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Short Communication

New catalytic route to (*E*)-β-silyl-α,β-unsaturated ketones ☆

Piotr Pawluć *

Department of Organometallic Chemistry, Faculty of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznan, Poland

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ABSTRACT

(*E*)-β-(Silyl)-α,β-unsaturated ketones have been efficiently synthesized *via* one-pot sequential ruthenium-catalyzed silylative homo-coupling of dimethylphenylvinylsilane or trimethylvinylsilane (Marciniec coupling) and rhodium-catalyzed selective desilylative acylation (Narasaka coupling) of (*E*)-1,2-bis(silyl)ethenes with acid anhydrides. Synthetic strategy relies on the selective mono-substitution of the bis(silyl)ethene intermediate.

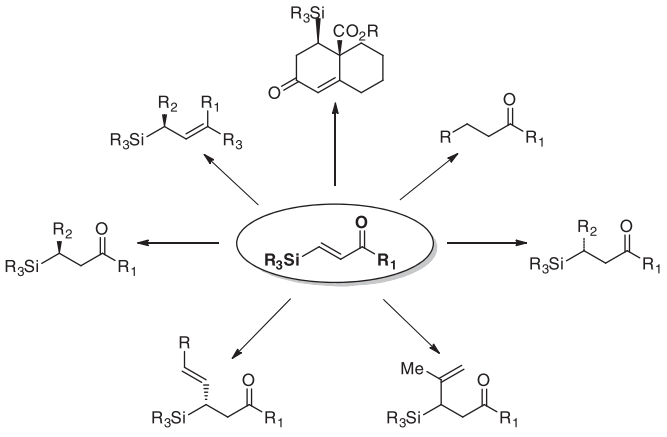
1. Introduction

β-Silyl carbonyl compounds are versatile building blocks that have significant potential in stereoselective synthesis [1]. Their use as precursors for a number of reactions such as asymmetric conjugate addition [2], Robinson annulation [3], Diels–Alder reaction [4] *etc*., greatly stimulates their synthetic advancements (Scheme 1).

Conventional approaches to (*E*)-β-silyl-α,β-unsaturated ketones involve multi-step reactions starting from alkyne derivatives [1]. Acyclic (*E*)-β-silyl-α,β-unsaturated ketones have been obtained in three-steps from silylacetylenes *via* sequential homologation–hydride reduction–Swern oxidation [2b,5]. The hydrosilylation of propargyl alcohols followed by oxidation provides an alternative route to β-silyl carbonyl systems [6]. Several independent methods such as consecutive alkyne hydrosilylation–carbonylation [7], silylmetalation–acylation [8], hydroacylation of silylalkynes [9] or silylation of acetals of β-sulfonyl ketones [10] have also been investigated. However, the application of these methods is often limited by complicated synthetic procedures involving the use of harmful starting materials and highly reactive organometallic compounds.

The silylative coupling of olefins with vinyl-substituted organosilicon compounds (Marciniec coupling), which has been developed in the last two decades as a new effective catalytic activation of the C–H bond of olefins and C–Si bond of organosilicon compounds (generally occurring in the presence of complexes containing initially or generating *in situ* M–H and M–Si bonds) [11] appears to be a valuable step in a combination with subsequent desilylation reactions [12]. The mechanism of the process proposed for the Ru-complexes proceeds *via* insertion of vinylsilane into Ru–H bond and β-Si transfer to the metal with elimination of ethylene to generate Ru–Si species, followed by insertion of alkene and β-H transfer to the metal with elimination of the substituted vinylsilane [11].

On the other hand, acylation of stereodefined vinylsilanes by acid halides in the presence of Lewis acids proceeds in a regioselective manner to afford α,β-unsaturated ketones [13]. Recently, Narasaka and co-workers have developed a rhodium complex-catalyzed acylation of vinylsilanes with acid anhydrides which avoids the use of large quantities of strong Lewis acids and highly reactive and harmful acid



Scheme 1. Synthetic utility of β-silyl-α,β-unsaturated ketones.

☆ Dedicated to Professor Bogdan Marciniec on the occasion of his 70th birthday in recognition of his significant contribution to organometallic chemistry and homogeneous catalysis.

* Fax: +48 618291508.
E-mail address: piotrpaw@amu.edu.pl.

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halides [14]. The mechanism of the catalytic acylation proceeds *via* transmetalation between the Rh(I) complex and vinylsilane to afford vinylrhodium intermediate, followed by sequential oxidative addition of acid anhydride and reductive elimination of α,β-unsaturated ketone with regeneration of Rh(I) catalyst [14]. The sequential Hiyama cross-coupling/Narasaka acylation of (*E*)-1-(diethoxymethylsilyl)-2-(dimethylphenylsilyl)-ethene has been also successfully applied to the synthesis of α,β-unsaturated carbonyl motifs [15].

