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Tetrahedron Letters

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Synthesis of 1-hydrocarbon substituted cyclopropyl silyl ketones

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ARTICLE INFO

Article history:

Received 28 June 2018

Revised 28 August 2018

Accepted 6 September 2018

Available online xxx

Keywords:

Cyclopropyl silyl ketone
 α,β -Unsaturated aldehyde
 Sulfoxonium ylide
 Cyclopropanation
 Umpolung

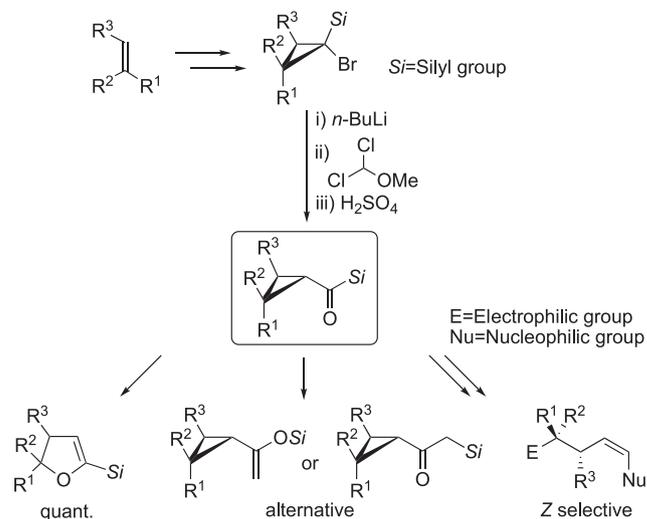
ABSTRACT

The synthesis of cyclopropyl silyl ketones possessing a hydrocarbon group at 1-position of three-membered ring was investigated. The reaction of sulfoxonium ylide with α,β -unsaturated acylsilanes derived from α,β -unsaturated aldehydes did not afford the desired acylsilane derivatives. Instead, the corresponding silyl enol ethers were yielded exclusively. On the other hand, the Corey-Chaykovsky cyclopropanation of α -substituted α,β -unsaturated aldehydes proceeded well to give 1-substituted cyclopropyl aldehydes. The silyl substitution of formyl proton in the obtained aldehydes via umpolung of carbonyl group afforded the target acylsilanes.

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Introduction

Acylsilanes will become useful synthetic intermediates due to specific properties of sterically congested and electrically positive silyl group and carbonyl group that is one of the most reactive organic functional groups [1]. We have currently investigated the newly synthetic methods for functionalized acylsilanes [2] and their synthetic utilities [3]. Especially, cyclopropyl silyl ketones have attracted attention as useful synthetic intermediates, because three specific reaction sites, cyclopropyl group, silyl group, and carbonyl group are present in one molecule [4]. Therefore we have become interested in exploring the chemistry of cyclopropyl silyl ketones. For example, we have previously described a route to cyclopropyl silyl ketones via three steps beginning with simple alkenes [5], and then reported that the efficient synthesis of silyl-substituted dihydrofurans [6], silyl enol ethers or β -ketosilanes [7] and the stereoselective synthesis of *Z*-homoallyl derivatives [8] using specific reaction sites of cyclopropyl silyl ketones (Scheme 1). Our synthetic method is convenient and gives many kinds of cyclopropyl silyl ketones having various hydrocarbon substituents on 2- and/or 3-positions of three-membered ring. However, this procedure is valuable to make cyclopropyl silyl ketones possessing an electrophilic group such as halogen, phenylthio group, phenylseleno group on 1-position, but cannot afford 1-hydrocarbon substituted cyclopropyl silyl ketones that would be useful building blocks in organic synthesis for multiply substituted hydrocarbon compounds [5a]. Hitherto, only one example of

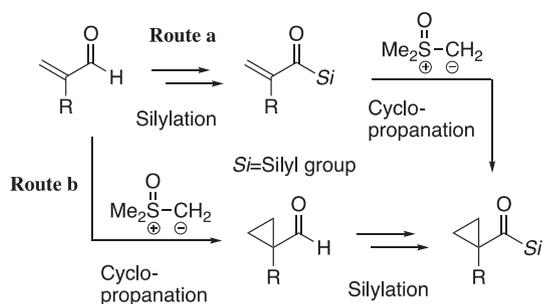


Scheme 1. Synthesis and reactions of cyclopropyl silyl ketones.

