),
10 -167.6 (8F, m).

[0153]

[Production Example 6]

Synthesis of N,N-bis(pentafluorophenylmethyl)-1-butylamine

Pentafluorobenzaldehyde (3.0 g, 15.3 mmol), 1-butylamine
15 (0.50 g, 7.0 mmol), and acetic acid (0.40 g) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxyborohydride (3.50 g, 17.0 mmol) was added, and the mixture was stirred at room temperature for 15 hr. The reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and
20 extracted with diethyl ether. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-90/10) to give the title compound (2.65 g, 90%).

25 ^1H NMR (CDCl_3) δ : 0.81 (3H, t), 1.17-1.23 (2H, m), 1.43-1.48 (2H, m), 2.40 (2H, t), 3.71 (4H, s).

^{19}F NMR (CDCl_3) δ : -143.8 (4F, dd), -156.1 (2H, t), -163.4 (4F, m).

[0154]

30 [Production Example 7]

Synthesis of N,N-bis(pentafluorophenylmethyl)-1-butylamine hydrochloride

N,N-Bis(pentafluorophenylmethyl)-1-butylamine (2.30 g, 5.1 mmol) obtained in Production Example 6 was dissolved in n-
35 hexane (30 mL), and 1 M hydrogen chloride-diethyl ether (20 mL)

was added. The mixture was stirred at room temperature for 1 hr, and the solvent was evaporated under reduced pressure to give the title compound (1.98 g, 80%).

¹H NMR (CDCl₃) δ: 0.99 (3H, t), 1.38-1.43 (2H, m), 2.03-2.11
5 (2H, m), 2.99-3.03 (2H, m), 4.36 (4H, m);
¹⁹F NMR (CDCl₃) δ: -136.8 (4F, d), -147.9 (2F, t), -159.5 (4H,
m).

[0155]

[Example 6]

10 Synthesis of N,N-bis(pentafluorophenylmethyl)-1-butylammonium
tetrakis(pentafluorophenyl)borate

N,N-Bis(pentafluorophenylmethyl)-1-butylamine
hydrochloride (0.93 g, 1.02 mmol) obtained in Production
Example 7 was dissolved in chloroform (30 mL), lithium
15 tetrakis(pentafluorophenyl)borate tri-diethyl ether complex
(0.50 g, 1.03 mol) was added, and the mixture was stirred at
room temperature for 1 hr. Insoluble material was collected by
filtration. The insoluble material was dissolved in water and
dichloromethane, and the organic layer was separated. The
20 organic layer was dried over anhydrous magnesium sulfate and
concentrated under reduced pressure to give the title compound
(0.65 g, 65%).

¹H NMR (DMSO-d₆) δ: 0.77 (3H, t), 1.13-1.22 (2H, m), 1.42-1.48
(2H, m), 2.43-2.50 (2H, m), 4.62 (2H, br s);
25 ¹⁹F NMR (DMSO-d₆) δ: -132.7 (8F, m), -143.0 (4F, br s), -156.3
(2F, br s), -161.7 (4F, t), -163.3 (4F, br s), -166.2 (8F, m).

[0156]

[Production Example 8]

Synthesis of N,N-dioctadecyl-(2,3,4,5,6-
30 pentafluorophenyl)methyl-1-amine

Pentafluorobenzaldehyde (0.50 g, 2.60 mmol) and N,N-
dioctadecylamine (1.50 g, 2.87 mmol) were dissolved in
tetrahydrofuran (30 mL), sodium triacetoxyborohydride (1.00 g,
4.72 mmol) was added, and the mixture was stirred at room
35 temperature for 15 hr. The reaction mixture was basified by

adding saturated aqueous sodium hydrogen carbonate solution and extracted with diethyl ether. The organic layer was washed with saturated brine, dried over anhydrous magnesium sulfate, and concentrated under reduced pressure. The residue was
5 purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (1.05 g, 52%).
¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.20-1.30 (64H, m), 2.38 (4H, t), 3.69 (2H, s);
¹⁹F NMR (CDCl₃) δ: -143.0 (8F, m), -157.1 (4F, m), -163.8 (8F,
10 m).

[0157]

[Production Example 9]

Synthesis of N,N-dioctadecyl-(2,3,4,5,6-pentafluorophenyl)methyl-1-amine hydrochloride

15 N,N-Dioctadecyl-(2,3,4,5,6-pentafluorophenyl)methyl-1-amine (2.95 g, 4.2 mmol) obtained in Production Example 8 was dissolved in n-hexane (30 mL), and 1 M hydrogen chloride-diethyl ether (20 mL) was added. The reaction mixture was stirred at room temperature for 1 hr, and the solvent was
20 evaporated under reduced pressure to give the title compound (3.05 g, 98%).

¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.21-1.35 (60H, m), 1.88-1.93 (4H, m), 2.92-3.02 (4H, m), 4.32 (2H, s);

¹⁹F NMR (CDCl₃) δ: -137.4 (1F, d), -148.6 (2F, t), -159.6 (2F,
25 m).

[0158]

[Example 7]

Synthesis of N,N-dioctadecyl-(2,3,4,5,6-pentafluorophenyl)methyl-1-ammonium

30 tetrakis(pentafluorophenyl)borate

N,N-Dioctadecyl-(2,3,4,5,6-pentafluorophenyl)methyl-1-amine hydrochloride (0.50 g, 0.68 mmol) obtained in Production Example 9 was dissolved in chloroform (30 mL), lithium
tetrakis(pentafluorophenyl)borate tri-diethyl ether complex
35 (0.58 g, 0.60 mol) was added, and the mixture was stirred at

room temperature for 1 hr. Insoluble material was collected by filtration, and the filtrate was concentrated under reduced pressure at 45°C to give the title compound (0.83 g, 98%).

¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.21-1.30 (60H, m), 1.73 (4H, br s), 3.05 (4H, br s), 4.27 (2H, s);

¹⁹F NMR (CDCl₃) δ: -134.1 (8H, br s), -139.8 (2H, br s), -146.0 (1H, br s), -158.5 (2H, br s), -163.6 (4H, t), -167.8 (8H, t).
[0159]

Methylcyclohexane was added to the compound of Example 7 to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0160]

[Production Example 10]

Synthesis of N,N-dicyclohexyl-2,2,2-trifluoroacetamide

Dicyclohexylamine (2.0 g, 11 mmol) and triethylamine (1.2 g, 12 mmol) were dissolved in tetrahydrofuran (50 mL), and trifluoroacetic anhydride (2.3 g, 11 mmol) was added dropwise at room temperature. After stirring at room temperature for 1 hr, 1 M hydrochloric acid was added. The mixture was extracted with n-hexane, dried over anhydrous magnesium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-90/10) to give the title compound (2.65 g, 87%).

¹H NMR (CDCl₃) δ: 1.08-1.84 (14H, m), 2.38-2.48 (4H, m), 3.01-3.08 (2H, m), 3.65-3.68 (2H, m);

¹⁹F NMR (CDCl₃) δ: -70.3 (3F, s).

[0161]

[Production Example 11]

Synthesis of N,N-dicyclohexyl-2,2,2-trifluoroethylamine

N,N-Dicyclohexyl-2,2,2-trifluoroacetamide (2.65 g, 9.56 mmol) obtained in Production Example 10 was dissolved in tetrahydrofuran (30 mL), and 1.0 M borane-tetrahydrofuran complex solution in tetrahydrofuran (20 mL) was added at room temperature. The reaction mixture was stirred at 60°C for 5 hr, allowed to cool to room temperature, and water (30 mL) was

carefully added dropwise under ice-cooling. The mixture was extracted with n-hexane, washed with saturated brine, dried over anhydrous magnesium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel
5 column chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (2.35 g, 93%).

^1H NMR (CDCl_3) δ : 0.87-1.25 (10H, m), 1.56-1.78 (10H, m), 2.51-2.57 (2H, m), 3.11 (2H, q);

^{19}F NMR (CDCl_3) δ : -72.9 (3F, t).

10 [0162]

[Production Example 12]

Synthesis of N,N-dicyclohexyl-2,2,2-trifluoroethylamine hydrochloride

N,N-Dicyclohexyl-2,2,2-trifluoroethylamine (2.35 g, 8.9
15 mmol) obtained in Production Example 11 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (2.65 g, 99%).

20 ^1H NMR (CDCl_3) δ : 1.18-1.40 (6H, m), 1.69-1.81 (6H, m), 1.95-2.07 (4H, m), 2.19 (4H, br s), 3.44-3.52 (2H, m), 3.76 (2H, q);

^{19}F NMR (CDCl_3) δ : -59.7 (3F, t).