Recently, we have reported a new efficient protocol for the highly stereoselective one-pot synthesis of (*E*)-styryl ketones from styrenes based on sequential ruthenium-catalyzed silylative coupling–rhodium-catalyzed desilylative acylation reaction [17]. We have envisaged that the ruthenium-catalyzed (*E*)-selective silylative homocoupling of dimethyl-phenylvinylsilane (Marciniec coupling) followed by rhodium-catalyzed desilylative acylation (Narasaka coupling) can be a valuable synthetic method for one pot conversion of vinylsilanes into (*E*)-β-silyl-α,β-unsaturated ketones, which are versatile intermediates in organic and organosilicon syntheses.

In this communication we report a facile one-pot preparation of (*E*)-β-(silyl)-α,β-unsaturated ketones from vinylsilanes *via* the corresponding (*E*)-1,2-bis(silyl)ethene intermediate.

2. Experimental

2.1. General procedure for the synthesis of (E)-β-(dimethylphenylsilyl)-α,β-unsaturated ketones and spectroscopic data of the selected products

A mixture consisting of 0.91 mL (5 mmol) of dimethylphenylvinylsilane, and 72.6 mg (0.1 mmol) of $RuHCl(CO)(PCy_3)_2$, and 10 mL of dry toluene was placed under Ar atmosphere in a Schlenk bomb flask fitted with a plug valve and heated at 110 °C for 24 h. After the action was completed (GC analysis), 0.25 mmol (97.2 mg) of $[RhCl(CO)_2]_2$ and 15 mmol of anhydride were added. The mixture was stirred for 24 h at 120 °C. After this time the solvent was evaporated and the mixture was diluted with 15% aqueous solution of NaOH (50 mL) and Et_2O (50 mL) and stirred for 10 min. At this time, the layers were separated and the aqueous layer was washed with Et_2O (3 × 50 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The remaining yellow oil was purified by silica gel chromathography (25:1 hexane: Et_2O) to give the corresponding ketone.

(*E*)-4-(dimethylphenylsilyl)but-3-en-2-one (2a)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.44 (s, 6H, $SiMe_2$), 2.28 (s, 3H, CH_3), 6.48 (d, 1H, *J* = 19.2 Hz, Si*CH*═CH), 7.13 (d, 1H, *J* = 19.1 Hz, SiCH═C*H*), 7.37–7.41 (m, 3H, Ph), 7.50–7.53 (m, 2H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ = −3.3 ($SiMe_2$), 26.3 (CH_3), 128.0, 129.5, 133.8, 136.3 (Ph), 144.1 (SiCH═*C*H), 145.5 (Si*C*H═CH), 198.6 (CO). MS m/z (rel. int.): 203 ([M − 1]$^+$ 52%), 190 (25), 189 (100), 127 (20). HRMS calcd for $C_{12}H_{15}OSi$: 203.08922; found 203.08882.

(*E*)-1-(Dimethylphenylsilyl)pent-1-en-3-one (2b)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.43 (s, 6H, $SiMe_2$), 1.08–1.12 (t, 3H, *J* = 7.3 Hz, CH_3), 2.60–2.65 (q, 2H, *J* = 7.3 Hz, CH_2), 6.52 (d, 1H, *J* = 19.2 Hz, Si*CH*═CH), 7.14 (d, 1H, *J* = 19.1 Hz, SiCH═C*H*), 7.37–7.39 (m, 3H, Ph), 7.49–7.52 (m, 2H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ = −3.2 ($SiMe_2$), 7.9 (CH_3), 32.7 (CH_2), 128.0, 129.5, 133.8, 136.5 (Ph), 143.2 (SiCH═*C*H), 144.0 (Si*C*H═CH), 200.8 (CO). MS m/z (rel. int.): 217 ([M − 1]$^+$ 12%), 190 (20), 189 (100), 145 (10), 135 (24). HRMS calcd for $C_{13}H_{17}OSi$: 217.10487; found 217.10387.

(*E*)-1-(Dimethylphenylsilyl)hex-1-en-3-one (2c)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.42 (s, 6H, $SiMe_2$), 0.91–0.96 (t, 3H, *J* = 7.4 Hz, CH_3), 1.58–1.67 (m, 2H, CH_2), 2.55–2.60 (t, 2H, *J* = 7.3 Hz, CH_2), 6.50 (d, 1H, *J* = 19.0 Hz, Si*CH*═CH), 7.13 (d, 1H, *J* = 19.1 Hz, SiCH═C*H*), 7.38–7.40 (m, 3H, Ph), 7.49–7.53 (m, 2H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ = −3.2 ($SiMe_2$), 13.8 (CH_3), 17.5 (CH_2), 41.4 (CH_2), 128.0, 129.5, 133.8, 136.2 (Ph), 143.5 (SiCH═*C*H), 144.2 (Si*C*H═CH), 200.4 (CO). MS m/z (rel. int.): 231 ([M]$^+$ 10%), 190 (15), 189 (100), 145 (10), 135 (25). HRMS calcd for $C_{14}H_{19}OSi$: 231.12051; found 231.12124.

