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(54) Title: METHOD FOR PREPARING IODOSILANES

(57) Abstract: Provided is methodology for the preparation of highly-desired iodosilanes such as H_2SiI_2 and $HSiI_3$, via a reaction of alkylaminosilanes with certain substituted acid iodides. In one embodiment, bis(diethylamino)silane is reacted with benzoyl iodide to provide diiodosilane.



METHOD FOR PREPARING IODOSILANES

Technical Field

[0001] This invention belongs to the field of silicon chemistry. In particular, it relates to methodology for preparing di- and tri-iodosilanes.

Background

[0002] Halosilanes are useful as precursors in the manufacturing of microelectronic devices; in particular, halosilanes such as H_2SiI_2 and HSiI_3 are useful as precursor compounds for the deposition of silicon-containing films used in the manufacture of microelectronic devices. Current solution based synthetic methodology describes the synthesis of H_2SiI_2 and other select iodosilanes from (i) aryl silanes (Keinan *et al.* J. Org. Chem., Vol. 52, No. 22, 1987, pp. 4846-4851; Kerrigan *et al.* U.S. Patent No. 10,106,425 or (ii) halosilanes such as SiH_2Cl_2 (U.S. Patent No. 10,384,944).

[0003] Keinan *et al.* describe a synthetic method towards SiH_2I_2 formation that employs stoichiometric treatment of Phenyl- SiH_3 , an arylsilane, with iodine in the presence of a catalyst such as ethyl acetate. The reaction by-products are the aromatic function from the arylsilane, liberated as benzene, and a complicated by-product mixture resulting from ethyl acetate decomposition. Tedious separation of the reaction by-products from the desired SiH_2I_2 complicates the process. In addition, arylsilane-based methods for preparing halosilanes typically generate product contaminated with iodine and/or hydrogen iodide, which are deleterious to the desired iodosilane product, so often antimony, silver, or copper are utilized to stabilize the iodosilane product.

[0004] U.S. Patent No. 10,106,425 teaches the use of an arylsilane, $(\text{CH}_3\text{C}_6\text{H}_4)\text{SiH}_3$, as reactant. The process as disclosed generates toluene as a by-product and is thus claimed as a less hazardous alternative to the Keinan method which generates benzene from Phenyl- SiH_3 . U.S. Patent No. 10,384,944 describes a halide exchange between, for example, LiI and SiH_2Cl_2 , thereby generating LiCl and SiH_2I_2 in a halogen exchange reaction.

Summary

[0005] In summary, the invention provides methodology for preparing highly desired iodosilanes such as H_2SiI_2 and HSiI_3 , via treatment of certain dialkylamido-silanes with an acyl iodide. In one example, diiodosilane can be prepared from the reaction of bis(diethylamino)silane with benzoyl iodide. The reaction surprisingly results in the substitution of the alkylamino group on the aminosilane with an iodo group. This transformation readily occurs at room temperature and atmospheric pressure and can be conducted neat as well as in the presence of aprotic solvents.

Detailed Description

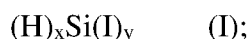
[0006] As used in this specification and the appended claims, the singular forms “a”, “an”, and “the” include plural referents unless the content clearly dictates otherwise. As used in this specification and the appended claims, the term “or” is generally employed in its sense including “and/or” unless the content clearly dictates otherwise.

[0007] The term “about” generally refers to a range of numbers that is considered equivalent to the recited value (e.g., having the same function or result). In many instances, the term “about” may include numbers that are rounded to the nearest significant figure.

[0008] Numerical ranges expressed using endpoints include all numbers subsumed within that range (e.g. 1 to 5 includes 1, 1.5, 2, 2.75, 3, 3.80, 4 and 5).

