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Facile preparation of hydrochlorosilane by alkali metal halide catalyzed Si-H/Si-Cl redistribution reaction



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ABSTRACT

Various alkali metal halides were found to catalyze the Si-H/Si-Cl redistribution reaction in different polar solvents efficiently. The scope of silane substrate was studied using KF as catalyst and 18-crown-6 as cocatalyst in DMI. The alkali metal halides catalyzed redistribution system provides a useful method to prepare hydrochlorosilanes more facilely. A possible mechanism was proposed to explain the process. © 2020 Elsevier Ltd. All rights reserved.

Hydrochlorosilanes are fundamental organosilicon compounds in preparation of functional polysiloxanes, polysilazanes, and many other organosilicon materials [1]. The reactivity of Si-H and Si-Cl of the molecules endows their wide application in hydrosilylation reaction and the different nucleophilic substitution reactions towards the silicon center [2]. There are great demands for hydrochlorosilanes in industrial fields of rubber, coating, pharmaceutical, etc. [3]. However, it has been still kept a synthetic challenge to prepare these compounds efficiently and facilely.

Many studies have been focused on synthesis of hydrochlorosilanes by selective substitution of hydrogen or chlorine atom at silicon center. With NaBH₄ as reductant, dialkyldichlorosilane was selectively reduced to dialkylhydrochlorosilane successfully [4a], but the formation of borane during the reaction increases safety risk and cost of the preparation process. Catalytic hydrogenolysis of chlorosilanes with iridium or rhodium complex catalyst provides another way to prepare hydrochlorosilanes [4b,c]. However, the expensive catalysts restrict practical application of this strategy. On the other hand, selective chlorination of hydrosilanes has attracted much attention [5]. In 1992, Kunai and co-workers reported a convenient method for selective synthesis of hydrochlorosilane from hydrosilane with CuCl₂ as chloride source and Cul as catalyst [5a]. In 2017, Karina Chulsky disclosed selective chlorination of Si-H bond with HCl catalyzed by $B(C_6F_5)_3$ or B $(C_6F_5)_3/Et_2O$ [5b]. Recently, Sturm reported a Lewis base catalyzed selective chlorination of hydrosilane with HCl solution [5c]. Although great progress has been made, these methods are mainly limited to laboratory synthesis, and there is still no disclosed economical and convenient industrial process for the preparation of hydrochlorosilane. Besides, hydrosilanes are usually prepared from reduction of chlorosilane or alkoxysilane with hydrides reducing agents. It is a resource waste to some extent that converting Si-H to Si-Cl by chlorination and producing H₂ or other byproducts.

As early as in 1947, Sommer reported the first attempt on preparation of hydrochlorosilane through the redistribution reaction of hydrosilane and chlorosilane catalyzed by AlCl₃ [6a]. Then the guaternary ammonium salt and tertiary amine were found also can catalyze the Si-H/Si-Cl redistribution [6b-d]. Both of these catalytic redistribution systems have obvious disadvantages of high temperature and strict operating conditions. In addition, the separation of AlCl₃ catalyst from the silane mixture is also difficult. However, the advantage of high atom utilization efficiency of the redistribution strategy is very attractive. We have had an interest in searching for suitable catalysts to overcome the shortcomings of the redistribution and use its advantages to develop new preparation routes for hydrochlorosilanes. On the other hand, the work of Sturm about activation of Si-H bond by Lewis bases also inspired us [5c]. According to the mode they established, Lewis base, for example, chloride ion, can activate Si-H bond, then the activated Si-H bond can easily react with HCl to be chlorinated. We guessed that the activated Si-H bond by chloride ion maybe also easily



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reacted with chlorosilane, so the redistribution reaction between hydrosilane and chlorosilane may happen. Herein we demonstrated our effort on realization of a simple and efficient redistribution system of hydrosilane and chlorosilane, by using the most readily available and cheapest alkali metal halide as catalyst. This maybe will provide a more facile way to prepare hydrochlorosilanes.

