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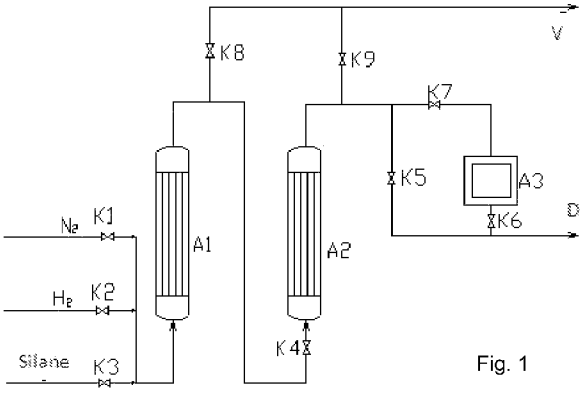

Fig. 1

(57) Abstract: A process for preparing silane from trichlorosilane is provided. The process comprises: reacting trichlorosilane with hydrogen in a first catalyst bed maintained at a temperature of from about 20 °C to about 100 °C to form a first product mixture comprising a dichlorosilane, and the first catalyst bed comprising a first anion exchange resin; separating the dichlorosilane from the first product mixture in a first distillation column; delivering the separated dichlorosilane from the first distillation column to a second catalyst bed maintained at a temperature of from about 20 °C to about 80 °C for a reaction with hydrogen to form a second product mixture comprising a silane, and the second catalyst bed comprising a second anion exchange resin; and separating the silane from the second product mixture in a second distillation column. Further, a system for preparing silane from trichlorosilane is also provided.

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PROCESS AND SYSTEM FOR PREPARING SILANE FROM TRICHLOROSILANE

CROSS-REFERENCE TO RELATED APPLICATION

This application claims priority to, and benefits of Chinese Patent Application No. 201010244420.6 filed with State Intellectual Property Office, P. R. C. on July 29, 2010, the entire content of which is incorporated herein by reference.

FIELD

The present disclosure relates to a process of silicon purification, especially to a process and a system for preparing silane by the reduction of trichlorosilane.

BACKGROUND

Silane is widely used as a key raw material in various fields, such as electronics, semiconductors, and photovoltaic industry.

Presently, UCC Silane Process (Union Carbide Corporation Silane Process) is commonly applied in the production of silane from trichlorosilane worldwidely, which is also called a disproportionation process. During a heterogeneous reaction of this process, trichlorosilane is subjected to disproportionation in the presence of a catalyst to form dichlorosilane. Then, the dichlorosilane is subjected to disproportionation in the presence of a catalyst to form silane. Two reactions regarding the process are as follows:

$2\ SiHCl_3 = SiH_2Cl_2 + SiCl_4$

$2\ SiH_2Cl_2 = SiH_4 + SiHCl_3$

The two reactions to form silane from trichlorosilane are both reversible disproportionation reactions and both have low conversion rate.

Research and development of a novel and efficient catalyst has been paid more and more attention. For example, a modified catalyst available from Mitsui Chemicals may result in a one-time conversion rate of from 8% to 10%. Moreover, various new and modified catalysts have been disclosed in many patent documents. Presently, modified catalysts may result in the highest one-time conversion rate of about 12%. However, modified catalysts are limited to experimental use for its instability, absorption to the final product silane, pollution, etc.

SUMMARY

In viewing thereof, the present disclosure is directed to solve at least one of the problems existing in the prior art. Accordingly, a process for preparing silane with high conversion rate is provided. Further, a system for preparing silane from trichlorosilane is also provided.

According to an aspect of the present disclosure, a process for preparing silane from trichlorosilane is provided, comprising: reacting trichlorosilane with hydrogen in a first catalyst bed maintained at a temperature of from about 20 ºC to about 100 ºC to form a first product mixture comprising a dichlorosilane, and the first catalyst bed comprising a first anion exchange resin; separating the dichlorosilane from the first product mixture in a first distillation column; delivering the separated dichlorosilane from the first distillation column to a second catalyst bed maintained at a temperature of from about 20 ºC to about 80 ºC for a reaction with hydrogen to form a second product mixture comprising a silane, and the second catalyst bed comprising a second anion exchange resin; and separating the silane from the second product mixture in a second distillation column.