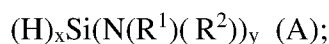
[0009] In a first aspect, the disclosure provides a method for preparing a compound having the

formula(I):



wherein x is 1 or 2, y is 2 or 3, and the sum of x plus y is 4, the method comprising:

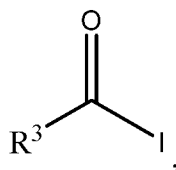
contacting a compound of the formula (A):



wherein (i) each R^1 and each R^2 is independently chosen from a C_1 - C_6 alkyl group, a C_3 - C_8 cycloaliphatic group, or hydrogen, or (ii) R^1 and R^2 are taken together with the nitrogen atom to which they are bonded to form a C_4 - C_7

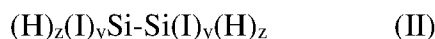
nitrogen-containing saturated or aromatic ring, provided that no more than one of R^1 and R^2 is hydrogen;

with a compound of the formula:



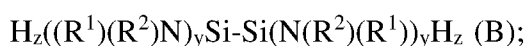
wherein R^3 is chosen from C_1 - C_6 alkyl, C_1 - C_6 substituted alkyl, benzyl, substituted benzyl, biphenyl, naphthyl, phenyl, or substituted phenyl, to produce a compound of formula (I).

[0010] In a second aspect, the disclosure provides a method for preparing a compound having the formula (II):



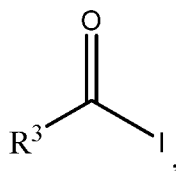
wherein y is 2 or 3, z is 0 or 1, and the sum of y and z is 3, the method comprising:

contacting a compound of the formula (B):



wherein (i) each R^1 and each R^2 is independently chosen from a C_1 - C_6 alkyl group, a C_3 - C_8 cycloaliphatic group, or hydrogen, or (ii) R^1 and R^2 are taken together with the nitrogen atom to which they are bonded to form a C_4 - C_7 nitrogen-containing saturated or aromatic ring, provided that no more than one of R^1 and R^2 is hydrogen;

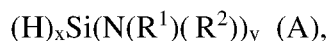
with a compound of the formula:



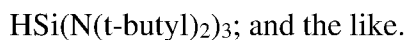
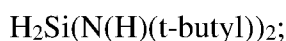
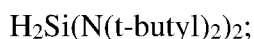
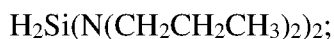
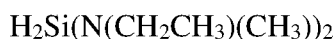
wherein R^3 is chosen from C_1 - C_6 alkyl, C_1 - C_6 substituted alkyl, benzyl, substituted benzyl, biphenyl, naphthyl, phenyl, or substituted phenyl, to produce a compound of formula (II).

[0011] In the above process, it will be appreciated that the product of formula (I) is produced when the reactant of formula (A) is utilized and the product of formula (II) is produced when the reactant of formula (B) is utilized. In one embodiment, the compound of formula (I) is H_2SiI_2 . In another embodiment, the compound of formula (I) is $HSiI_3$.

[0012] In one embodiment, the compound of the formula



is chosen from compounds such as



[0013] As used herein, the term “substituted phenyl” denotes a phenyl group substituted one to five times with a group chosen from C_1 - C_6 alkyl, C_1 - C_6 alkylamino, di-(C_1 - C_6 alkyl)amino, and halo. In one embodiment, halo is fluoro.

[0014] As used herein, the term “substituted C₁-C₆ alkyl” denotes a C₁-C₆ alkyl group substituted one to five times with a group chosen from C₁-C₆ alkylamino, di-(C₁-C₆ alkyl)amino, and halo.

[0015] As used herein, the term “substituted benzyl” denotes a benzyl group having one to five substituents on the aromatic ring chosen from C₁-C₆ alkylamino, di-(C₁-C₆ alkyl)amino, and halo.

[0016] The methodology of the invention can be practiced neat or in the presence of an aprotic solvent which is non-reactive with the starting materials or iodosilane products. Such solvents include aliphatic, aromatic, and halogenated hydrocarbon solvents which are devoid of moieties and functional groups such as oxygen, esters, carboxy groups, and ketones. Examples include benzene, toluene, hexane, cyclohexane, pentane, dichloromethane, 1,2-dichloroethane, tetralin, decalin, mesitylene, dibromoethane, tetrachloroethylene, or chlorobenzene, and the like.

[0017] The reaction generally proceeds at room temperature and atmospheric pressure.

[0018] The method for preparing the precursor compounds of the invention can be conducted in standard batch or continuous mode reactors. One of ordinary skill in the art would recognize the scale and type of reactors which could be utilized in the context of the reagents and products so produced.