We first investigated the possible redistribution reaction of ClCH₂SiH₃/ClCH₂SiCl₃ with AlCl₃ as the catalyst. Although AlCl₃ was reported can catalyze the redistribution reaction between hydrosilane and chlorosilane at high temperature without solvent [6a], no reaction was observed for the AlCl₃ catalyzed ClCH₂SiH₃/ ClCH₂SiCl₃ system in tetrahydrofuran (THF) at room temperature (Table 1, entry 1). The result may be due to that in THF, AlCl₃ cannot be well dissociated to produce enough chloride ion. To verify the assumption. THF was replaced with 1.3-dimethyl-2-imidazolidinone (DMI), which has a better ability to separate charges for its higher dielectric constant [7] according to Coulomb's law. As expected, the redistribution reaction occurred quickly with two hydrochlorosilanes formed in a total yield of 84% (Table 1, entry 2). The result verified presence of the possible Si-H/Si-Cl redistribution reaction we assumed. Considering the better solubility of lithium salts, we then used LiCl as the catalyst to investigate the workable solvent for this redistribution system. It was found that many solvents, including N, N'-1, 3-dimethylpropyleneurea (DMPU), N-Methyl pyrrolidone (NMP), hexamethyl phosphoric triamide (HMPA), tetrahydrofuran (THF), and diethylene glycol dimethyl ether (diglyme) worked well for the redistribution reaction between hydrosilane and chlorosilane (Table 1, entries 3-8). Besides, the redistribution reaction did not take place in Bu₂O or Et₂O (Table 1, entries 9 and 10), which may be due to the weaker solvent polarity of them and the low solubility of LiCl in these solvents [8a]. We also noticed that the reaction has lower conversion efficiency in CH₃CN, although it has higher polarity than diglyme. The relatively low solubility of LiCl in CH₃CN may be the reason [8b]. These results indicated the appropriate polarity of solvent and the solubility of catalyst in it are keys for the redistribution reaction.

Table 1

Optimization of the reaction.^a

 $\mathsf{CICH}_2\mathsf{SiH}_3 + \mathsf{CICH}_2\mathsf{SiCI}_3 \xrightarrow[\text{solvent, RT}]{\text{Metal halide (cat.)}} (1) \ \mathsf{CICH}_2\mathsf{SiH}_2\mathsf{CI} + (2) \ \mathsf{CICH}_2\mathsf{SiHCI}_2$

n) Products (yield) ^b
No reaction
(1): 16%, (2): 68%
(1): 15%, (2): 70%
(1): 15%, (2): 68%
(1): 16%, (2): 61%
(1): 11%, (2): 70%
(1): 10%, (2): 69%
(1): 21%, (2): 18%
(1): 5%, (2): 8%
No reaction
No reaction
(1): 16%, (2): 69%
(1): 16%, (2): 69%
(1): 28%, (2): 24%
(1): 15%, (2): 70%
(1): 15%, (2): 70%
(1): 14%, (2): 71%
(1): 16%, (2): 65%
(1): 14%, (2): 69%

 a Reaction conditions: CICH_2SiH_3 (0.005 mol), CICH_2SiCl_3 (0.01 mol), catalyst (3.0 mol/%), solvent (1 mL), room temperature.

^b Yields were determined by ¹H NMR.