According to another aspect of the present disclosure, a system for preparing silane from trichlorosilane is provided, comprising: a first catalyst bed configured to allow reacting trichlorosilane with hydrogen at a temperature of from about 20 ºC to about 100 ºC to form a first product mixture comprising a dichlorosilane, wherein the first catalyst bed comprises a first anion exchange resin; a first distillation column connected to the first catalyst bed and configured to receive the first product mixture and separate the dichlorosilane from the first product mixture; a second catalyst bed connected to the first distillation column and configured to receive the seperated dichlorosilane and allow the separated dichlorosilane to react with hydrogen at a temperature of from about 20 ºC to about 80 ºC to form a second product mixture comprising a silane, wherein the second catalyst bed comprises a second anion exchange resin; and a second distillation column connected to the second catalyst bed and configured to receive the second product mixture and separate the silane from the second product mixture.

With the process for preparing silane from trichlorosilane according to an embodiment of the present disclosure, a highest conversion rate may be up to 18% or 20%. Further, the

amount of the byproduct silicon tetrachloride is significantly reduced, which may greatly reduce energy consumption of the whole process. During the whole reactions of the process, the utilization rate of silicon is very high.

Additional aspects and advantages of the embodiments of the present disclosure will be given in part in the following descriptions, become apparent in part from the following descriptions, or be learned from the practice of the embodiments of the present disclosure.

BRIEF DISCRIPTION OF DRAWINGS

These and other aspects and advantages of the present disclosure will become apparent and more readily appreciated from the following descriptions taken in conjunction with the drawings, in which:

Fig. 1 is a partial flow chart of a process for preparing silane from trichlorosilane according to an embodiment of the present disclosure;

Fig. 2 illustrates a system for preparing silane from trichlorosilane according to an embodiment of the present disclosure.

DETAILED DESCRIPTION

Reference will be made in detail to embodiments of the present disclosure. The embodiments described herein are explanatory, illustrative, and used to generally understand the present disclosure. The embodiments shall not be construed to limit the present disclosure.

According to an aspect of the present disclosure, a process for preparing silane from trichlorosilane comprises: reacting trichlorosilane with hydrogen in a first catalyst bed maintained at a temperature of from about 20 ºC to about 100 ºC to form a first product mixture comprising a dichlorosilane, and the first catalyst bed comprising a first anion exchange resin; separating dichlorosilane from the first product mixture in a first distillation column; delivering the separated dichlorosilane from the first distillation column to a second catalyst bed maintained at a temperature of from about 20 ºC to about 80 ºC for reaction with hydrogen to form a second product mixture comprising silane, and the second catalyst bed comprising a second anion exchange resin; and separating silane from the second product mixture in a second distillation column.

In one embodiment, the reactions may be carried out under anhydrous and anaerobic conditions. In one embodiment, moisture and oxygen are removed from reaction containers prior to the reactions. In a further embodiment, the process further comprises purging the first and second catalyst beds with nitrogen for about 10 minutes to about 60 minutes and vacuumizing the first and second catalyst beds to reach a pressure below 100Pa prior to the reaction. In a further alternative embodiment, the process further comprises purging the first and second catalyst beds with nitrogen for about 10 minutes to about 60 minutes and vacuumizing the first and second catalyst beds to reach a pressure below 10Pa prior to the reaction. In an alternative embodiment, the purging and vacuumizing steps may be performed five to ten times.

The trichlorosilane and hydrogen may be commercially available, or may be prepared by any technique known to those skilled in the art. In one embodiment, the trichlorosilane may have a purity of above 99.99 wt%, and the hydrogen may have a purity of 99.999 wt% so as to form silane with high purity.

In one embodiment, the molar ratio of the trichlorosilane to the hydrogen in the first catalyst bed is about 4:1 to about 1:6. In an alternative embodiment, the molar ratio of the trichlorosilane to the hydrogen in the first catalyst bed is about 2:1 to about 1:4 to obtain improved conversion rate.

In one embodiment, the first catalyst bed may be maintained at a temperature of from about 20 ºC to about 100 ºC, particularly from about 60 ºC to about 100 ºC.

In one embodiment, the trichlorosilane and hydrogen may be dehydrated prior to the reaction in the first and second catalyst beds. In a further embodiment, the trichlorosilane and hydrogen may be dehydrated in a dehydration column.

In one embodiment, during the reaction, the trichlorosilane and hydrogen are passed from the bottom to the top of the first catalyst bed.

When passing through the first catalyst bed, trichlorosilane and hydrogen will react with the first anion exchange resin, which may promote the reaction for converting trichlorosilane and hydrogen into dichlorosilane and hydrogen chloride.