synthesis of cyclopropyl silyl ketones having a methyl group on 1-position of cyclopropane ring has been reported by Danhaizer about 30 years ago [4a,4e,9]. In this letter, we wish to describe the investigation into the silylation and cyclopropanation of α,β -unsaturated aldehydes as starting materials in two routes to prepare 1-hydrocarbon substituted cyclopropyl silyl ketones in which no one has ever synthesized (Scheme 2). Herein we would also like to state about the reaction behavior of α,β -unsaturated acylsilanes in Corey-Chaykovsky reaction [10].

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Scheme 2. Two synthetic routes to cyclopropyl silyl ketones from α,β -unsaturated aldehydes.

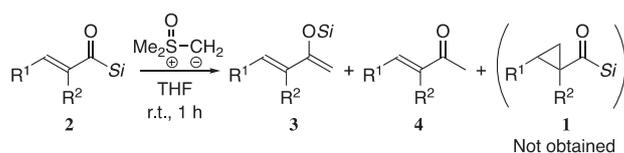
Results and discussion

Initially the cyclopropanation of α,β -unsaturated acylsilanes by Corey-Chaykovsky reaction to obtain cyclopropyl silyl ketones **1** was carried out (Route a in Scheme 2). The results were summarized in Table 1. First of all, to optimize the conditions of Corey-Chaykovsky reaction using α,β -unsaturated acylsilanes, the reaction of readily accessible β -substituted α,β -unsaturated acylsilanes **2** with dimethylsulfoxonium methylide was explored (Entries 1–4). The β -substituted α,β -unsaturated acylsilanes **2** were prepared according to the literature methods [2c]. The 3-phenyl substituted ethenyl silyl ketones reacted with sulfoxonium ylide [11] that was generated by the reaction of trimethylsulfoxonium iodide with *n*-butyllithium. However, in these reactions, the Corey-Chaykovsky cyclopropanation did not proceed at all, and the corresponding silyl enol ethers **3** and hydrolyzed ketones **4** were exclusively afforded (Entry 1). The reaction under low temperature showed lower yield (Entry 2). The ylide derived from sulfoxonium chloride

reacted with acylsilane to give nearly the same result (Entry 3). The reaction using ylide generated from sulfoxonium iodide with sodium hydride instead of *n*-butyllithium preferentially provided **4** with low yield (Entry 4). In other reactions using different acylsilanes, **3** and **4** were also yielded independent of the kind of substituents on silicon atom or alkene moiety (Entries 5–7). Then the cyclopropanation of 2-methylethenyl silyl ketone, which should afford the desired 1-methyl substituted cyclopropyl silyl ketone, also proceeded to give the corresponding silyl enol ether exclusively (Entry 8). The results mentioned above suggest the following mechanism for the reaction (Scheme 3). Generally, Corey-Chaykovsky cyclopropanation proceeds via 1,4-addition of sulfoxonium ylide to α,β -unsaturated ketone **2** to afford cyclopropyl ketone through an intermediate **A**. However, in the reaction using α,β -unsaturated silyl ketone and sulfur ylide, 1,2-addition took precedence due to the resonance stabilization of allyl anions **C** and **D** derived via Brook rearrangement [12] of silyl group through the 1,2-adduct intermediate **B**. Resultingly, the elimination of dimethyl sulfoxide from allyl anion **C** gave the silyl enol ether **3** [7]. In other words, the presence of easy rearrangeable silyl group prevents the Corey-Chaykovsky cyclopropanation of α,β -unsaturated carbonyl compounds. These results strongly suggest that the target compounds, 1-hydrocarbon substituted cyclopropyl silyl ketones cannot be synthesized via route a in Scheme 2.