We then selected DMI as the solvent to optimize the catalyst. It was found that compared with LiCl, the redistribution reaction proceeded more slowly using NaCl or KCl as catalyst (see in supporting information Figs. 36–38). Considering all of them can be completely dissolved in DMI, the difference in reaction rate of the redistribution may be due to the different solvation effect of metal cation with the dipolar aprotic solvent, i.e., Li⁺ has a larger solvation sheath than Na⁺ and K⁺ for its smaller cation size [9a], which may be more conducive to the dissociation of metal chloride ion pairs [9b]. The similar catalytic effect of these different metal chlorides also supports our previous assumption that the dissociated chloride ion of the catalyst played a key role in this redistribution process. Besides, it was found that apart from the chlorides, other common halides, such as KF, KBr, and KI, had similar catalytic effect on the redistribution (Table 1, entries 14–16). These results may reflect the similar Si-H bond activation effect of all the halogen ions produced from the dissociation of halides in dipolar aprotic solvent. On the other hand, we also found that for the model substrate system, ClCH₂SiH₃/ClCH₂SiCl₃, with each of these halides salts as catalyst, the redistribution conversion efficiencies of the reaction were close (Table 1, entries 2, 3, 11-16). As is known, 18-crown-6 was usually used to encapsulate K⁺ of KF to produce naked F⁻ for preparation of pentacoordinate silicates [10]. We speculated that the interaction of 18-crown-6 with K⁺ may also promote the redistribution of KF catalytic system and elevate the redistribution conversion efficiency. Thus, the redistribution reaction of ClCH₂SiH₃/ClCH₂SiCl₃ catalyzed by KF/18-crown-6 was carried out to verify the point (Table 1, entries 17–18). Compared with that of using KF alone as catalyst, the addition of 18-crown-6 as cocatalyst increased the redistribution conversion of 0.5 h from 52% to 81% (Table 1, entries 13, 17). But no further increase of conversion efficiency was observed even extending the reaction time to 3 h. Considering the total yield of hydrochlorosilanes in the same reaction time, as well as the cost, LiCl and KF/18-crown-6 are preferred catalysts in DMI.

On the basis of the above optimized reaction condition, we then investigated the substrate scope of the reaction in DMI, with KF/18-crown-6 or LiCl as the catalyst (Table 2). Various substituted silane substrates, including chloroalkyl, alkyl, and phenyl silane, were studied. Except for the systems of Et₂SiH₂/Et₂SiCl₂ and Ph₂-SiH₂/Ph₂SiCl₂ (Table 2, entries 7, 8, 12, 13), all the redistribution reactions proceeded efficiently with the yield mainly ranging from 44% to 71%. The large steric hindrance of double phenyl and ethyl groups may influence the interaction between substrate and F⁻ and inhibited the Si-H bond activation effect, while the relatively better conversion efficiency of Ph₂SiH₂/Ph₂SiCl₂ system may be attributed to the higher electron-withdrawing ability of phenyl than ethyl (Table 2, entries 7, 8, 12, 13). Compared with that of using LiCl as catalyst alone, the redistribution conversion catalyzed by KF/18crown-6 is 5% higher for the substrates ClCH₂SiMeH₂/ClCH₂-SiMeCl₂ and Cl₂CHSiMeH₂/Cl₂CHSiMeCl₂ (Table 2, entries 3–6), but much lower for the substrates Et₂SiH₂/Et₂SiCl₂ and Ph₂SiH₂/ Ph₂SiCl₂ (Table 2, entries 7, 8, 12, 13). The results reflected that KF/18-crown-6 catalytic system may be more easily influenced by steric hindrance effect. For substrates with less crowded substituents, the introduction of 18-crown-6 may improve the redistribution conversion slightly. Compared with previously reported redistribution system [6], the very simple and mild reaction condition is one of the great advantages of this alkali metal halides catalytic system. To further demonstrate the utility of the new system, we then carried out a larger scale redistribution reaction to prepare CICH₂SiMeHCl and Cl₂CHSiMeHCl. The pure hydrochlorosilane product ClCH₂SiMeHCl or Cl₂CHSiMeHCl were easily separated through packed fractional distillation.

To have an insight into the catalytic mechanism, we further studied Si-H/Si-Cl redistribution reaction with PhSiH3/PhSiCl3 as