The reaction of trichlorosilane and hydrogen in the first catalyst bed may form dichlorosilane and hydrogen chloride. The reactions in the first catalyst bed are as follows:

Main reaction: $SiHCl_3 + H_2 = SiH_2Cl_2 + HCl$

Main side reaction: 2 $SiHCl_3$ = SiH_2Cl_2 + $SiCl_4$

Other side reactions may also take place.

The first anion exchange resin may be any one known to those skilled in the art. Catalytic active groups in the first anion exchange resin may react with chlorosilane (such as trichlorosilane), so that the intermediate product may be changed, and the activation energy of the reaction may be reduced, thus promoting the reaction of chlorosilane with hydrogen. In one embodiment, the first anion exchange resin may be a macroporous anion exchange resin, so as to facilitate chlorosilane and hydrogen to pass from the bottom to the top of the first catalyst bed without generating large pressure drop. In a further embodiment, the first anion exchange resin may comprise tertiary ammonium salt group and/or quaternary ammonium salt group as the catalytic active groups.

In one embodiment, the first anion exchange resin may have a particle size of about 0.2 millimeters to about 1.0 millimeter, particularly 0.6 millimeters. In one embodiment, the first anion exchange resin may have a filling height of about 400 millimeters to about 1,000 millimeters.

In one embodiment, trichlorosilane may react with hydrogen in the presence of the first anion exchange resin for about 2 seconds to about 5 seconds. In a further embodiment, a pressure in the first catalyst bed may be maintained at about 1 bar to 12 bar during the reaction, particularly 4 bar to 10 bar.

The first product mixture may comprise dichlorosilane and hydrogen chloride formed by the main reaction, unreacted trichlorosilane and hydrogen, and silicon tetrachloride and other materials formed by the side reactions. In one embodiment, the first product mixture may be delivered into the first distillation column to separate the dichlorosilane from the first product mixture. Remaining fractions of the first product mixture may be recycled.

In one embodiment, the process further comprises purging the second catalyst bed with nitrogen for about 10 minutes to about 60 minutes and vacuumizing the second catalyst bed to reach a pressure below 100Pa prior to the reaction. In a further alternative embodiment, the process further comprises purging the second catalyst bed with nitrogen for about 10 minutes to about 60 minutes and vacuumizing the second catalyst bed to reach a pressure below 10Pa prior to the reaction. In an alternative embodiment, the purging and vacuumizing steps may be performed five to ten times.

In one embodiment, the molar ratio of the dichlorosilane to the hydrogen in the second catalyst bed is about 3:1 to about 1:5. In a further embodiment, the molar ratio of the dichlorosilane to the hydrogen in the second catalyst bed is about 1:1 to about 1:3 to obtain an improved conversion rate.

In one embodiment, the second catalyst bed may be maintained at a temperature of from about 20 ºC to about 80 ºC. In a further embodiment, the second catalyst bed may be maintained at a temperature of from about 40 ºC to about 80 ºC.

In one embodiment, the dichlorosilane separated in the first distillation column and hydrogen may be dehydrated prior to the reaction in the second catalyst bed. In a further embodiment, the dichlorosilane and hydrogen may be dehydrated in a dehydration column.

In one embodiment, during the reaction, the dichlorosilane and hydrogen are passed from the bottom to the top of the second catalyst bed.

The reactions in the second catalyst bed may be as follows.

Main reaction: $SiH_2Cl_2 + 2H_2 = SiH_4 + 2HCl$

Main side reaction: $2\ SiH_2Cl_2 = SiH_4 + SiHCl_3$

Other side reactions may also take place.

The second anion exchange resin may be any one known to those skilled in the art, and in one embodiment of the present disclosure, the first and second anion exchange resin may be the same. Catalytic active groups in the second anion exchange resin may react with chlorosilane (such as dichlorosilane), so that the intermediate product may be changed, and the activation energy of the reaction may be reduced, thus promoting the reaction of chlorosilane with hydrogen. In one embodiment, the second anion exchange resin may be a macroporous anion exchange resin, so as to facilitate chlorosilane and hydrogen to pass from the bottom to the top of the second catalyst bed without generating large pressure drop. In a further embodiment, the second anion exchange resin may comprise tertiary ammonium salt group and/or quaternary ammonium salt group as the catalytic active groups.

In one embodiment, the second anion exchange resin may have a particle size of about 0.2 millimeters to about 1.0 millimeter, particularly 0.6 millimeters.

In one embodiment, the second anion exchange resin may have a filling height of about 400 millimeters to about 1,000 millimeters.

The first and second catalyst beds may have identical reaction principle and aim. In some embodiments, the first and second anion exchange resins may be identical or different, with identical or different particle sizes or filling heights.