[0163]

[Example 8]

25 Synthesis of N,N-dicyclohexyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate

N,N-Dicyclohexyl-2,2,2-trifluoroethylamine hydrochloride (0.33 g, 1.1 mmol) obtained in Production Example 12 was dissolved in chloroform (30 mL), lithium
30 tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (1.0 g, 1.1 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 45°C
35 to give the title compound (0.98 g, 94%).

^1H NMR (CDCl_3) δ : 1.13-2.06 (20H, m), 3.42-3.48 (2H, m), 3.66 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.0 (3F, s), -134.0 (8H, m), -163.8 (4F, m), -167.8 (8F, m).

5 [0164]

[Production Example 13]

Synthesis of N,N-dihexyl-2,2-difluoroethylamine

1-Hexanal (2.5 g, 25 mmol) and 2,2-difluoroethylamine (1 g, 12 mmol) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxyborohydride (6 g, 28 mmol) was added, and the mixture
10 was stirred at room temperature for 15 hr. The reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with diethyl ether. The organic layer was washed with saturated brine, dried over
15 anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (2.98 g, 97%).

^1H NMR (CDCl_3) δ : 0.87 (6H, t), 1.21-1.44 (16H, m), 2.46-2.50 (4H, m), 2.76 (2H, dt), 5.74 (1H, tt);

^{19}F NMR (CDCl_3) δ : -120.4 (2F, dt).

[0165]

[Production Example 14]

Synthesis of N,N-dihexyl-2,2-difluoroethylamine hydrochloride

25 N,N-Dihexyl-2,2-difluoroethylamine (2.95 g, 11.8 mmol) obtained in Production Example 13 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the
30 title compound (3.05 g, 90%).

^1H NMR (CDCl_3) δ : 0.90 (6H, t), 1.30-1.41 (12H, m), 1.78-1.86 (4H, m), 3.07-3.17 (4H, m), 3.36-3.45 (2H, m), 6.78 (1H, tt);

^{19}F NMR (CDCl_3) δ : -118.3 (2H, dt).

[0166]

35 [Example 9]

Synthesis of N,N-dihexyl-2,2-difluoroethylammonium
tetrakis(pentafluorophenyl)borate

N,N-Dihexyl-2,2-difluoroethylamine hydrochloride (0.40 g,
1.4 mmol) obtained in Production Example 14 was dissolved in
5 chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate
tri-diethyl ether complex (1.25 g, 1.38 mmol) was added, and
the mixture was stirred at room temperature for 1 hr.
Insoluble material was removed by filtration, and the filtrate
was concentrated under reduced pressure. The residue was dried
10 under reduced pressure at 45°C to give the title compound (1.04
g, 81%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.24-1.36 (12H, m), 1.64-1.72
(4H, m), 3.16-3.21 (4H, m), 3.40-3.48 (2H, m), 6.18 (1H, t);
 ^{19}F NMR (CDCl_3) δ : -65.3 (2F, dt), -134.1 (8H, m), -163.4 (4F,
15 t), -167.6 (8F, m).

[0167]

[Production Example 15]

Synthesis of N,N-dihexyl-3,3,3-trifluoropropylamine

1-Hexanal (1 g, 10 mmol), 2,2-difluoroethylamine (0.5 g,
20 4.0 mmol), and acetic acid (0.3 mL) were dissolved in
tetrahydrofuran (30 mL), sodium triacetoxyborohydride (2.5 g,
12 mmol) was added, and the mixture was stirred at room
temperature for 15 hr. The mixture was basified by adding
saturated aqueous sodium hydrogen carbonate solution and
25 extracted with n-hexane. The organic layer was washed with
saturated brine, dried over anhydrous sodium sulfate, and
concentrated under reduced pressure. The residue was purified
by silica gel column chromatography (n-hexane/ethyl
acetate=100/0-95/5) to give the title compound (1.35 g, 100%).
30 ^1H NMR (CDCl_3) δ : 0.87-0.92 (6H, m), 1.25-1.45 (16H, m), 2.16-
2.29 (2H, m), 2.36-2.41 (4H, m), 2.67-2.71 (2H, m);
 ^{19}F NMR (CDCl_3) δ : -66.5 (3F, t).

[0168]

[Production Example 16]

35 Synthesis of N,N-dihexyl-3,3,3-trifluoropropylamine

hydrochloride

N,N-Dihexyl-3,3,3-trifluoropropylamine (1.0 g, 3.55 mmol) obtained in Production Example 15 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (1.1 g, 97%).

^1H NMR (CDCl_3) δ : 0.90 (6H, t), 1.31-1.39 (12H, m), 1.77-1.84 (4H, m), 2.87-2.99 (1H, m), 3.00-3.05 (3H, m), 3.18 (1H, m);

10 ^{19}F NMR (CDCl_3) δ : -66.6 (3F, t)

[0169]

[Example 10]

Synthesis of N,N-dihexyl-3,3,3-trifluoropropylammonium tetrakis(pentafluorophenyl)borate

15 N,N-Dihexyl-3,3,3-trifluoropropylamine hydrochloride (0.30 g, 0.94 mmol) obtained in Production Example 16 was dissolved in chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.85 g, 0.94 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 45°C to give the title compound (0.90 g, 100%).

^1H NMR (CDCl_3) δ : 0.87 (6H, t), 1.21-1.35 (12H, m), 1.63-1.69 (4H, m), 2.56-2.61 (2H, m), 3.07 (4H, m), 3.30-3.35 (2H, m);

25 ^{19}F NMR (CDCl_3) δ : -66.4 (3F, t), -134.1 (8F, br s), -163.4 (4F, t), -167.7 (8F, m).

[0170]

[Production Example 17]

30 Synthesis of N,N-dioctadecyl-2,2,3,3,3-pentafluoropropylamine

1-Octadecanal (1.5 g, 5.59 mmol), 2,2,3,3,3-pentafluoropropylamine (0.40 g, 2.7 mmol), and acetic acid (0.3 mL) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxyborohydride (1.2 g, 5.7 mmol) was added, and the mixture was stirred at room temperature for 15 hr. The mixture

was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The
5 residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (1.65 g, 100%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.25-1.43 (64H, m), 2.56 (4H, br s), 2.95-3.10 (2H, m);

10 ^{19}F NMR (CDCl_3) δ : -85.2 (3F, br s), -121.2 (2F, br s).

[0171]

[Production Example 18]

Synthesis of N,N-dioctadecyl-2,2,3,3,3-pentafluoropropylamine hydrochloride

15 N,N-Dioctadecyl-2,2,3,3,3-pentafluoropropylamine (1.5 g, 2.29 mmol) obtained in Production Example 17 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced
20 pressure to give the title compound (1.45 g, 92%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.40 (64H, m), 1.94 (4H, br s), 3.22 (4H, t), 3.77 (2H, t);

^{19}F NMR (CDCl_3) δ : -86.3 (3F, br s), -117.8 (2H, t).

[0172]

25 [Example 11]

Synthesis of N,N-dioctadecyl-2,2,3,3,3-pentafluoropropylammonium tetrakis(pentafluorophenyl)borate

N,N-Dioctadecyl-2,2,3,3,3-pentafluoropropylamine hydrochloride (0.50 g, 0.72 mmol) obtained in Production
30 Example 18 was dissolved in chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.65 g, 0.72 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced
35 pressure. The residue was dried under reduced pressure at 45°C

to give the title compound (0.89 g, 93%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.18-1.31 (64H, m), 1.72 (4H, br s), 3.21-3.23 (4H, m), 3.67 (2H, t);

^{19}F NMR (CDCl_3) δ : -85.9 (3F, t), -118.6 (2F, m), -134.0 (8F, br s), -163.4 (4F, t), -167.6 (8F, m).

[0173]

Methylcyclohexane was added to the compound of Example 11 to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

10 [0174]

[Production Example 19]

Synthesis of N,N-dioctyl-2,2,2-trifluoroethylamine

1-Octanal (5.5 g, 43 mmol), 2,2,2-trifluoroethylamine (2.0 g, 20 mmol), and acetic acid (0.5 mL) were dissolved in tetrahydrofuran (50 mL), sodium triacetoxyborohydride (10 g, 47 mmol) was added, and the mixture was stirred at room temperature for 15 hr. The reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (5.56 g, 85%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.27-1.43 (24H, m), 2.56 (4H, t), 3.00 (2H, q);

^{19}F NMR (CDCl_3) δ : -71.3 (3F, s).

[0175]

[Production Example 20]

Synthesis of N,N-dioctyl -2,2,2-trifluoroethylamine hydrochloride

N,N-Dioctyl-2,2,2-trifluoroethylamine (1.5 g, 4.64 mmol) obtained in Production Example 19 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the

title compound (1.45 g, 87%).

¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.26-1.34 (20H, m), 1.94 (4H, br s), 3.15 (4H, br s), 3.78 (2H, q);

¹⁹F NMR (CDCl₃) δ: -63.4 (3F, t).