As mentioned above, it became clear that the presence of silyl group interfered with the 1,4-addition of the ylide to the acylsilanes that would lead to the formation of desired cyclopropyl ketones, then the order of silylation and cyclopropanation using α,β -unsaturated aldehydes were reversed (Route b in Scheme 2). According to the literature, α,β -unsaturated aldehydes having a *n*-hexyl, benzyl, isopropyl, or phenyl group were prepared from simple and accessible aldehydes, as starting materials for cyclopropanation [13,14]. Firstly Corey-Chaykovsky reaction of enone

Table 1
Reaction of α,β -unsaturated acylsilanes **2** with dimethylsulfoxonium methylide.



Entry	Substrate	Temp. (°C)	Total yield (%) ^a	Relative ratio 3/4
1		r.t.	83	98/2
2		–50	50 ^b	92/8
3		r.t.	83 ^{b,c}	>99/1
4		r.t.	22 ^d	8/92
5		r.t.	98 ^b	>99/1
6		r.t.	61 ^b	84/16
7		r.t.	75 ^b	90/10
8		r.t.	70	>99/1

Molar ratio; acylsilane (**2**)/ylide = 1:1.2.

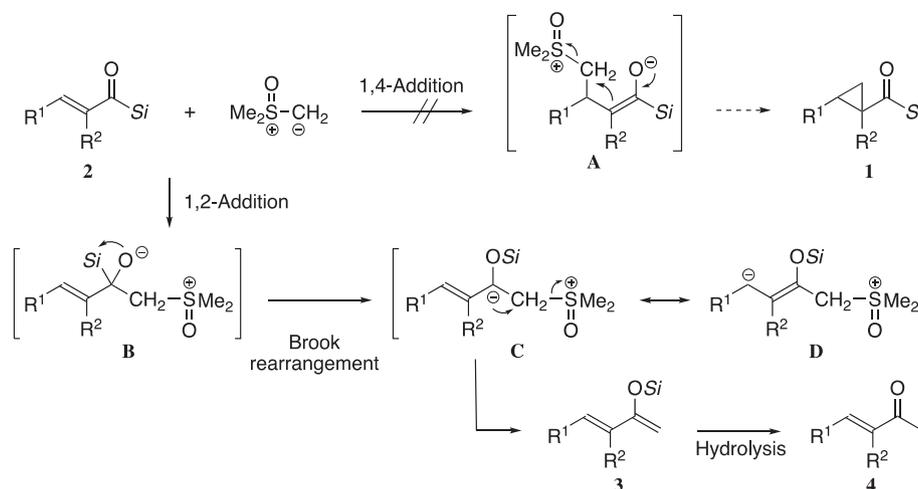
Ylide was generated by reaction of trimethylsulfoxonium iodide with *n*-BuLi.

^a Isolated yield.

^b Determined by ¹H NMR.

^c Ylide was prepared from sulfoxonium chloride with *n*-BuLi.

^d Ylide was prepared from sulfoxonium iodide with NaH.



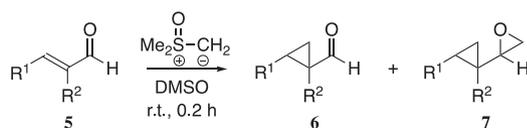
Scheme 3. Plausible reaction mechanism.

5 with dimethylsulfoxonium methylide was investigated to convert the olefin moiety of **5** into three-membered ring. The results were summarized in Table 2. The reaction of 2-methyleneoctanal with sulfoxonium ylide generated by the treatment of trimethylsulfoxonium iodide with sodium hydride proceeded to afford the corresponding cyclopropyl aldehyde **6** in good yield with the formation of epoxide as a by-product **7** (Entry 1). Meanwhile, the reaction using sulfoxonium ylide prepared by treating sulfoxonium iodide with *n*-butyllithium in THF did not proceed well. In these reactions, the desired cyclopropyl aldehydes were obtained independent with the kind of substituents on the α -carbon (Entries

1–4). Furthermore, the reaction of perillaldehyde gave the corresponding cyclopropyl aldehyde possessing substituents on C¹ and C² of cyclopropane ring (Entry 5). The obtained cyclopropyl aldehyde was a single diastereomer that was yielded by nucleophilic attack of ylide from the same side of propenyl group on cyclohexene plane. Though, it is unclear at present why this reaction proceeded with high stereoselectivity. In the near future, other common cyclopropanation methods will be tried to investigate the scope and limitations of this procedure.