In one embodiment, dichlorosilane may react with hydrogen in the presence of the second anion exchange resin for about 2 seconds to about 5 seconds. In a further embodiment, a pressure in the second catalyst bed may be maintained at about 1 bar to 10 bar during the reaction, particularly 4 bar to 8 bar, and further particularly 2 bar to 6 bar.

The second product mixture comprises silane and hydrogen chloride formed by the main reaction, unreacted dichlorosilane and hydrogen, and trichlorosilane and other materials formed by the side reactions. In one embodiment, the second product mixture may be delivered into the second distillation column to separate the silane from the second product mixture. Remaining fractions of the second product mixture may be recycled.

The first and second catalyst beds may be any one known in the art, which can be self-made or commercially available, provided that reactions therein are achieved sufficiently. For example, the first and second catalyst beds may be a single-stage bed or a two-stage bed, preferably a single-stage bed; and the first and catalyst beds may be made from ceramic or metal, with metal as preferable. The catalysts used in the first and second catalyst beds may be independently any one known in the art, which can act the catalyst for the above recited reactions. In one embodiment of present disclosure, the catalysts used in the first and second catalyst beds may be amino-functionalized polystyrenes, amino-functionalized inorganic or organopolysiloxane catalysts.

In another aspect of present disclosure, a system for preparing silane from trichlorosilane is also provided. As shown in Fig. 2, according to an embodiment of the present disclosure, a system 1000 for preparing silane from trichlorosilane comprises: a first catalyst bed 100, a first distillation column 200, a second catalyst bed 300, and and a second distillation column 400. According to one embodiment of present disclosure, the first catalyst bed 100 comprises a first anion exchange resin, and the first catalyst bed 100 is configured to allow reacting trichlorosilane with hydrogen at a temperature of from about

20 ºC to about 100 ºC to form a first product mixture comprising a dichlorosilane. The first distillation column 200 is connected to the first catalyst bed 100 and is configured to receive the first product mixture and separate the dichlorosilane from the first product mixture. The second catalyst bed comprises a second anion exchange resin connected to the first distillation column 200 and is configured to receive the seperated dichlorosilane and allow the separated dichlorosilane to react with hydrogen at a temperature of from about 20 ºC to about 80 ºC to form a second product mixture comprising a silane. The second distillation column 400 is connected to the second catalyst bed 300 and is configured to receive the second product mixture and separate the silane from the second product mixture. In some embodiments, the first and second anion exchange resins each independently have a particle size of about 0.2 millimeters to about 1.0 millimeter. In some embodiments, the first and second anion exchange resins each independently have a filling height of about 400 millimeters to about 1,000 millimeters. In some embodiments, the first and second anion exchange resins are each independently macroporous anion exchange resins containing tertiary ammonium salt group and/or quaternary ammonium salt group.

Fig. 1 shows a partial flow chart of a process for preparing silane from trichlorosilane according to an embodiment of the present disclosure. As shown in Fig. 1, a dehydration column A1, a catalyst bed A2, a chromatograph A3, valves K1-9 are included in the flow chart. Line V represents connection to a vacuum pump and line D represents connection to a distillation column.

The process for preparing silane from trichlorosilane will be described below with reference to Fig. 1.

Firstly, valves K2, K3, and K7-9 are closed, and remaining valves are opened. Secondly, nitrogen is applied to purge the reaction system for about 10 minutes to about 60 minutes. Then, valves K8 and K9 are opened, and finally the reaction system is vacuumized to reach a pressure of about 1 Pa to about 100 Pa. The above mentioned steps are performed about five to about ten times to remove the moisture and oxygen in the reaction system.

Then, valves K1, K8 and K9 are closed and valves K2, K3 and K5 are opened. Trichlorosilane and hydrogen are injected into the dehydration column A1, and then

delivered to the catalyst bed A2 for reaction with hydrogen in the catalyst bed A2 to obtain a first product mixture comprising dichlorosilane. The first product mixture is delivered to the distillation column through line D. Valve K7 is opened when the reaction is stable. A part of the first product mixture is introduced into the chromatograph A3, and its composition is analyzed.