[0019] Compounds of the formulae (I) and (II) are useful as precursors in the formation of silicon-containing films on the surface of a microelectronic device by methods such as atomic layer deposition (see for example, U.S. Patent Nos. 10,580,645 and 10,424,477, incorporated herein by reference). Also, by way of example, compounds of formulae (I) and (II) can be introduced into a deposition chamber for the purposes of thermal CVD or ALD, or for the purposes of performing plasma-enhanced ALD or CVD. In these cases, a co-reactant gas can be introduced to deposit an SiO₂ film, via oxidation in an oxidizing environment with O₂, O₃, N₂O, or mixtures thereof. Similarly, compounds of formulae (I) and (II) can be introduced into a deposition chamber for the purposes of thermal CVD or ALD, or for the purposes of performing plasma-enhanced ALD or CVD. In these cases, a co-reactant gas can be introduced to deposit a silicon nitride film, via nitridation with N₂, NH₃, hydrazine or alkylhydrazine containing mixtures. The deposited films serve as dielectric layers within the microelectronic device.

Examples

Example 1: Diiodosilane

[0020] A 25.0 g (143 mMol) sample of bis(diethylamino)silane was combined with 4 equivalents, 133.0 g (574 mMol) of benzoyl iodide and 500 mL of hexanes. The reaction was allowed to proceed for 2 hours, after which time, the desired diiodosilane product was detected by ^1H NMR and all starting material was consumed. In the case of addition of benzoyl iodide to bis(diethylamino)silane, the observed yield was 7% (determined from concentration in hexanes). In the case of addition of bis(diethylamino)silane to benzoyl iodide, the observed yield was 3.5% (determined from concentration in hexanes). NMR samples were spiked to confirm with diiodosilane to confirm the identity of the signal.

^1H NMR (400 MHz, C_6D_6): δ 3.61 ($^1J_{\text{Si-H}} = 141.5$ Hz, H-SiI_2 , 2H).

Example 2: Diiodosilane

[0021] A 20.0 g (115 mMol) sample of bis(diethylamino)silane was combined with 4 equivalents, 78.0 g (459 mMol) of acetyl iodide and 400 mL of hexanes. The reaction was allowed to proceed for 10 minutes, after which time, trace amounts of the desired diiodosilane product was detected by ^1H NMR and all starting material was consumed. Upon attempted isolation, decomposition of the product occurred. In the case of addition of acetyl iodide to bis(diethylamino)silane, the observed isolated yield was 0%.

^1H NMR (400 MHz, C_6D_6): δ 3.61 ($^1J_{\text{Si-H}} = 141.5$ Hz, H-SiI_2 , 2H).

Example 3: Diiodosilane

[0022] A 0.68 g (3.9 mMol) sample of bis(diethylamino)silane was combined with 4 equivalents, 4.69 g (15.6 mMol) of 2-fluorobenzoyl iodide and 10 mL of hexanes. The reaction was allowed to proceed for 10 minutes, after which time, 43% conversion of the desired diiodosilane product was detected by ^1H NMR and all starting material was consumed. After an additional, 20 hr of stirring, the case of addition of 2-fluorobenzoyl iodide to bis(diethylamino)silane, the observed yield was 20%.

^1H NMR (400 MHz, C_6D_6): δ 3.61 ($^1J_{\text{Si-H}} = 141.5$ Hz, H-SiI_2 , 2H).

Example 4: Diiodosilane

[0023] A 0.27 g (1.6 mMol) sample of bis(diethylamino)silane was combined with 4 equivalents, 2.3 g (6.2 mMol) of 3,5-difluorobenzoyl iodide and 5 mL of hexanes. The reaction was allowed to proceed for 10 minutes, after which time, 81% conversion of the desired diiodosilane product was detected by ^1H NMR and all starting material was consumed. After an additional, 20 hr of stirring, the case of addition of 3,5-difluorobenzoyl iodide to bis(diethylamino)silane, the observed yield was 20%.

^1H NMR (400 MHz, C_6D_6): δ 3.61 ($^1J_{\text{Si-H}} = 141.5$ Hz, H-SiI_2 , 2H).