5 [0176]

[Example 12]

Synthesis of N,N-dioctyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate

N,N-Dioctyl-2,2,2-trifluoroethylamine hydrochloride (0.50
10 g, 1.39 mmol) obtained in Production Example 20 was dissolved
in chloroform (10 mL), lithium
tetrakis(pentafluorophenyl)borate tri-diethyl ether complex
(1.25 g, 1.38 mmol) was added, and the mixture was stirred at
room temperature for 1 hr. Insoluble material was removed by
15 filtration, and the filtrate was concentrated under reduced
pressure. The residue was dried under reduced pressure at 45°C
to give the title compound (1.25 g, 87%).

¹H NMR (CDCl₃) δ: 0.86 (6H, t), 1.19-1.29 (20H, m), 1.67-1.73
(4H, m), 3.18-3.22 (4H, m), 3.62-3.67 (2H, m);

20 ¹⁹F NMR (CDCl₃) δ: -66.0 (3F, t), -134.1 (8F, br s), -163.4 (4F,
t), -167.6 (8F, m).

[0177]

[Production Example 21]

Synthesis of N,N-didodecyl-2,2,2-trifluoroethylamine

25 1-Dodecanal (5.5 g, 30 mmol), 2,2,2-trifluoroethylamine
(1.5 g, 15 mmol), and acetic acid (0.5 mL) were dissolved in
tetrahydrofuran (50 mL), sodium triacetoxyborohydride (7 g, 33
mmol) was added, and the mixture was stirred at room
temperature for 15 hr. The reaction mixture was basified by
30 adding saturated aqueous sodium hydrogen carbonate solution and
extracted with n-hexane. The organic layer was washed with
saturated brine, dried over anhydrous sodium sulfate, and
concentrated under reduced pressure. The residue was purified
by silica gel column chromatography (n-hexane/ethyl
35 acetate=100/0-95/5) to give the title compound (6.5 g, 99%).

¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.26-1.43 (40H, m), 2.56 (4H, t), 3.00 (2H, q);

¹⁹F NMR (CDCl₃) δ: -71.3 (3F, t).

[0178]

5 [Production Example 22]

Synthesis of N,N-didodecyl-2,2,2-trifluoroethylamine hydrochloride

N,N-Didodecyl-2,2,2-trifluoroethylamine (0.5 g, 1.06 mmol) obtained in Production Example 21 was dissolved in n-
10 hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (2.09 g, 96%).

¹H NMR (CDCl₃) δ: 0.87 (6H, t), 1.26-1.34 (36H, m), 1.93 (4H, br s), 3.15 (4H, br s), 3.78 (2H, q);

¹⁹F NMR (CDCl₃) δ: -63.4 (3F, t).

[0179]

[Example 13]

Synthesis of N,N-didodecyl-2,2,2-trifluoroethylammonium
20 tetrakis(pentafluorophenyl)borate

N,N-Didodecyl-2,2,2-trifluoroethylamine hydrochloride (0.50 g, 1.06 mmol) obtained in Production Example 22 was dissolved in chloroform (30 mL), lithium
tetrakis(pentafluorophenyl)borate tri-diethyl ether complex
25 (0.96 g, 1.06 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 45°C to give the title compound (1.19 g, 100%).

30 ¹H NMR (CDCl₃) δ: 0.87 (6H, t), 1.19-1.29 (36H, m), 1.65-1.71 (4H, m), 3.15-3.22 (4H, m), 3.65 (2H, q);

¹⁹F NMR (CDCl₃) δ: -66.4 (3F, br s), -134.0 (8F, m), -163.4 (4F, t), -167.6 (8F, m).

[0180]

35 Methylcyclohexane was added to the compound of Example 13

to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0181]

[Production Example 23]

5 Synthesis of N,N-dioctadecyl-2,2-difluoroethylamine

1-Octadecanal (3.0 g, 11 mmol), 2,2-difluoroethylamine (0.45 g, 5.6 mmol), and acetic acid (0.3 mL) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxyborohydride (2.50 g, 12 mmol) was added, and the mixture was stirred at room
10 temperature for 15 hr. The reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified
15 by silica gel column chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (2.9 g, 89%).
 ^1H NMR (CDCl_3) δ : 0.89(6H, t), 1.17-1.47 (64H, m), 2.49 (4H, t), 2.77 (2H, dt), 5.75 (1H, t);
 ^{19}F NMR (CDCl_3) δ : -120.4 (2H, dt).

20 [0182]

[Production Example 24]

Synthesis of N,N-dioctadecyl-2,2-difluoroethylamine hydrochloride

N,N-Dioctadecyl-2,2-difluoroethylamine (2.0 g, 3.41 mmol)
25 obtained in Production Example 23 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (2.09 g, 98%).
30 ^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.18-1.41 (58H, m), 1.79-1.83 (4H, m), 3.07-3.11 (4H, m), 3.32-3.38 (4H, m), 6.78 (1H, t);
 ^{19}F NMR (CDCl_3) δ : -118.2 (2F, dt).

[0183]

[Example 14]

35 Synthesis of N,N-dioctadecyl-2,2-difluoroethylammonium

tetrakis(pentafluorophenyl)borate

N,N-Dioctadecyl-2,2-difluoroethylamine hydrochloride (0.50 g, 0.80 mmol) obtained in Production Example 24 was dissolved in chloroform (30 mL), lithium
5 tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.72 g, 0.79 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 45°C
10 to give the title compound (0.97 g, 97%).

¹H NMR (CDCl₃) δ: 0.86 (3H, t), 1.17-1.43 (58H, m), 1.71 (4H, br s), 3.16 (4H, br s), 3.42 (2H, t), 6.25 (1H, t);

¹⁹F NMR (CDCl₃) δ: -121.3 (2F, d), -134.0 (8F, d), -163.4 (8F, t), -167.6 (8F, t).

15 [0184]

Methylcyclohexane was added to the compound of Example 14 to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0185]

20 [Production Example 25]

Synthesis of 1-(2,2,2-trifluoroethyl)piperidine hydrochloride

1-(2,2,2-Trifluoroethyl)piperidine (1.5 g, 8.97 mmol) was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred
25 for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (1.85 g, 100%).

¹H NMR (CDCl₃) δ: 1.43-1.94 (4H, m), 2.37 (2H, br s), 3.10 (2H, br s), 3.56 (2H, br s), 3.77 (2H, t);

¹⁹F NMR (CDCl₃) δ: -63.0 (3F, t).

30 [0186]

[Example 15]

Synthesis of 1-(2,2,2-trifluoroethyl)piperidinium tetrakis(pentafluorophenyl)borate

1-(2,2,2-Trifluoroethyl)piperidine hydrochloride (0.30 g,
35 1.47 mmol) obtained in Production Example 25 was dissolved in

chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (1.33 g, 1.46 mmol) was added, and the mixture was stirred at room temperature for 1 hr.

Insoluble material was removed by filtration, and the filtrate
5 was concentrated under reduced pressure. The residue was dried under reduced pressure at 45°C to give the title compound (1.19 g, 96%).

^1H NMR (CDCl_3) δ : 1.47-1.62 (2H, m), 1.92-1.99 (2H, m), 2.10-2.20 (2H, m), 3.01 (2H, br s), 3.57-3.63 (4H, m);

10 ^{19}F NMR (CDCl_3) δ : -63.2 (3F, t), -134.0 (8F, br s), -163.8 (4F, t), -167.9 (8F, m).

[0187]

[Production Example 26]

Synthesis of N,N-dioctadecyl-3,3,3-trifluoropropylamine

15 1-Octadecanal (2.4 g, 8.94 mmol), 3,3,3-trifluoropropylamine (0.50 g, 4.42 mmol), and acetic acid (0.3 mL) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxymethylborohydride (2.10 g, 9.9 mmol) was added, and the mixture was stirred at room temperature for 15 hr. The
20 reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column
25 chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (2.4 g, 88%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.18-1.43 (64H, m), 2.23-2.27 (2H, m), 2.38-2.44 (4H, m), 2.70 (2H, t);

^{19}F NMR (CDCl_3) δ : -66.5 (3F, t).

30 [0188]

[Production Example 27]

Synthesis of N,N-dioctadecyl-3,3,3-trifluoropropylamine hydrochloride

N,N-Dioctadecyl-3,3,3-trifluoropropylamine (1.5 g, 2.43
35 mmol) obtained in Production Example 26 was dissolved in n-

hexane (10 mL), 1.0 M hydrogen chloride-diethyl ether solution (10 mL) was added, and the mixture was stirred for 3 hr. The precipitate was collected by filtration, washed with n-hexane, and dried under reduced pressure to give the title compound

5 (1.45 g, 91%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.40 (60H, m), 1.70-1.81 (4H, m), 2.86-3.03 (6H, m), 3.17-3.22 (2H, m);

^{19}F NMR (CDCl_3) δ : -66.6 (3F, t).