According to embodiments of the present disclosure, conversion rates of the reaction of trichlorosilane with hydrogen and the reaction of dichlorosilane with hydrogen may be significantly increased in the presence of the first and second anion exchange resins. It has been found by the inventors that the conventional UCC silane process by means of disproportionation reaction, which may have a very low conversion rate when the reaction comes to equilibrium. According to an embodiment of the present disclosure, detailed reactions of trichlorosilane in the presence of hydrogen are as follows:

$$SiHCl_3 + H_2 = SiH_2Cl_2 + HCl$$

$$SiH_2Cl_2 + 2H_2 = SiH_4 + 2\ HCl.$$

The present disclosure will be described in detail with reference to the following embodiments. EMBODIMENTs 1 to 5 describe the reaction between trichlorosilane and hydrogen, and EMBODIMENTs 6 to 10 describe the reaction between dichlorosilane and hydrogen.

EMBODIMENT 1

The reaction system was purged with nitrogen for 40 minutes, and then the reaction system was vacuumized to reach a pressure of about 90 Pa. The purging and vacuuming steps were performed 10 times.

Trichlorosilane and hydrogen with a molar ratio of 1:2 were mixed and delivered to the first catalyst bed via an air inlet on the bottom of the first catalyst bed with a flow rate of about 1.5 L/min. The first catalyst bed was filled with the first anion exchange resin with a filling height of 800 mm, and the first anion exchange resin was a macroporous anion exchange resin comprising tertiary ammonium salt group (Amberlyst A-21) and had an average particle size of 0.59 mm.

Trichlorosilane was reacted with hydrogen in the presence of the first anion exchange resin in the first catalyst bed to form a first product mixture. During the reaction, the first catalyst bed is maintained at a temperature of 80 ºC and a pressure of 10 bar.

The first product mixture was withdrawn from the top of the first catalyst bed and delivered to the first distillation column. Dichlorosilane was separated from the first product mixture in the first distillation column, and remaining fractions were separated and recycled.

EMBODIMENT 2

EMBODIMENT 2 is substantially the same as EMBODIMENT 1 except that: trichlorosilane and hydrogen with a molar ratio of 4:1 were mixed.

EMBODIMENT 3

EMBODIMENT 3 is substantially the same as EMBODIMENT 1 except that: trichlorosilane and hydrogen with a molar ratio of 1:6 were mixed.

EMBODIMENT 4

EMBODIMENT 4 is substantially the same as EMBODIMENT 1 except that: the first catalyst bed was filled with the first anion exchange resin with a filling height of 400 mm.

EMBODIMENT 5

EMBODIMENT 5 is substantially the same as EMBODIMENT 1 except that: the first catalyst bed was filled with the first anion exchange resin with a filling height of 1000 mm.

EMBODIMENT 6

The dichlorosilane from EMBODIMENTs 1-5 and hydrogen with a molar ratio of 1:3 were mixed and delivered to the second catalyst bed via an air inlet on the bottom of the second catalyst bed with a flow rate of about 1.5 L/min. The second catalyst bed was filled with the second anion exchange resin with a filling height of 800 mm, and the second anion exchange resin was a macroporous anion exchange resin comprising quaternary ammonium salt group (Dowex MWA-1) and had an average particle size of 0.68 mm.

Dichlorosilane was reacted with hydrogen in the presence of the second anion exchange resin in the second catalyst bed to form a second product mixture. During the reaction, the second catalyst bed is maintained at a temperature of 80 ºC and a pressure of 6 bar.

The second product mixture was withdrawn from the top of the second catalyst bed and delivered to the second distillation column. Silane was separated from the second product mixture, and remaining fractions were separated and recycled.

EMBODIMENT 7

EMBODIMENT 7 is substantially the same as EMBODIMENT 6 except that: dichlorosilane and hydrogen with a molar ratio of 3:1 were mixed.

EMBODIMENT 8

EMBODIMENT 8 is substantially the same as EMBODIMENT 6 except that: dichlorosilane and hydrogen with a molar ratio of 1:5 were mixed.

EMBODIMENT 9

EMBODIMENT 9 is substantially the same as EMBODIMENT 6 except that: the second catalyst bed was filled with the second anion exchange resin with a filling height of 400 mm.

EMBODIMENT 10

EMBODIMENT 10 is substantially the same as EMBODIMENT 6 except that: the second catalyst bed was filled with the second anion exchange resin with a filling height of 1000 mm.

COMPARATIVE EMBODIMENT 1

The reaction system was purged with nitrogen for 40 minutes, and then the reaction system was vacuumized to reach a pressure of about 90 Pa. The purging and vacuuming steps were performed 10 times.

Trichlorosilane was delivered to the first catalyst bed via an air inlet on the bottom of the first catalyst bed with a flow rate of about 1.5 L/min. The first catalyst bed was filled with the first anion exchange resin with a filling height of 800 mm, and the first anion exchange resin was a macroporous anion exchange resin comprising tertiary ammonium salt group (Amberlyst A-21) and had an average particle size of 0.59 mm.