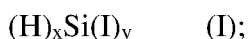
Example 5 Diiodosilane

[0024] A 3.3 g (18.9 mMol) sample of bis(diethylamino)silane was combined with 4 equivalents, 1.0 g (4.7 mMol) of 2,2-dimethylpropanoyl iodide and 3 mL of hexanes. The reaction was allowed to proceed for 10 minutes, after which time, 20% of the desired diiodosilane product was detected by ^1H NMR with 72% of the starting material remaining. After an additional, 20 hr of stirring, the case of addition of 2,2-dimethylpropanoyl iodide to bis(diethylamino)silane, the observed yield was 0.1%.

^1H NMR (400 MHz, C_6D_6): δ 3.61 ($^1J_{\text{Si-H}} = 141.5$ Hz, H-SiI_2 , 2H).

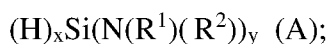
Aspects

[0025] In a first aspect, the disclosure provides a method for preparing a compound having the formula (I):



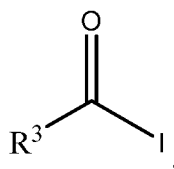
wherein x is 1 or 2, y is 2 or 3, and the sum of x plus y is 4, the method comprising:

contacting a compound of the formula (A):



wherein (i) each R^1 and each R^2 is independently chosen from a C_1 - C_6 alkyl group, a C_3 - C_8 cycloaliphatic group, or hydrogen, or (ii) R^1 and R^2 are taken together with the nitrogen atom to which they are bonded to form a C_4 - C_7 nitrogen-containing saturated or aromatic ring, provided that no more than one of R^1 and R^2 is hydrogen;

with a compound of the formula:



wherein R^3 is chosen from C_1 - C_6 alkyl, C_1 - C_6 substituted alkyl, benzyl, substituted benzyl, biphenyl, naphthyl, phenyl, or substituted phenyl, to produce a compound of formula (I).

[0026] In a second aspect, the disclosure provides the method of the first aspect, wherein the compound of formula (I) is H_2SiI_2 .

[0027] In a third aspect, the disclosure provides the method of the first aspect, wherein the compound of formula (I) is $HSiI_3$.

[0028] In a fourth aspect, the disclosure provides the method of the first or second aspect, wherein R^1 and R^2 are chosen from methyl, ethyl, propyl, sec-butyl, and t-butyl.

[0029] In a fifth aspect, the disclosure provides the method of any one of the first through fourth aspects, wherein at least one of R^1 and R^2 is t-butyl.

[0030] In a sixth aspect, the disclosure provides the method of the first, second, or third aspects, wherein at least one of R^1 and R^2 is cyclohexyl.

[0031] In a seventh aspect, the disclosure provides the method of the first, second, or third aspects, wherein R^1 and R^2 are taken together with the nitrogen atom to which they are attached, to form a pyrrole, pyrrolidine, or piperidine ring.

[0032] In an eighth aspect, the disclosure provides the method of any one of the first through the seventh aspects, wherein R^3 is C_1 - C_6 alkyl.

[0033] In a ninth aspect, the disclosure provides the method of any one of the first through seventh aspects, wherein R^3 is phenyl.

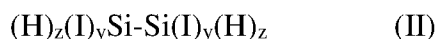
[0034] In a tenth aspect, the disclosure provides the method of any one of the first through seventh aspects, wherein R^3 is substituted phenyl.

[0035] In an eleventh aspect, the disclosure provides the method of any one of the first through seventh, or tenth aspects, wherein R^3 is 4-fluorophenyl.

[0036] In a twelfth aspect, the disclosure provides the method of any one of the first through seventh or tenth aspects, wherein R^3 is 3,5-difluorophenyl.

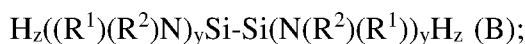
[0037] In a thirteenth aspect, the disclosure provides the method of the first, second, or third aspects, wherein at least one of R^1 and R^2 is ethyl and R^3 is methyl or phenyl.