[0189]

10 [Example 16]

Synthesis of N,N-dioctadecyl-3,3,3-trifluoropropylammonium tetrakis(pentafluorophenyl)borate

N,N-Dioctadecyl-3,3,3-trifluoropropylamine hydrochloride (0.50 g, 0.764 mmol) obtained in Production Example 27 was
15 dissolved in chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.65 g, 0.72 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced
20 pressure. The residue was dried under reduced pressure at 70°C to give the title compound (0.89 g, 96%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.18-1.38 (60H, m), 1.65-1.72 (4H, m), 2.60-2.67 (2H, m), 3.04-3.07 (4H, m), 3.27-3.32 (2H, m);

25 ^{19}F NMR (CDCl_3) δ : -64.4 (3F, t), -131.8 (8F, m), -161.4 (4F, m), -165.6 (8F, m).

[0190]

Methylcyclohexane was added to the compound of Example 16 to prepare a 10 wt% methylcyclohexane solution thereof, and the
30 solution was confirmed to be a homogeneous solution.

[0191]

[Production Example 28]

Synthesis of N,N-dioctadecyl-2-fluoroethylamine

1-Octadecanal (3.0 g, 11 mmol) and a tert-butanol
35 solution (10 wt%, 3.3 g, 5.2 mmol) of 2-fluoroethylamine, and

acetic acid (0.3 mL) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxyborohydride (2.50 g, 12 mmol) was added, and the mixture was stirred at room temperature for 15 hr. The reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-90/10) to give the title compound (2.02 g, 32%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.18-1.64 (65H, m), 2.44-2.51 (4H, m), 2.77 (2H, dt), 4.50 (1H, dt);

^{19}F NMR (CDCl_3) δ : -2220.6 (1F, br s).

[0192]

15 [Production Example 29]

Synthesis of N,N-dioctadecyl-2-fluoroethylamine hydrochloride

N,N-Dioctadecyl-2-fluoroethylamine (1.50 g, 2.64 mmol) obtained in Production Example 28 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (1.45 g, 91%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.40 (60H, m), 1.78-1.82 (4H, m), 3.04-3.11 (4H, m), 3.32-3.40 (2H, m), 5.02 (2H, dt);

25 ^{19}F NMR (CDCl_3) δ : -223.5 (1F, m).

[0193]

[Example 17]

Synthesis of N,N-dioctadecyl-2-fluoroethylammonium tetrakis(pentafluorophenyl)borate

30 N,N-Dioctadecyl-2-fluoroethylamine hydrochloride (0.50 g, 0.83 mmol) obtained in Production Example 29 was dissolved in chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.75 g, 0.83 mmol) was added, and the mixture was stirred at room temperature for 1 hr.

35 Insoluble material was removed by filtration, and the filtrate

was concentrated under reduced pressure. The residue was dried under reduced pressure at 70°C to give the title compound (0.89 g, 86%).

¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.18-1.31 (60H, m), 1.65-1.73
5 (4H, m), 3.11-3.13 (4H, m), 3.34-3.43 (2H, m), 4.76 (2H, dt);
¹⁹F NMR (CDCl₃) δ: -133.9 (8F, br s), -163.3 (4F, t), -167.5 (8F, m), -223.4 (1H, br s).
[0194]

Methylcyclohexane was added to the compound of Example 17
10 to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0195]

[Production Example 30]

Synthesis of 1,4-bis(2,2,2-trifluoroethyl)piperazine

15 To 1,4-bis(trifluoroacetyl)piperazine (3.5 g, 13 mmol) in tetrahydrofuran (30 mL) was added a tetrahydrofuran solution (40 mL, 40 mmol) of 1 M borane-tetrahydrofuran complex at room temperature, and the mixture was stirred at 60°C for 3 hr. The mixture was ice-cooled, water was carefully added, and the
20 mixture was extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=95/5-80/20) to give the title compound
25 (2.08 g, 66%).

¹H NMR (CDCl₃) δ: 2.71 (8H, s), 2.96 (4H, q);

¹⁹F NMR (CDCl₃) δ: -70.2 (6F, t).

[0196]

[Production Example 31]

30 Synthesis of 1,4-bis(2,2,2-trifluoroethyl)piperazine dihydrochloride

1,4-Bis(2,2,2-trifluoroethyl)piperazine (1.50 g, 6.0 mmol) obtained in Production Example 30 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution
35 (20 mL) was added, and the mixture was stirred for 1 hr. The

mixture was concentrated under reduced pressure to give the title compound (1.85 g, 96%).

^1H NMR (DMSO- d_6) δ : 3.03 (8H, br s), 3.67 (4H, br s);

^{19}F NMR (DMSO- d_6) δ : -65.8 (6F, br s).

5 [0197]

[Example 18]

Synthesis of 1,4-bis(2,2,2-trifluoroethyl)piperazinium
bis[tetrakis(pentafluorophenyl)borate]

1,4-Bis(2,2,2-trifluoroethyl)piperazine dihydrochloride
10 (0.50 g, 1.55 mmol) obtained in Production Example 31 was
dissolved in dichloromethane (50 mL), lithium
tetrakis(pentafluorophenyl)borate tri-diethyl ether complex
(2.81 g, 3.09 mmol) was added, and the mixture was stirred at
room temperature for 3 hr. Insoluble material was collected by
15 filtration, dissolved in water and dichloromethane, and the
organic layer was separated. The organic layer was dried over
anhydrous magnesium sulfate, and concentrated under reduced
pressure to give the title compound (0.87 g, 35%).

^1H NMR (DMSO- d_6) δ : 2.78 (8H, br s), 3.93 (4H, br s);

20 ^{19}F NMR (DMSO- d_6) δ : -67.6 (6F, br s), -132.7 (16F, br s), -
161.6 (8F, t), -166.2 (16F, t).

[0198]

[Production Example 32]

Synthesis of N,N-didocosyl-2,2,2-trifluoroethylamine

25 1-Docosanal (4.0 g, 12.3 mmol), 2,2,2-trifluoroethylamine
(0.50 g, 5.0 mmol), and acetic acid (0.05 mL) were dissolved in
tetrahydrofuran (50 mL), sodium triacetoxymethylborohydride (2.80 g,
13 mmol) was added, and the mixture was stirred at room
temperature for 15 hr. The reaction mixture was basified by
30 adding saturated aqueous sodium hydrogen carbonate solution and
extracted with n-hexane. The organic layer was washed with
saturated brine, dried over anhydrous sodium sulfate, and
concentrated under reduced pressure. The residue was purified
by silica gel column chromatography (n-hexane/ethyl
35 acetate=100/0-95/5) to give the title compound (1.38 g, 40%).

¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.25-1.30 (76H, m), 1.40-1.43 (4H, m), 2.56 (4H, t), 3.00 (2H, q);
¹⁹F NMR (CDCl₃) δ: -71.3 (3F, t).

[0199]

5 [Production Example 33]

Synthesis of N,N-didocosyl-2,2,2-trifluoroethylamine hydrochloride

N,N-Didocosyl-2,2,2-trifluoroethylamine (1.30 g, 1.82 mmol) obtained in Production Example 32 was dissolved in n-
10 hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (10 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (1.34 g, 98%).

¹H NMR (CDCl₃) δ: 0.88 (6H, t), 1.25-1.40 (76H, m), 1.85-2.00
15 (4H, m), 3.14 (4H, t), 3.75 (2H, q);
¹⁹F NMR (CDCl₃) δ: -63.5 (3F, t).

[0200]

[Example 19]

Synthesis of N,N-didocosyl-2,2,2-trifluoroethylammonium
20 tetrakis(pentafluorophenyl)borate

N,N-Didocosyl-2,2,2-trifluoroethylamine hydrochloride (0.40 g, 0.53 mmol) obtained in Production Example 33, lithium tetrakis(pentafluorophenyl)borate mono-diethyl ether complex (0.41 g, 0.53 mmol), and methylcyclohexane (20 mL) were mixed,
25 and the mixture was stirred at room temperature for 3 hr. The reaction mixture was washed with water, and the organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure at 45°C to give the title compound (0.65 g, 88%).

30 ¹H NMR (CDCl₃) δ: 0.87 (6H, t), 1.25-1.29 (76H, m), 1.60-1.65 (4H, m), 3.12-3.16 (4H, m), 3.60 (2H, q);
¹⁹F NMR (CDCl₃) δ: -65.9 (3F, t), -132.8 (8F, t), -162.3 (4F, m), -166.2 (8F, m).