Trichlorosilane was reacted with hydrogen in the presence of the first anion exchange resin in the first catalyst bed to form a first product mixture. During the reaction, the first catalyst bed is maintained at a temperature of 80 ºC and a pressure of 5 bar.

The first product mixture was withdrawn from the top of the first catalyst bed and delivered to the first distillation column. Dichlorosilane was separated from the first product mixture in the first distillation column, and remaining fractions were separated and recycled.

COMPARATIVE EMBODIMENT 2

The dichlorosilane from COMPARATIVE EMBODIMENT 1 was delivered to the second catalyst bed via an air inlet on the bottom of the second catalyst bed with a flow rate of about 1.5 L/min. The second catalyst bed was filled with the second anion exchange resin with a filling height of 800 mm, and the second anion exchange resin was a macroporous anion exchange resin comprising quaternary ammonium salt group (Dowex MWA-1) and had an average particle size of 0.68 mm.

Dichlorosilane was reacted with hydrogen in the presence of the second anion exchange resin in the second catalyst bed to form a second product mixture. During the reaction, the second catalyst bed is maintained at a temperature of 80 ºC and a pressure of 5 bar.

The second product mixture was withdrawn from the top of the second catalyst bed and delivered to the second distillation column. Silane was separated from the second product mixture, and remaining fractions were separated and recycled.

TESTS

The first and second product mixtures were collected and tested in the chromatograph (GC-2014). Testing results were shown in Table 1 and Table 2. Some

byproducts formed by side reactions were not recorded in Table 1 and Table 2. The conversion rate (η) was calculated by the following formula:

$$\eta_{SiH2Cl2} = \frac{C_{SiH2Cl2}}{C_{SiHCl3} + C_{SiCl4} + C_{SiH2Cl2}} \times 100\%$$

$$\eta_{SiH4} = \frac{C_{SiH4}}{C_{SiH2Cl2} + C_{SiHCl3} + C_{SiH4}} \times 100\%$$

Table 1

EMBODIMENT	Content of $SiHCl_3$ (wt%)	Content of $SiCl_4$ (wt%)	Content of SiH_2Cl_2 (wt%)	$\eta_{SiH2Cl2}$ (wt%)
EMBODIMENT 1	68.57	2.43	15.71	18.1
EMBODIMENT 2	74.92	2.43	12.51	13.9
EMBODIMENT 3	71.60	2.02	14.25	16.2
EMBODIMENT 4	79.36	2.09	10.32	11.2
EMBODIMENT 5	71.42	2.26	14.32	16.3
COMPARATIVE EMBODIMENT 1	87.84	5.43	6.72	6.72

Table 2

EMBODIMENT	Content of SiH_2Cl_2 (wt%)	Content of $SiHCl_3$ (wt%)	Content of SiH_4 (wt%)	η_{SiH4} (wt%)
EMBODIMENT 6	53.71	5.62	14.69	19.8
EMBODIMENT 7	62.45	3.11	12.35	16.1
EMBODIMENT 8	58.48	4.68	13.04	17.1
EMBODIMENT 9	71.56	2.63	9.60	11.5
EMBODIMENT 10	57.20	4.69	13.60	18.0
COMPARATIVE EMBODIMENT 2	83.06	8.44	8.49	8.49

As shown in Table 1, compared with COMPARATIVE EMBODIMENT 1, with the reaction for converting trichlorosilane into dichlorosilane in EMBODIMENTs 1-5, the conversion rate may be improved, and the amount of silicon tetrachloride may be largely decreased. As shown in Table 2, compared with COMPARATIVE EMBODIMENT 2, with the reaction for converting dichlorosilane into silane in EMBODIMENTs 6-10, the conversion rate may also be largely improved. As shown in Table 1 and Table 2, with the process for preparing silane from trichlorosilane according to an embodiment of the present disclosure, the yield of the final product silane may be improved largely.

Although explanatory embodiments have been shown and described, it would be appreciated by those skilled in the art that changes, alternatives, and modifications all falling into the scope of the claims and their equivalents can be made in the embodiments without departing from spirit and principles of the disclosure.