Table 2

Substrate scope of the Si-H/Si-Cl redistribution reaction.^a

$R_{4-x}SiH_{x} + R_{4-x}SiCl_{x} \xrightarrow{\text{catalyst}} R_{4-x}SiH_{x-y}Cl_{y} \stackrel{\text{catalyst:}}{\downarrow} KF/18-C-6 (1)$							
Entry	$R_{4\text{-}x}SiH_x/R_{4\text{-}x}SiCl_x$	Ratio	Cat. (mol %)	T (h)	Products (yield) ^b		
1	ClCH ₂ SiH ₃ /ClCH ₂ SiCl ₃	1:2	1 (3)	10	ClCH ₂ SiHCl ₂ : 71%, ClCH ₂ SiH ₂ Cl: 14%		
2	ClCH ₂ SiH ₃ /ClCH ₂ SiCl ₃	2:1	1 (3)	10	ClCH ₂ SiHCl ₂ : 20%		
					ClCH ₂ SiH ₂ Cl: 65%		
3	ClCH ₂ SiMeH ₂ /ClCH ₂ SiMeCl ₂	1:1	1 (2)	10	ClCH ₂ SiMeHCl: 69% (56% ^c)		
4	ClCH ₂ SiMeH ₂ /ClCH ₂ SiMeCl ₂	1:1	2 (2)	10	ClCH ₂ SiMeHCl: 64%		
5	Cl ₂ CHSiMeH ₂ /Cl ₂ CHSiMeCl ₂	1:1	1 (2)	36	$Cl_2CHSiMeHCl: 70\% (60\%)$		
6	Cl ₂ CHSiMeH ₂ /Cl ₂ CHSiMeCl ₂	1:1	2 (2)	48	Cl ₂ CHSiMeHCl: 65%		
7	Et ₂ SiH ₂ /Et ₂ SiCl ₂	1:1	1 (4)	20	No reaction		
8	Et ₂ SiH ₂ /Et ₂ SiCl ₂	1:1	2 (4)	14	Et ₂ SiHCl: 15%		
9	PhSiH ₃ /PhSiCl ₃	1:2	1 (3)	20	PhSiHCl ₂ : 63%		
					PhSiH ₂ Cl: 11%		
10	PhSiH ₃ /PhSiCl ₃	2:1	1 (3)	20	PhSiHCl ₂ : 18%		
					PhSiH ₂ Cl: 44%		
11	PhMeSiH ₂ /PhMeSiCl ₂	1:1	1 (4)	20	PhMeSiHCl: 52%		
12	Ph ₂ SiH ₂ /Ph ₂ SiCl ₂	1:1	1 (4)	48	Ph ₂ SiHCl: 17%		
13	Ph ₂ SiH ₂ /Ph ₂ SiCl ₂	1:1	2 (4)	14	Ph ₂ SiHCl: 31%		

^a Reaction conditions: [entry 3–8, 11–13] hydrosilane/chlorosilane, (0.01/0.01 mol); [entry 1, 6] hydrosilane/chlorosilane, (0.01/0.02 mol); [entry 2, 7] hydrosilane/chlorosilane (0.02/0.01 mol), DMI (1 mL).

^b yields were determined by ¹H NMR.

^c Isolated yield with larger scale. Reaction conditions: hydrosilane/chlorosilane (0.2/0.2 mol), DMI (10 mL), KF/18-C-6 (2 mol %), Room temperature.

Table 3

Control experiments of the Si-H/Si-Cl redistribution reaction.^a

$PhSiH_3$	+ PhSiCl ₃ –	catalyst	- (1) PhSiH ₂	CI + (2) PhSiHCl ₂
Entry	Catalyst	Solvent	Time (h)	Products (vield) ^b
1	None	THF	6	No reaction
2	None	diglyme	6	No reaction
3	None	DMI	6	(1): 7%, (2): 19%
4	Li ₂ CO ₃	DMI	6	(1): 50%, (2): 13%
5	TBAC	DMI	6	(1): 51%, (2): 6%
6	LiCl	DMI	6	(1): 60%, (2): 11%
7	LiH	DMI	6	(1): 54%, (2): 14%

 a Reaction conditions: PhSiH_3 (0.005 mol), PhSiCl_3 (0.01 mol), catalyst (3.0 mol/ %), solvent (1 mL), room temperature.