[0201]

35 Methylcyclohexane was added to the compound of Example 19

to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0202]

[Production Example 34]

5 Synthesis of N,N-(3,7,11,15-tetramethylhexadecyl)-2,2,2-trifluoroethylamine

3,7,11,15-Tetramethylhexadecanal (2.3 g, 7.8 mmol), 2,2,2-trifluoroethylamine (0.20 g, 2.0 mmol), and acetic acid (0.3 mL) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxymethylborohydride (1.5 g, 7.8 mmol) was added, and the
10 mixture was stirred at room temperature for 15 hr. The reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over
15 anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (1.13 g, 85%).

^1H NMR (CDCl_3) δ : 0.84-0.88 (30H, m), 1.07-1.56 (48H, m), 2.57-
20 2.61 (4H, m), 3.00 (2H, q);

^{19}F NMR (CDCl_3) δ : -70.9 (3F, t).

[0203]

[Production Example 35]

Synthesis of N,N-(3,7,11,15-tetramethylhexadecyl)-2,2,2-trifluoroethylamine hydrochloride
25

N,N-(3,7,11,15-Tetramethylhexadecyl)-2,2,2-trifluoroethylamine (2.35 g, 3.56 mmol) obtained in Production Example 34 was dissolved in n-hexane (20 mL), 1.0 M hydrogen chloride-diethyl ether solution (10 mL) was added, and the
30 mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (2.48 g, 100%).

^1H NMR (CDCl_3) δ : 0.84-0.95 (30H, m), 1.13-1.57 (48H, m), 3.13-3.25 (4H, m), 3.76 (2H, q);

35 ^{19}F NMR (CDCl_3) δ : -63.4 (3F, t).

[0204]

[Example 20]

Synthesis of N,N-(3,7,11,15-tetramethylhexadecyl)-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate

5 N,N-(3,7,11,15-Tetramethylhexadecyl)-2,2,2-trifluoroethylamine hydrochloride (0.50 g, 0.72 mmol) obtained in Production Example 35, lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.66 g, 0.72 mmol), and methylcyclohexane (15 mL) were mixed,
10 and the mixture was stirred at room temperature for 3 hr. The mixture was washed with water, and the organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure at 45°C to give the title compound (0.96 g, 68%).
¹H NMR (CDCl₃) δ: 0.82-0.89 (30H, m), 1.13-1.57 (48H, m), 3.15-
15 3.25 (4H, m), 3.60 (2H, q);
¹⁹F NMR (CDCl₃) δ: -67.1 (3F, t), -133.9 (8F, d), -164.4 (4F, t), -167.5 (8F, t).

[0205]

Methylcyclohexane was added to the compound of Example 20
20 to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0206]

[Production Example 36]

Synthesis of N,N-(3,7,11-trimethyldodecyl)-2,2,2-trifluoroethylamine
25

3,7,11-Trimethyldodecanal (1.2 g, 5.3 mmol), 2,2,2-trifluoroethylamine (0.20 g, 2.0 mmol), and acetic acid (0.3 mL) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxymethylborohydride (1.5 g, 7.8 mmol) was added, and the
30 mixture was stirred at room temperature for 15 hr. The reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced
35 pressure. The residue was purified by silica gel column

chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (0.95 g, 91%).

^1H NMR (CDCl_3) δ : 0.83-0.88 (24H, m), 1.00-1.57 (34H, m), 2.58-2.60 (4H, m), 3.00 (2H, q);

5 ^{19}F NMR (CDCl_3) δ : -71.1 (3F, t).

[0207]

[Example 21]

Synthesis of N,N-(3,7,11-trimethyldodecyl)-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate

10 N,N-(3,7,11-Trimethyldodecyl)-2,2,2-trifluoroethylamine

(0.56 g, 1 mmol) obtained in Production Example 36 was

dissolved in methylcyclohexane (20 mL), 1.0 M hydrogen

chloride-diethyl ether solution (1 mL) was added, and lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex

15 (0.91 g, 1 mmol) was added. The reaction mixture was stirred at room temperature for 1 hr. The mixture was washed with water, and the organic layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure at 45°C to give the title compound (1.20 g, 74%).

20 ^1H NMR (CDCl_3) δ : 0.81-0.89 (24H, m), 1.03-1.51 (34H, m), 2.95-3.00 (4H, m), 3.41 (2H, q);

^{19}F NMR (CDCl_3) δ : -68.2 (3F, t), -133.9 (8F, d), -164.4 (4F, t), -167.5 (8F, t).

[0208]

25 Methylcyclohexane was added to the compound of Example 21 to prepare a 10 wt% methylcyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0209]

[Example 22]

30 Production of composition containing N,N-dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and N,N-dioctadecyl-2,2,2-trifluoroethylamine

N,N-Dioctadecyl-2,2,2-trifluoroethylamine (0.90 g, 1.49 mmol) obtained in Production Example 2 was dissolved in

35 chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate

tri-diethyl ether complex (0.91 g, 1.00 mmol) and 1.0 M hydrogen chloride-diethyl ether solution were added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was
5 concentrated under reduced pressure. The residue was dried under reduced pressure at 70°C to give the title composition (1.51 g).

^1H NMR (CDCl_3) δ : 0.86 (6H, t), 1.23-1.30 (60H, m), 1.68-1.76 (4H, m), 3.12-3.16 (4H, m), 3.61 (2H, q);

10 ^{19}F NMR (CDCl_3) δ : -65.3 (4.8F, t), -133.9 (8F, m), -163.8 (4F, t), -167.8 (8F, t).

[0210]

n-Hexane was added to the composition of Example 22 to prepare a 20 wt% n-hexane solution thereof, and the solution
15 was confirmed to be a homogeneous solution.

[0211]

Isohexane was added to the composition of Example 22 to prepare a 20 wt% isohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

20 [0212]

n-Heptane was added to the composition of Example 22 to prepare a 20 wt% n-heptane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0213]

25 ISOPAR E (registered trade mark) was added to the composition of Example 22 to prepare a 20 wt% ISOPAR E (registered trade mark) solution thereof, and the solution was confirmed to be a homogeneous solution.

[0214]

30 Cyclohexane was added to the composition of Example 22 to prepare a 20 wt% cyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0215]

Methylcyclohexane was added to the composition of Example
35 22 to prepare a 20 wt% methylcyclohexane solution thereof, and

the solution was confirmed to be a homogeneous solution.

[0216]

[Production Example 37]

Synthesis of N,N-ditetradecyl-2,2,2-trifluoroethylamine

5 1-Tetradecanal (4.0 g, 18.8 mmol), 2,2,2-trifluoroethylamine (0.90 g, 9.1 mmol), and acetic acid (0.3 mL) were dissolved in tetrahydrofuran (30 mL), sodium triacetoxyborohydride (4.0 g, 18.9 mmol) was added, and the mixture was stirred at room temperature for 15 hr. The
10 reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column
15 chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (4.0 g, 89%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.26-1.43 (48H, m), 2.55 (4H, t), 3.00 (2H, q);

^{19}F NMR (CDCl_3) δ : -71.3 (3F, t).

20 [0217]

[Production Example 38]

Synthesis of N,N-ditetradecyl-2,2,2-trifluoroethylamine hydrochloride

N,N-Ditetradecyl-2,2,2-trifluoroethylamine (1.50 g, 3.05
25 mmol) obtained in Production Example 37 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (1.45 g, 90%).

30 ^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.20-1.40 (44H, m), 1.96 (4H, br s), 3.15 (4H, br s), 3.78 (2H, q);

^{19}F NMR (CDCl_3) δ : -63.4 (3F, t).

[0218]

[Example 23]

35 Synthesis of N,N-ditetradecyl-2,2,2-trifluoroethylammonium

tetrakis(pentafluorophenyl)borate

N,N-Ditetradecyl-2,2,2-trifluoroethylamine (0.60 g, 1.14 mmol) obtained in Production Example 38 was dissolved in chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (1.04 g, 1.14 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 70°C to give the title compound (1.34 g, 100%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.36 (44H, m), 1.65-1.70 (4H, m), 3.14-3.18 (4H, m), 3.62 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.4 (3F, t), -134.0 (8F, m), -163.4 (4F, t), -167.6 (8F, t).

[0219]

Cyclohexane was added to Example 23 to prepare a 20 wt% cyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0220]

[Example 24]

Production of composition containing N,N-ditetradecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and didodecyl ether

N,N-Ditetradecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate (58 mg, 0.05 mmol) obtained in Example 23 and didodecyl ether (40 mg, 0.10 mmol) were added and mixed to give the title composition.

^1H NMR (CDCl_3) δ : 0.85-0.88 (18H, m), 1.24-1.35 (116H, m), 1.51-1.58 (8H, m), 1.64-1.70 (4H, m), 3.11-3.15 (4H, m), 3.38 (8H, t), 3.60 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.6 (3F, s), -134.0 (8F, s), -163.5 (4F, t), -167.6 (8F, t).

[0221]

n-Hexane was added to the composition of Example 24 to prepare a 20 wt% n-hexane solution thereof, and the solution

was confirmed to be a homogeneous solution.