WHAT IS CLAIMED IS:

1. A process for preparing silane from trichlorosilane, comprising:

reacting trichlorosilane with hydrogen in a first catalyst bed maintained at a temperature of from about 20 ºC to about 100 ºC to form a first product mixture comprising a dichlorosilane, and the first catalyst bed comprising a first anion exchange resin;

separating the dichlorosilane from the first product mixture in a first distillation column;

delivering the separated dichlorosilane to a second catalyst bed maintained at a temperature of from about 20 ºC to about 80 ºC for a reaction with hydrogen to form a second product mixture comprising a silane, and the second catalyst bed comprising a second anion exchange resin; and

separating the silane from the second product mixture in a second distillation column.

2. The process according to claim 1, wherein the process further comprises: purging the first and second catalyst beds with nitrogen for about 10 minutes to about 60 minutes and vacuumizing the first and second catalyst beds to reach a pressure below 100Pa prior to the reaction.

3. The process according to claim 2, wherein the process further comprises: purging the first and second catalyst beds with nitrogen for about 10 minutes to about 60 minutes and vacuumizing the first and second catalyst beds to reach a pressure below 10Pa prior to the reaction.

4. The process according to claim 2, wherein the trichlorosilane, dichlorosilane, and hydrogen are dehydrated prior to the reaction in the first and second catalyst beds.

5. The process according to claim 1, wherein the molar ratio of the trichlorosilane to the hydrogen in the first catalyst bed is about 4:1 to about 1:6.

6. The process according to claim 1, wherein the molar ratio of the dichlorosilane to the hydrogen in the second catalyst bed is about 3:1 to about 1:5.

7. The process according to claim 1, wherein the first and second anion exchange resins each independently have a particle size of about 0.2 millimeters to about 1.0 millimeter.

8. The process according to claim 1, wherein the first and second anion exchange resins each independently have a filling height of about 400 millimeters to about 1,000 millimeters.

9. The process according to claim 1, wherein the first and second anion exchange resins are each independently macroporous anion exchange resins containing tertiary ammonium salt group and/or quaternary ammonium salt group.

10. The process according to claim 1, wherein the first catalyst bed is maintained at a pressure of about 1 bar to about 12 bar during the reaction.

11. The process according to claim 1, wherein the second catalyst bed is maintained at a pressure of about 1 bar to about 10 bar during the reaction.

12. A system for preparing silane from trichlorosilane, comprising:

a first catalyst bed configured to allow reacting trichlorosilane with hydrogen at a temperature of from about 20 ºC to about 100 ºC to form a first product mixture comprising a dichlorosilane, wherein the first catalyst bed comprises a first anion exchange resin;

a first distillation column connected to the first catalyst bed and configured to receive the first product mixture and separate the dichlorosilane from the first product mixture;

a second catalyst bed connected to the first distillation column and configured to receive the seperated dichlorosilane and allow the separated dichlorosilane to react with hydrogen at a temperature of from about 20 ºC to about 80 ºC to form a second product mixture comprising a silane, wherein the second catalyst bed comprises a second anion exchange resin; and

a second distillation column connected to the second catalyst bed and configured to receive the second product mixture and separate the silane from the second product mixture.

13. The system according to claim 12, wherein the first and second anion exchange resins each independently have a particle size of about 0.2 millimeters to about 1.0 millimeter.

14. The system according to claim 12, wherein the first and second anion exchange resins each independently have a filling height of about 400 millimeters to about 1,000 millimeters.

15. The system according to claim 12, wherein the first and second anion exchange resins are each independently macroporous anion exchange resins containing tertiary ammonium salt group and/or quaternary ammonium salt group.