[0038] In a fourteenth aspect, the disclosure provides a method for preparing a compound having the formula (II):

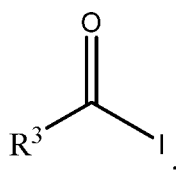


wherein y is 2 or 3, z is 0 or 1, and the sum of y and z is 3, the method comprising:

contacting a compound of the formula (B):



wherein (i) each R^1 and each R^2 is independently chosen from a C_1 - C_6 alkyl group, a C_3 - C_8 cycloaliphatic group, or hydrogen, or (ii) R^1 and R^2 are taken together with the nitrogen atom to which they are bonded to form a C_4 - C_7 nitrogen-containing saturated or aromatic ring, provided that no more than one of R^1 and R^2 is hydrogen;
with a compound of the formula:



wherein R^3 is chosen from C_1 - C_6 alkyl, C_1 - C_6 substituted alkyl, benzyl, substituted benzyl, biphenyl, naphthyl, phenyl, or substituted phenyl, to produce a compound of formula (II).

[0039] In a fifteenth aspect, the disclosure provides the method of the fourteenth aspect, wherein R^1 and R^2 are chosen from methyl, ethyl, propyl, sec-butyl, and t-butyl.

[0040] In a sixteenth aspect, the disclosure provides the method of the fourteenth aspect, wherein at least one of R^1 or R^2 is t-butyl.

[0041] In a seventeenth aspect, the disclosure provides the method of the fourteenth aspect, wherein at least one of R^1 or R^2 is cyclohexyl.

[0042] In an eighteenth aspect, the disclosure provides the method of the fourteenth aspect, wherein R^1 and R^2 are taken together with the nitrogen atom to which they are attached, to form a pyrrole, pyrrolidine, or piperidine ring.

[0043] In a nineteenth aspect, the disclosure provides the method of any one of the fourteenth through eighteenth aspects, wherein R^3 is C_1 - C_6 alkyl.

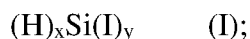
[0044] In a twentieth aspect, the disclosure provides the method of any one of the fourteenth through eighteenth aspects, wherein R^3 is phenyl.

[0045] In a twenty-first aspect, the disclosure provides the method of the fourteenth aspect, wherein at least one of R^1 and R^2 is ethyl and R^3 is methyl or phenyl.

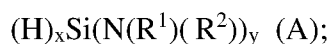
[0046] Having thus described several illustrative embodiments of the present disclosure, those of skill in the art will readily appreciate that yet other embodiments may be made and used within the scope of the attached claims. Numerous advantages of the disclosure covered by this document have been set forth in the foregoing description. It will be understood, however, that this disclosure is, in many respects, only illustrative. Changes may be made in details without exceeding the scope of the disclosure. The disclosure's scope is, of course, defined in the language in which the appended claims are expressed.

What is claimed is:

1. A method for preparing a compound having the formula (I):

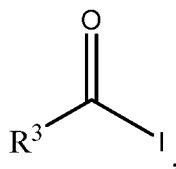


wherein x is 1 or 2, y is 2 or 3, and the sum of x plus y is 4, the method comprising:
contacting a compound of the formula (A):



wherein (i) each R^1 and each R^2 is independently chosen from a C_1 - C_6 alkyl group, a C_3 - C_8 cycloaliphatic group, or hydrogen, or (ii) R^1 and R^2 are taken together with the nitrogen atom to which they are bonded to form a C_4 - C_7 nitrogen-containing saturated or aromatic ring, provided that no more than one of R^1 and R^2 is hydrogen;

with a compound of the formula:

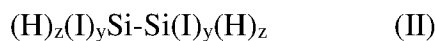


wherein R^3 is chosen from C_1 - C_6 alkyl, C_1 - C_6 substituted alkyl, benzyl, substituted benzyl, biphenyl, naphthyl, phenyl, or substituted phenyl,

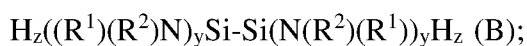
to produce a compound of formula (I).

2. The method of claim 1, wherein the compound of formula (I) is H_2SiI_2 .
3. The method of claim 1, wherein the compound of formula (I) is $HSiI_3$.