[0222]

[Production Example 39]

Synthesis of N,N-dihexadecyl-2,2,2-trifluoroethylamine

5 1-Hexadecanal (4.0 g, 16.6 mmol), 2,2,2-trifluoroethylamine (0.84 g, 8.5 mmol), and acetic acid (0.5 mL) were dissolved in tetrahydrofuran (50 mL), sodium triacetoxyborohydride (4.0 g, 18.9 mmol) was added, and the mixture was stirred at room temperature for 15 hr. The
10 reaction mixture was basified by adding saturated aqueous sodium hydrogen carbonate solution, and extracted with n-hexane. The organic layer was washed with saturated brine, dried over anhydrous sodium sulfate, and concentrated under reduced pressure. The residue was purified by silica gel column
15 chromatography (n-hexane/ethyl acetate=100/0-95/5) to give the title compound (4.56 g, 98%).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.26-1.43 (56H, m), 2.56 (4H, t), 3.00 (2H, q);

^{19}F NMR (CDCl_3) δ : -71.3 (3F, t).

20 [0223]

[Production Example 40]

Synthesis of N,N-dihexadecyl-2,2,2-trifluoroethylamine hydrochloride

N,N-Dihexadecyl-2,2,2-trifluoroethylamine (1.50 g, 2.74
25 mmol) obtained in Production Example 39 was dissolved in n-hexane (30 mL), 1.0 M hydrogen chloride-diethyl ether solution (20 mL) was added, and the mixture was stirred for 1 hr. The reaction mixture was concentrated under reduced pressure to give the title compound (1.55 g, 97%).

30 ^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.40 (52H, m), 1.93 (4H, br s), 3.15 (4H, br s), 3.77 (2H, q);

^{19}F NMR (CDCl_3) δ : -63.4 (3F, t).

[0224]

[Example 25]

35 Synthesis of N,N-dihexadecyl-2,2,2-trifluoroethylammonium

tetrakis(pentafluorophenyl)borate

N,N-Dihexadecyl-2,2,2-trifluoroethylamine (0.60 g, 1.03 mmol) obtained in Production Example 40 was dissolved in chloroform (30 mL), lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (0.95 g, 1.05 mmol) was added, and the mixture was stirred at room temperature for 1 hr. Insoluble material was removed by filtration, and the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 70°C to give the title compound (1.51 g).

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.19-1.36 (54H, m), 1.65-1.70 (4H, m), 3.14-3.18 (4H, m), 3.62 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.4 (3F, t), -134.0 (8F, m), -163.4 (4F, t), -167.6 (8F, t).

[0225]

Cyclohexane was added to the composition obtained in Example 25 to prepare a 20 wt% cyclohexane solution thereof, and the solution was confirmed to be a homogeneous solution. [0226]

[Example 26]

Production of composition containing N,N-dihexadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and dihexadecyl ether

N,N-Dihexadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate (60 mg, 0.05 mmol) obtained in Example 25 and dihexadecyl ether (23 mg, 0.05 mmol) were added and mixed to give the title composition.

^1H NMR (CDCl_3) δ : 0.86-0.90 (12H, m), 1.20-1.36 (104H, m), 1.52-1.58 (4H, m), 1.63-1.70 (4H, m), 3.15-3.19 (4H, m), 3.39 (4H, t), 3.64 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.7 (3F, s), -134.1 (8F, s), -163.5 (4F, t), -167.6 (8F, t).

[0227]

n-Hexane was added to the composition of Example 26 to prepare a 20 wt% n-hexane solution thereof, and the solution

was confirmed to be a homogeneous solution.

[0228]

[Example 27]

Production of composition containing N,N-dioctadecyl-2,2,2-
5 trifluoroethylammonium tetrakis(pentafluorophenyl)borate and
dioctyl ether

N,N-Dioctadecyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate (64 mg, 0.05 mmol) obtained
in Example 1 and dioctyl ether (12 mg, 0.05 mmol) were added
10 and mixed to give the title composition.

^1H NMR (CDCl_3) δ : 0.88 (12H, t), 1.20-1.37 (80H, m), 1.52-1.58
(4H, m), 1.65-1.70 (4H, m), 3.14-3.19 (4H, m), 3.39 (4H, t),
3.63 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.6 (3F, br s), -134.0 (8F, br s), -163.4
15 (4F, t), -167.6 (8F, t).

[0229]

n-Hexane was added to the composition of Example 27 to
prepare a 20 wt% n-hexane solution thereof, and the solution
was confirmed to be a homogeneous solution.

20 [0230]

[Example 28]

Production of composition containing N,N-dioctadecyl-2,2,2-
trifluoroethylammonium tetrakis(pentafluorophenyl)borate and
didodecyl ether

25 N,N-Dioctadecyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate (64 mg, 0.05 mmol) obtained
in Example 1 and didodecyl ether (15 mg, 0.05 mmol) were added
and mixed to give the title composition.

^1H NMR (CDCl_3) δ : 0.86-0.90 (12H, m), 1.20-1.38 (96H, m), 1.53-
30 1.60 (4H, m), 1.65-1.72 (4H, m), 3.15-3.20 (4H, m), 3.39 (4H,
t), 3.65 (2H, q);

^{19}F NMR (CDCl_3) δ : -66.4 (3F, br s), -134.0 (8F, br s), -163.4
(4F, t), -167.5 (8F, m).

[0231]

35 n-Hexane was added to the composition of Example 28 to

prepare a 20 wt% n-hexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0232]

[Example 29]

5 Production of composition containing N,N-dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and dihexadecyl ether

N,N-Dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate (64 mg, 0.05 mmol) obtained
10 in Example 1 and dihexadecyl ether (23 mg, 0.05 mmol) were added and mixed to give the title composition.

^1H NMR (CDCl_3) δ : 0.86-0.98, (12H, m), 1.20-1.35 (112H, m), 1.52-1.75 (8H, m), 3.15-3.20 (4H, m), 3.39 (4H, t), 3.65 (2H, q);

15 ^{19}F NMR (CDCl_3) δ : -66.5 (3F, br s), -134.2 (8F, br s), -163.6 (4F, t), -167.7 (8F, m).

[0233]

n-Hexane was added to the composition of Example 29 to prepare a 20 wt% n-hexane solution thereof, and the solution
20 was confirmed to be a homogeneous solution.

[0234]

[Example 30]

Production of composition containing N,N-dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and
25 diphenyl ether

N,N-Dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate (64 mg, 0.05 mmol) obtained in Example 1 and diphenyl ether (8.5 mg, 0.05 mmol) were added and mixed to give the title composition.

30 ^1H NMR (CDCl_3) δ : 0.86-0.90, (6H, m), 1.20-1.40 (58H, m), 1.62-1.68 (4H, m), 3.12-3.16 (4H, m), 3.61 (4H, q), 6.99-7.03 (4H, m), 7.08-7.12 (2H, m), 7.31-7.36 (4H, m);

^{19}F NMR (CDCl_3) δ : -66.9 (3F, br s), -134.1 (8F, br s), -163.4 (4F, t), -167.6 (8F, m).

35 [0235]

n-Hexane was added to the composition of Example 30 to prepare a 20 wt% n-hexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0236]

5 [Example 31]

Production of composition containing N,N-dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and octadecyl phenyl ether

N,N-Dioctadecyl-2,2,2-trifluoroethylammonium
10 tetrakis(pentafluorophenyl)borate (64 mg, 0.05 mmol) obtained in Example 1 and octadecyl phenyl ether (18 mg, 0.05 mmol) were added and mixed to give the title composition.

^1H NMR (CDCl_3) δ : 0.84-0.98 (12H, m), 1.20-1.44 (85H, m), 1.62-1.81 (6H, m), 3.13-3.18 (4H, m), 3.61 (4H, q), 3.95 (2H, t),
15 6.88-6.94 (2H, m), 7.25-7.30 (3H, m);

^{19}F NMR (CDCl_3) δ : -66.8 (3F, br s), -134.1 (8F, br s), -163.4 (4F, t), -167.6 (8F, m).

[0237]

n-Hexane was added to the composition of Example 31 to
20 prepare a 20 wt% n-hexane solution thereof, and the solution was confirmed to be a homogeneous solution.

[0238]

[Example 32]

Production of composition containing N,N-dioctadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and
25 cyclopentyl methyl ether

N,N-Dioctadecyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate (64 mg, 0.05 mmol) obtained
in Example 1 and cyclopentyl methyl ether (10 mg, 0.10 mmol)
30 were added and mixed to give the title composition.

^1H NMR (CDCl_3) δ : 0.88 (6H, t), 1.20-1.35 (60H, m), 1.53-1.56 (4H, m), 1.61-1.76 (16H, m), 3.12-3.17 (4H, m), 3.28 (6H, s), 3.61 (2H, q), 3.79-3.83 (2H, m);

^{19}F NMR (CDCl_3) δ : -66.6 (3F, br s), -133.9 (8F, br s), -163.5
35 (4F, t), -167.6 (8F, m).