^b Yields were determined by ¹H NMR.

the model substrate (Table 3). The solvents used herein are also Lewis bases, which have been shown to activate the Si-H bond [5c]. To prove the catalytic effect of metal chloride in the redistribution reaction, we then compared the reaction of PhSiH₃ and PhSiCl₃ in different solvents with and without LiCl. It was found that the redistribution reaction slowly proceeded in DMI, but no change was observed in THF or diglyme within experimental time (Table 3, entries 1-3). The stronger Lewis basicity of DMI than that of THF and diglyme [11] may account for the phenomenon. After addition of LiCl, the reaction in DMI was obviously accelerated (Table 3, entry 6), which clearly indicates the catalytic effect of LiCl. During the Si-H/Si-Cl redistribution process, the proportion of PhSiHCl₂ increased quickly at the initial stage, then the increase slowed down after 3 h. The proportion of PhSiH₂Cl increased first, and then decreased to about 11%, which may be due to the redistribution of the formed PhSiH₂Cl with the excess PhSiCl₃ substrate (Fig. 1).

For a better understanding of the role LiCl plays in this redistribution process, we then examined the catalytic effect of Li₂CO₃ and tetrabutylammonium chloride (TBAC) (Table 3, entries 4, 5). It was found that both of them can catalyze the redistribution of PhSiH₃ and PhSiCl₃ in DMI successfully. The result indicates that the range of catalyst is actually not limited to alkali metal halide salt, other



Fig. 1. Products yields to time during the Si-H/Si-Cl redistribution reaction of PhSiH₃/PhSiCl₃ (1:2) catalyzed by LiCl in DMI.

soluble metallic salt or non-metallic salt can also be effective catalyst for the reaction. On the other hand, the result may prove that both the Li⁺ and Cl⁻ ion play an important role in the activation of Si-H bond. The role Cl⁻ plays in this process can be easily understood by referring to the reported work about activation of Si-H bond by Lewis bases [5c], i.e., the Si-H bond is activated through the attack of Cl⁻ at the silicon center to form a pentacoordinate intermediate. In addition, the metal cation may act as the H⁻ acceptor through the interaction with the activated Si-H bond, which further promotes the dissociation of Si-H bond. Therefore, we proposed a tentative mechanism for the Si-H/Si-Cl redistribution reaction as depicted in Schemes 1 and 2. The Si-H bond of hydrosilane is first activated through attack of Lewis base like Cl⁻ or DMI at the silicon center to form a pentacoordinate intermediate. Then the activated Si-H bond of the intermediate breaks to form hydride and the corresponding substituted product. The existence of Li⁺



Scheme 1. Plausible mechanism of DMI solvent catalytic Si-H/Si-Cl redistribution process.



Scheme 2. Plausible mechanism of LiCl catalytic Si-H/Si-Cl redistribution process.

in the solution may accelerate this process. On the other hand, the hydride produced *in situ* can reduce Si-Cl to Si-H bond again and produce Cl⁻, which then participates in the next chlorination process. The two processes occur simultaneously and drive the redistribution reaction between hydrosilane and chlorosilane.

To verify the LiCl catalytic mechanism described in Scheme 2, the redistribution reaction of PhSiH₃/PhSiCl₃ catalyzed by LiH was carried out in DMI, which proceeded successfully as expected (Table 3, entry 7). In addition, we noticed that various hydrides have been reported to catalyze the Si-H/Si-C exchange reaction of hydrosilane in different solvents, and a similar mechanism involving hypervalent silicon hydrides intermediate was proposed [12]. A fast racemization of the optically active 1-Np-PhMeSiH catalyzed by hydrides was also observed, which can be rationalized in terms of the formation of a pentacoordinate hydridoorganosilyl anion [12b]. Considering chlorine on silicon is a much better leaving group than alkyl or aryl substituent [13], the Si-H bond may redistribute with Si-Cl bond without rupturing the Si-C bond through similar ligand exchange process. Therefore, we think the mechanism study of Si-H/Si-C redistribution reaction catalyzed by hydrides may be a good reference for understanding the mechanism we proposed herein to explain the Si-H/Si-Cl redistribution reaction.

In summary, we have established a metal halide catalyzed Si-H/ Si-Cl redistribution system. Various simple and common alkali metal halides can be utilized as the catalyst. The redistribution reaction may take place through Lewis base participated silicon pentacoordinate intermediate. The very mild reaction conditions and the economically practical strategy are the advantages the redistribution system possesses, compared with the Si-H/Si-Cl redistribution system reported previously. Further improvement of the redistribution system is pursued in our laboratory.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2020.152235.

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