4. The method of claim 1, wherein R¹ and R² are chosen from methyl, ethyl, propyl, sec-butyl, and t-butyl.
5. The method of claim 1, wherein at least one of R¹ and R² is t-butyl.
6. The method of claim 1, wherein at least one of R¹ and R² is cyclohexyl.
7. The method of claim 1, wherein R¹ and R² are taken together with the nitrogen atom to which they are attached, to form a pyrrole, pyrrolidine, or piperidine ring.
8. The method of claim 1, wherein R³ is C₁-C₆ alkyl.
9. The method of claim 1, wherein R³ is phenyl.
10. The method of claim 1, wherein R³ is substituted phenyl.
11. The method of claim 10, wherein R³ is 4-fluorophenyl.
12. The method of claim 10, wherein R³ is 3,5-difluorophenyl.
13. The method of claim 1, wherein at least one of R¹ and R² is ethyl and R³ is methyl or phenyl.
14. A method for preparing a compound having the formula (II):

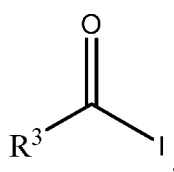


wherein y is 2 or 3, z is 0 or 1, and the sum of y and z is 3, the method comprising:
contacting a compound of the formula (B):



wherein (i) each R^1 and each R^2 is independently chosen from a C_1 - C_6 alkyl group, a C_3 - C_8 cycloaliphatic group, or hydrogen, or (ii) R^1 and R^2 are taken together with the nitrogen atom to which they are bonded to form a C_4 - C_7 nitrogen-containing saturated or aromatic ring, provided that no more than one of R^1 and R^2 is hydrogen;

with a compound of the formula:



wherein R^3 is chosen from C_1 - C_6 alkyl, C_1 - C_6 substituted alkyl, benzyl, substituted benzyl, biphenyl, naphthyl, phenyl, or substituted phenyl,

to produce a compound of formula (II).

15. The method of claim 14, wherein R^1 and R^2 are chosen from methyl, ethyl, propyl, sec-butyl, and t-butyl.
16. The method of claim 14, wherein at least one of R^1 or R^2 is t-butyl.
17. The method of claim 14, wherein at least one of R^1 or R^2 is cyclohexyl.
18. The method of claim 14, wherein R^1 and R^2 are taken together with the nitrogen atom to which they are attached, to form a pyrrole, pyrrolidine, or piperidine ring.
19. The method of claim 14, wherein R^3 is C_1 - C_6 alkyl.
20. The method of claim 14, wherein R^3 is phenyl.

21. The method of claim 14, wherein at least one of R^1 and R^2 is ethyl and R^3 is methyl or phenyl.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US2021/055707

A. CLASSIFICATION OF SUBJECT MATTER

C01B 33/107(2006.01)i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C01B 33/107(2006.01); C07F 7/12(2006.01)

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

Korean utility models and applications for utility models

Japanese utility models and applications for utility models

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

eKOMPASS(KIPO internal), STN(Registry, Caplus) & Keywords: iodosilane, dialkylamido-silane, acyl iodide

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y A	US 2016-0264426 A1 (AIR LIQUIDE ADVANCED MATERIALS, INC. et al.) 15 September 2016 (2016-09-15) claims 1, 2	1-5,8-16,19-21 6,7,17,18
Y	VORONKOV, M. G. et al., "Acyl iodides in organic synthesis: XII. Reactions with organosilicon amines", Russian journal of organic chemistry, 2008, vol. 44, No. 11, pages 1585-1589 page 1586	1-5,8-16,19-21
Y	WO 2017-201456 A1 (L' AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE et al.) 23 November 2017 (2017-11-23) claims 1, 10, 15, 16	1-5,8-16,19-21
Y	HASSLER, K. et al., "Syntheses, infrared and raman vibrational spectra, normal coordinate analyses and ²⁹ Si-NMR-Spectra of halogenated disilanes X _n Si ₂ H _{6-n} (X= F, Cl, Br, I)", Journal of molecular structure, 1995, Vol. 348, pages 353-356 page 353, 2. SYNTHESSES; table 2	14-16,19-21



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"D" document cited by the applicant in the international application

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

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International application No.

PCT/US2021/055707

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GUPPER, A. et al., "Synthesis and properties of 1,2-dichlorodisilane and 1,1,2-trichlorodisilane", European journal of inorganic chemistry, 2001, Vol. 2001, No. 8, pages 2007-2011 the whole document	1-21

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/US2021/055707

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