[0239]

n-Hexane was added to the composition of Example 32 to prepare a 20 wt% n-hexane solution thereof, and the solution was confirmed to be a homogeneous solution.

5 [0240]

[Example 33]

Production of composition containing N,N-dihexadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and ditetradecyl ether

10 N,N-Dihexadecyl-2,2,2-trifluoroethylamine hydrochloride (8.10 g, 13.9 mmol) obtained in Production Example 40 was dissolved in dichloromethane (80 mL), ditetradecyl ether (8.10 g, 13.9 mmol) and lithium tetrakis(pentafluorophenyl)borate tri-diethyl ether complex (14.2 g, 13.7 mmol) was added, and
15 the mixture was stirred at room temperature for 1 hr. Water was added to the reaction mixture, and the mixture was stirred at room temperature for 1 hr. The aqueous layer was separated, and the organic layer was washed with water. The organic layer was dried over anhydrous sodium sulfate, and then filtered, and
20 the filtrate was concentrated under reduced pressure. The residue was dried under reduced pressure at 80°C to give the title composition (22.5 g).

¹H NMR (CDCl₃) δ: 0.87-0.89 (12H, m), 1.20-1.80 (104H, m), 3.21-3.24 (4H, m), 3.38-3.41 (4H, m), 3.69 (2H, q);

25 ¹⁹F NMR (CDCl₃) δ: -66.7 (3F, br s), -134.0 (8F, m), -163.4 (4F, t), -167.5 (8F, t).

[0241]

n-Hexane was added to the composition obtained in Example 33 to prepare a 20 wt% n-hexane solution thereof, and the
30 solution was confirmed to be a homogeneous solution.

[0242]

[Example 34]

Production of composition containing N,N-dihexadecyl-2,2,2-trifluoroethylammonium tetrakis(pentafluorophenyl)borate and
35 didodecyl ether

N,N-Dihexadecyl-2,2,2-trifluoroethylammonium
tetrakis(pentafluorophenyl)borate (439.2 mg, 0.36 mmol)
obtained in Example 25 was mixed with didodecyl ether (254 mg,
0.72 mmol), and n-hexane (1.756 g) was added to prepare a
5 homogeneous hexane solution of the title composition. The
solution was confirmed to be a homogeneous solution. This
solution was concentrated under reduced pressure and analyzed
by NMR.
¹H NMR (CDCl₃) δ: 0.87-0.91 (12H, m), 1.20-1.42 (100H, m), 1.53-
10 1.69 (12H, m), 3.20-3.24 (4H, m), 3.40 (8H, t), 3.67 (2H, q);
¹⁹F NMR (CDCl₃) δ: -66.9 (3F, t), -134.0 (8F, m), -163.8 (4F, t),
-167.4 (8F, t).

[0243]

[Comparative Example 1]

15 Attempts were made to prepare a 10 wt% methylcyclohexane
solution or 10 wt% n-hexane solution of N,N-dimethylanilinium
tetrakis(pentafluorophenyl)borate, but a homogeneous solution
was not obtained. In the below-mentioned Experimental Example,
N,N-dimethylanilinium tetrakis(pentafluorophenyl)borate was
20 used as a co-catalyst in Comparative Example 1.

[0244]

[Comparative Example 2]

Attempts were made to prepare a 10 wt% n-hexane solution
of N,N-dioctadecylmethylanmonium
25 tetrakis(pentafluorophenyl)borate, but a homogeneous solution
was not obtained. In the below-mentioned Experimental Example,
N,N-dioctadecylmethylanmonium tetrakis(pentafluorophenyl)borate
was used as a co-catalyst in Comparative Example 2.

[0245]

30 [Experimental Example] (Evaluation of polymerization
performance)

A general polymerization method using the compound or
composition of the present invention as a co-catalyst is shown
below.

35 [0246]

Into 100 mL autoclave in a glove box were added 1-octene, triisobutylaluminum (TIBA, 0.55 M hexane solution), and a solvent (methylcyclohexane (MCH) or toluene) to give a comonomer solution. Dimethylsilylene(tert-butylamide)-(tetramethylcyclopentadienyl)-titanium (IV)-dichloride (CGC, polymerization catalyst), triisobutylaluminum (0.55 M hexane solution), and a solvent were added to prepare a catalyst solution at a predetermined concentration, and the solution was transferred to a Schlenk flask. The co-catalyst was dissolved in a solvent, and a co-catalyst solution at a predetermined concentration was prepared and transferred to the Schlenk flask. The comonomer solution, the catalyst solution, and the co-catalyst solution were mixed, and adjusted such that the total amount of the solvent and the total amount of triisobutylaluminum would be constant during the reaction. The inside of the autoclave was purged with ethylene, the catalyst solution and the co-catalyst solution were successively added to the autoclave, and the ethylene pressure was immediately adjusted to a predetermined pressure, and the mixture was stirred at a predetermined temperature (25°C or 100°C) for a predetermined time. The reaction mixture was ice-cooled, the ethylene gas was removed, the mixture was poured into methanol (100 mL) containing hydrochloric acid (3 mL), and the mixture was stirred at room temperature for 30 min. The precipitate was collected by filtration and dried under reduced pressure at 60°C to give an ethylene-octene copolymer.

[0247]

[Measurement of melting point]

Measurement by the differential scanning calorimetry method (DSC) was performed using DSC6220 instrument (Seiko Instruments Inc.). A sample (polymer) was heated at a rate of 10°C/min from 40°C to 150°C, and the melting point was measured.

[0248]

The results of the polymerization reaction at 25°C and 100°C are shown in Table 1 and Table 2, respectively.

[0249]

[Table 1]

co-catalyst	catalytic amount (μmol)	solvent	time (min)	yield (g)	activity (kg/mol of Ti·h)	melting point (°C)
Comparative Example 2	0.1	MCH	6	0.033	3300	77.6
Example 1	0.05	MCH	1.5	0.102	81600	70.5
Example 2	0.05	MCH	1.5	0.134	107200	63.3, 68.0
Example 4	0.05	MCH	1.5	0.253	202400	75.2
Example 7	0.5	MCH	1.5	1.12	89960	80.3
Example 11	0.1	MCH	1.5	0.465	185840	89.1
Example 22	0.1	MCH	1.5	0.378	151200	71.5
Comparative Example 1	0.1	toluene	1.5	0.85	340000	79.3
Example 1	0.05	toluene	1.5	1.15	920000	84.0
Example 4	0.05	toluene	1.5	1.23	984000	70.8

reaction conditions; catalyst: CGC, catalyst:co-catalyst=1:1,
TIBA (total amount 3000 μmol), total amount of solvent (40 mL),
5 1-octene (1 mL), ethylene pressure (8 atm), 25°C.

[0250]

[Table 2]

co-catalyst	catalytic amount (μmol)	solvent	time (min)	yield (g)	activity (kg/mol of Ti·h)	melting point (°C)
Comparative Example 2	0.02	MCH	3	0.026	26000	90.0
Example 1	0.02	MCH	3	0.460	460000	92.3
Example 2	0.02	MCH	3	0.150	145300	93.2
Example 22	0.02	MCH	3	0.330	333300	91.3

reaction conditions; catalyst: CGC, catalyst:co-catalyst=1:1,
10 TIBA (total amount 100 μmol), total amount of solvent (40 mL),
1-octene (1 mL), ethylene pressure (8 atm), 100°C.

[0251]

According to Table 1 and Table 2, it was confirmed that
15 the fluorine-containing ammonium borate compounds of Examples 1,
2, 4, 7, 11, and 22 showed higher polymerization activity than

the compounds of Comparative Example 1 and Comparative Example 2 not containing a fluorine atom, regardless of the polymerization temperature and the kind of the solvent used. Furthermore, as shown in Table 1, the compositions (or
5 complexes) of the present invention (Examples 1, 2, 4, and 22) afforded polymers having a lower melting point than Comparative Example depending on the polymerization conditions, and the uptake amount of comonomer is considered to have increased.

[Industrial Applicability]

10 [0252]

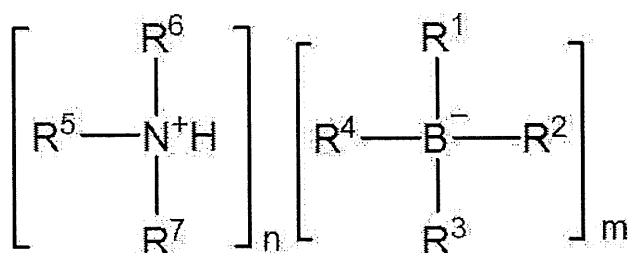
The compound or composition of the present invention shows high metal complex catalyst activation ability in the polymerization reactions of olefin, diene, and acetylene, and is useful as a co-catalyst. Also, according to the present
15 invention, an industrial production method of the compound or composition of the present invention can also be provided.