Since the direct conversion of aldehyde **6** to silyl ketone **1** is impossible, the umpolung of carbonyl group of **6** was employed by thioacetal formation [15]. The aldehydes **6** were converted to thioacetals **8** in good yields by reaction with 1,3-propanedithiol and iodine [16]. These results were shown in Table 3. The depro-

Table 2
Reaction of α,β -unsaturated aldehydes **5** with dimethylsulfoxonium methylide.



Entry	Substrate	Temp. (°C)	Total yield (%)	Relative ratio 6/7
1		50	73 ^a	93/7
2		50	54 ^a	>99/1
3		r.t.	48 ^b	96/4
4		r.t.	94 ^b	>99/1
5 ^c		r.t.	45 ^a	>99/1

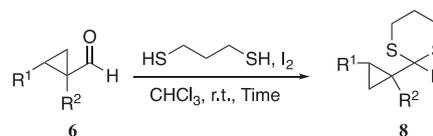
Molar ratio; enone (**5**)/ylide = 1:1.2.

^a Isolated yield.

^b Determined by ¹H NMR.

^c 0.5 eq. of ylide was used.

Table 3
Protection and carbonyl umpolung of cyclopropyl aldehydes **6**.



Entry	Substrate	Time (h)	Yield ^a (%)
1		1	96
2		23	77
3		20	65
4		1	66
5		1	56

Molar ratio; cyclopropyl aldehyde (**6**)/HS(CH₂)₃SH/I₂ = 1:1.1:0.1.

^a Isolated yield.

nation of resulting thioacetals **8** with *n*-butyllithium, followed by electrophilic reaction with chlorosilanes to 2-lithiodithiane intermediates afforded 2-silylated 1,3-dithiane derivatives **9** [17]. The results were summarized in Table 4. The reactivity of silylation was affected by the kind of substituents on cyclopropane ring and silyl group. The reaction of dithiane possessing *n*-hexyl group on three-membered ring with Me₃SiCl as a silylation reagent promptly proceeded to afford the corresponding 2-silyldithiane **9** quantitatively (Entry 1). However, the yields decreased with the increasing bulkiness of the silyl groups (Entries 2–4). In particular, the reaction with *t*-BuMe₂SiCl needed 24 h to the end (Entry 4), and the substitution of *i*-Pr₃Si group did not proceed at all (Entry 5). The silylation using silyl chloride having phenyl groups on silicon atom afforded a similar result (Entries 6–8). In an analogous way, the yields of the reaction using cyclopropyldithianes **8** possessing substituent other than *n*-alkyl group on three-membered ring decreased with the increasing bulkiness of the substituent groups (Entries 9–12). Especially, the reaction of 1-phenylcyclopropyldithiane or bicyclo[4,1,0]heptyldithiane needed excessive amounts of reactants to obtain the desired products in good yields (Entries 11 and 12).

Finally, deprotection of 2-cyclopropyl-2-silyl-1,3-dithianes **9** was investigated. The treatment of silyldithianes **9** with iodine and calcium carbonate in the THF/water combined solvent gave the corresponding cyclopropyl silyl ketones **1** (Table 5) [18]. These reactions proceeded in moderate to good yields independent of the substituents on cyclopropane ring and silyl groups, except for the reaction of cyclopropyldithiane having bulky silyl group and bicyclo[4,1,0]heptyldithiane (Entries 4 and 10). These results suggest that the attack of iodide to sulfur atom of dithiane possessing a huge alkyl or silyl group will be difficult. Since the dedithioacetalization of silyldithiane having a phenyl group on cyclopropane ring proceeded in moderate yield (Entry 9), the newest convenient and efficient method was attempted (Scheme 4). The

Table 4
Silylation of dithiane derivatives **8**.