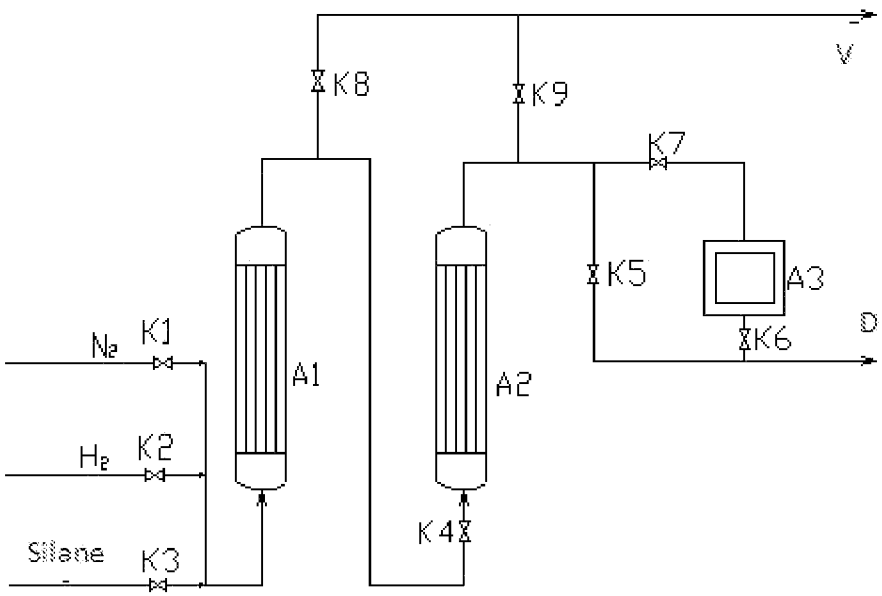

Fig. 1

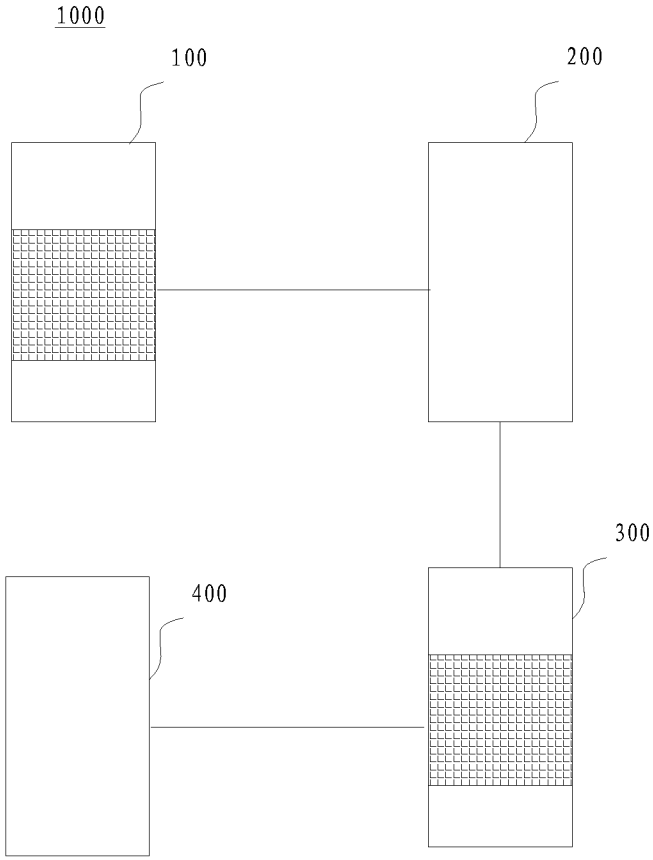

Fig. 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2011/077336

A. CLASSIFICATION OF SUBJECT MATTER

See Extra Sheet

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)

IPC: C01B 33/-

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

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

WPI, EPODOC, CNPAT, CNKI: ????CHLORO?SILANE?, H2SiCl2, SiH2Cl2, SiCl2H2, HYDROGEN?, H2, ANION, RESIN?, HSiCl3, SiHCl3, SiCl3H, TCS, DCS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X/A	US4113845A(UNION CARBIDE CORP)12 Sep. 1978(12.09.1978) lines 55-56 of column 3, lines 2 and 20-21 of column 4, lines 20-23 of column 5, lines 13-21 and 45-55 of column 6, lines 16-23 of column 10, examples 1, 7 and 8, figures 1, 3	12-15/1-11
X	CN1774397A(DEGUSSA AG et al.)17 May 2006(17.05.2006) lines 10-29 of page 4 in the description	12-15
A	JP1226712A(KOJUNDO SILICON KK)11 Sep. 1989(11.09.1989) the forth paragraph of right-up column of page 2 and figures 1-3	1-15
A	US3968199A(UNION CARBIDE CORP)06 Jul. 1976(06.07.1976)the whole	1-15

☐ 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

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim (S) or which is cited to establish the publication date of another citation or other special reason (as specified)

"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

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"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

Date of the actual completion of the international search	Date of mailing of the international search report
29 Sep. 2011(29.09.2011)	**27 Oct. 2011 (27.10.2011)**
Name and mailing address of the ISA/CN The State Intellectual Property Office, the P.R.China 6 Xitucheng Rd., Jimen Bridge, Haidian District, Beijing, China 100088 Facsimile No. 86-10-62019451	Authorized officer LI, Jie Telephone No. (86-10)62084725

Form PCT/ISA /210 (second sheet) (July 2009)

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2011/077336

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2011/077336

Continuation of: A. CLASSIFICATION OF SUBJECT MATTER

C01B 33/04(2006.01)i

C01B 33/107(2006.01)n

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