[0253]

This application is based on a patent application No. 2020-144177 filed in Japan (filing date: August 28, 2020), a
20 patent application No. 2020-196704 filed in Japan (filing date: November 27, 2020), and a patent application No. 2021-037078 filed in Japan (filing date: March 9, 2021), the contents of which are incorporated in full herein.

[CLAIMS]

1. A compound represented by the following formula (1):



(1)

wherein

R^1 , R^2 , R^3 , and R^4 are each independently a C_{6-14} aryl group substituted by one or more fluorine atoms or one or more fluoro C_{1-4} alkyl groups,

R^5 is a C_{1-30} alkyl group substituted by one or more substituents selected from the group consisting of a C_{6-14} aryl group substituted by one or more fluorine atoms, and a fluorine atom,

R^6 and R^7 are each independently an optionally substituted C_{1-30} alkyl group, an optionally substituted C_{3-15} cycloalkyl group, or an optionally substituted C_{6-14} aryl group, or

R^6 and R^7 are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group,

n is 1, and

m is 1 or 2.

2. The compound according to claim 1, wherein R^1 , R^2 , R^3 , and R^4 are each independently a phenyl group, a 1-naphthyl group, a 2-naphthyl group, a 2-biphenylyl group, a 3-biphenylyl group, a 4-biphenylyl group, a 1-anthryl group, a 2-anthryl group, a 9-anthryl group, a 9-phenanthryl group, or a 3-phenanthryl group, each of which is substituted by one or more fluorine atoms or one or more fluoro C_{1-4} alkyl groups.

3. The compound according to claim 1, wherein all of R¹, R², R³, and R⁴ are pentafluorophenyl groups, 2,2',3,3',4',5,5',6,6'-nonafluoro-4-(1,1'-biphenyl) groups, 2,3,4,5,6,7,8-heptafluoro-1-naphthyl groups, or 1,3,4,5,6,7,8-heptafluoro-2-naphthyl groups.

4. The compound according to any one of claims 1 to 3, wherein R⁵ is a C₁₋₆ alkyl group substituted by a phenyl group or a naphthyl group, each of which is substituted by one or more fluorine atoms, or a fluoro C₁₋₆ alkyl group.

5. The compound according to any one of claims 1 to 4, wherein R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s),
- (2) a halogen atom, and
- (3) a C₁₋₃₀ alkoxy group; or

a C₃₋₈ cycloalkyl group optionally substituted by substituent(s) selected from the group consisting of

- (1) a halogen atom,
- (2) a C₁₋₃₀ alkyl group,
- (3) a C₁₋₃₀ alkoxy group,
- (4) a halo C₁₋₃₀ alkyl group, and
- (5) a halo C₁₋₃₀ alkoxy group.

6. The compound according to any one of claims 1 to 4, wherein R⁶ and R⁷ are bonded to each other to form, together with the nitrogen atom bonded thereto, an optionally substituted cyclic group derived from a 3- to 8-membered monocyclic nitrogen-containing non-aromatic heterocyclic group.

7. The compound according to any one of claims 1 to 4, wherein R⁵ is a fluoro C₁₋₆ alkyl group, R⁶ and R⁷ are each independently a C₁₋₃₀ alkyl group optionally

substituted by substituent(s) selected from the group consisting of

- (1) a C₆₋₁₄ aryl group optionally substituted by halogen atom(s),
- (2) a halogen atom, and
- 5 (3) a C₁₋₃₀ alkoxy group.

8. The compound according to any one of claims 1 to 7, wherein n and m are each 1.

10 9. The compound according to any one of claims 1 to 8, wherein a total carbon number of R⁵, R⁶, and R⁷ is not less than 25.

10. A composition comprising the compound according to any one of claims 1 to 9, and a compound of the following formula (2):

15



wherein

R and R' are each independently an optionally substituted C₁₋₃₀ alkyl group, an optionally substituted C₃₋₁₅ cycloalkyl group, or an optionally substituted C₆₋₁₄ aryl group.

25

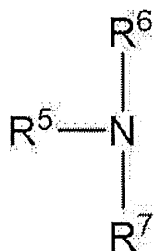
11. The composition according to claim 10, wherein R and R' are each independently an optionally substituted C₁₋₃₀ alkyl group.

12. The composition according to claim 10 or 11, wherein a content of the compound represented by the formula (2) with respect to 1 mol of the compound represented by the formula (1) is 0.01 to 10 mol.

30

13. The composition according to claim 10 or 11, wherein a content of the compound represented by the formula (2) with respect to 1 mol of the compound represented by the formula (1) is 0.1 to 3 mol.

14. A composition comprising the compound according to any one of claims 1 to 9, and a compound of the following formula (3):



5 (3)

wherein R^5 , R^6 , and R^7 are as defined above
(in the composition, the compound represented by the formula (3) is an amine compound deprotonated from the cation constituting the formula (1) according to claim 1).

10

15. The composition according to claim 14, wherein a content of the compound represented by the formula (3) with respect to 1 mol of the compound represented by the formula (1) is 0.01 to 10 mol.

15

16. The composition according to claim 14, wherein a content of the compound represented by the formula (3) with respect to 1 mol of the compound represented by the formula (1) is 0.5 to 3 mol.

20

17. The composition according to any one of claims 14 to 16, further comprising a compound represented by the following formula (2):



wherein

R and R' are each independently an optionally substituted C_{1-30}

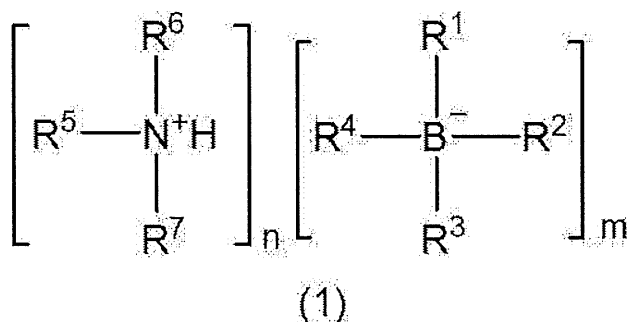
alkyl group, an optionally substituted C₃₋₁₅ cycloalkyl group,
or an optionally substituted C₆₋₁₄ aryl group.

18. A co-catalyst for polymerizing at least one monomer
5 selected from the group consisting of olefin, diene, and
acetylene, comprising the compound according to any one of
claims 1 to 9, or the composition according to any one of
claims 10 to 17.

10 19. A method for producing a polymer, comprising polymerizing
at least one kind of monomer selected from the group consisting
of olefin, diene and acetylene by using the compound according
to any one of claims 1 to 9 or the composition according to any
one of claims 10 to 17 as a co-catalyst.

15

20. A method for producing a compound represented by the
following formula (1):



20 wherein

R¹, R², R³, and R⁴ are each independently a C₆₋₁₄ aryl group
substituted by one or more fluorine atoms or one or more fluoro
C₁₋₄ alkyl groups,

R⁵ is a C₁₋₃₀ alkyl group substituted by one or more

25 substituents selected from the group consisting of

(1) a C₆₋₁₄ aryl group substituted by one or more fluorine atoms
and

(2) a fluorine atom,

R⁶ and R⁷ are each independently an optionally substituted C₁₋₃₀

30 alkyl group, an optionally substituted C₃₋₁₅ cycloalkyl group,

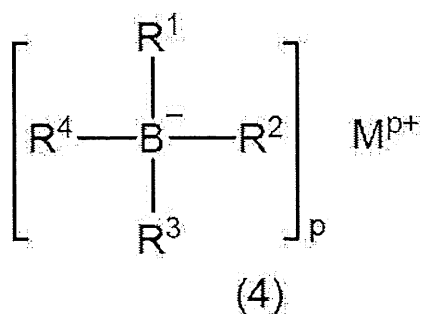
or an optionally substituted C₆₋₁₄ aryl group, or
 R⁶ and R⁷ are bonded to each other to form, together with the
 nitrogen atom bonded thereto, an optionally substituted cyclic
 group,

5 n is 1, and

m is 1 or 2,

the method comprising a step of reacting, in the presence of a
 protic acid, a compound represented by the following formula

(4):



10

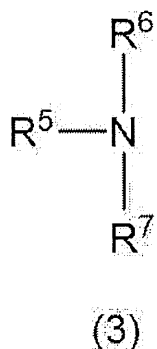
wherein

R¹, R², R³, and R⁴ are as defined above,

M^{p+} is an alkali metal ion or an alkaline earth metal ion, and

p is 1 or 2,

15 with a compound represented by the following formula (3):



wherein R⁵, R⁶ and R⁷ are as defined above.