Entry	R	Si	Time (h)	Yield ^a (%)
1		Me ₃ Si	1	>99
2		Et ₃ Si	1	57
3		<i>n</i> -OctMe ₂ Si	1	67
4		<i>t</i> -BuMe ₂ Si	24	63
5		<i>i</i> -Pr ₃ Si	24	–
6		PhMe ₂ Si	24	70
7		Ph ₂ MeSi	24	66
8		Ph ₃ Si	24	–
9		Me ₃ Si	1	53
10		Me ₃ Si	1	67
11 ^b		Me ₃ Si	1	82
12 ^c		Me ₃ Si	1	92

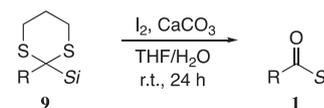
Molar ratio; dithiane (**8**)/*n*-BuLi/SiCl = 1:2.0:2.4.

^a Isolated yield.

^b 8 eq. of *n*-BuLi and 10 eq. of Me₃SiCl were used.

^c 4 eq. of *n*-BuLi and 5 eq. of Me₃SiCl were used.

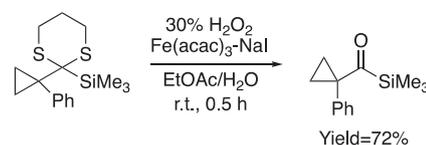
Table 5
Synthesis of cyclopropyl silyl ketones **1**.



Entry	R	Si	Yield ^a (%)
1		Me ₃ Si	78
2		Et ₃ Si	90
3		<i>n</i> -OctMe ₂ Si	56
4		<i>t</i> -BuMe ₂ Si	Complex mixture
5		PhMe ₂ Si	77
6		Ph ₂ MeSi	65
7		Me ₃ Si	71
8		Me ₃ Si	56
9		Me ₃ Si	46
10		Me ₃ Si	Trace

Molar ratio; silyldithiane (**9**)/I₂/CaCO₃ = 1:6:6.

^a Isolated yield.



Scheme 4. Synthesis of acylsilane from the reaction using 30% H₂O₂ catalyzed by Fe(III)-NaI.

reaction of silyldithiane with 30% hydrogen peroxide catalyzed by Fe(acac)₃-NaI afforded the corresponding acylsilane in good yield [19]. Though Kiriara have been reported that the deprotection methods gave different acylsilanes from the corresponding dithiane derivatives in high yields independent of the kind of substituents [19], the reaction of bicyclo[4,1,0]heptyldithiane afforded complex mixture in our case.

Conclusion

In conclusion, synthesis of 1-hydrocarbon substituted cyclopropyl silyl ketones starting from α,β -unsaturated aldehydes has been described. The Corey-Chaykovsky cyclopropanation reaction of α,β -unsaturated acylsilanes derived from silylation of the corresponding aldehydes did not proceed to give the desired 1-alkyl substituted cyclopropyl silyl ketones at all. In these reactions the corresponding silyl enol ethers or hydrolyzed ketones were exclusively afforded via Brook rearrangement. Therefore, the order of silylation and cyclopropanation using α,β -unsaturated aldehydes were reversed. The Corey-Chaykovsky cyclopropanation of α -substituted α,β -unsaturated aldehydes with dimethylsulfoxonium methylide proceeded well to yield the cyclopropyl aldehydes. Before silylation of carbonyl group in the obtained aldehydes, umpolung of the carbonyl group via formation of dithiane derivatives from aldehydes was carried out. The deprotection of resulting thioacetals with *n*-butyllithium, followed by electrophilic reaction with chlorosilanes to 2-lithiodithiane intermediates afforded 2-silylated 1,3-dithiane derivatives. Finally, deprotection of 2-cyclopropyl-2-silyl-1,3-dithianes with iodine or hydrogen

peroxide catalyzed by Fe(acac)₃-NaI gave the corresponding desired acylsilanes. Further studies are aimed at expanding the scope of these reactions in our laboratory. The results will be reported in due course.

Acknowledgments

This work was supported by Kanazawa University SAKIGAKE Project and Japan Society for the Promotion of Science (JSPS) KAKENHI Grant Number 1805198.

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