(*E*)-1-(Dimethylphenylsilyl)-4-methylpent-1-en-3-one (2d)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.43 (s, 6H, $SiMe_2$), 1.12 (d, 6H, *J* = 6.8 Hz, $CH(CH_3)_2$), 2.92–2.96 (m, 1H, $CH(CH_3)_2$), 6.56 (d, 1H, *J* = 19.1 Hz, Si*CH*═CH), 7.20 (d, 1H, *J* = 19.1 Hz, SiCH═C*H*), 7.35–7.40 (m, 3H, Ph), 7.49–7.52 (m, 2H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ = −3.0 ($SiMe_2$), 18.6 (CH_3), 37.5 (CH), 128.0, 129.6, 133.9, 136.6 (Ph), 142.2 (SiCH═*C*H), 144.4 (Si*C*H═CH), 202.8 (CO).HRMS calcd for $C_{14}H_{19}OSi$: 231.12051; found 231.12112.

(*E*)-3-(Dimethylphenylsilyl)-1-phenylprop-2-en-1-one (2e)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.43 (s, 6H, $SiMe_2$), 7.20 (d, 1H, *J* = 18.7 Hz, −Si*CH*═CH−), 7.34–7.42 (m, 4H, Ph and −SiCH═*CH*−), 7.48–7.52 (m, 5H, Ph), 7.85–7.89 (m, 2H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ = −3.0 (−$SiMe_2$−), 128.0, 128.6, 128.8, 129.6, 132.8, 133.8, 136.5, 137.4 (Ph), 139.4 (SiCH═*C*H), 147.4 (Si*C*H═CH), 190.4 (CO). MS m/z (rel. int.): 265 ([M]$^+$ 100%), 251 (25), 189 (20), 135 (18), 105 (30), 77 (25). HRMS calcd for $C_{17}H_{17}OSi$: 265.10486; found 265.10380.

(*E*)-1-(dimethylphenylsilyl)-4-methylpenta-1,4-dien-3-one (2f)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.44 (s, 6H, $SiMe_2$), 1.94 (s, 3H, CH_3), 5.82–5.92 (m, 2H, $=CH_2$), 7.05 (d, 1H, *J* = 18.6 Hz, Si*CH*═CH), 7.36–7.40 (m, 3H, Ph), 7.50–7.54 (m, 2H, Ph). ^{13}C NMR (75 MHz, $CDCl_3$): δ = −3.0 ($SiMe_2$), 17.9 (CH_3), 125.3, 127.9, 133.8, 138.5 (Ph), 129.5 (>C═*C*H$_2$), 142.2 (>*C*═CH$_2$), 144.7 (SiCH═*C*H), 145.5 (Si*C*H═CH), 196.6 (CO). MS m/z (rel. int.): 229 ([M − 1]$^+$ 25%), 215 (100), 197 (10), 141 (30), 135 (50), 105 (10). HRMS calcd for $C_{14}H_{17}OSi$: 229.10487; found 229.10530.

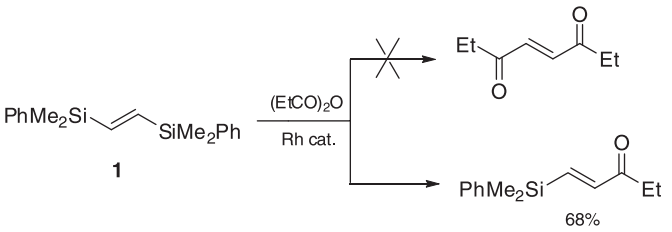
2.2. General procedure for the synthesis of (E)-β-(trimethylsilyl)-α,β-unsaturated ketones

A mixture consisting of 0.73 mL (5 mmol) of trimethylvinylsilane, and 25.6 mg (0.05 mmol) of $[RuCl_2(CO)_3]_2$, and 10 mL of dry toluene was placed under Ar atmosphere in a Schlenk bomb flask fitted with a plug valve and heated at 110 °C for 48 h. After the reaction was completed (GC analysis), 0.25 mmol (97.2 mg) of $[RhCl(CO)_2]_2$ and 15 mmol of anhydride were added. The mixture was stirred for 24 h at 120 °C. After this time the solvent was evaporated and the mixture was diluted with 15% aqueous solution of NaOH (50 mL) and Et_2O (50 mL) and stirred for 10 min. At this time, the layers were separated and the aqueous layer was washed with Et_2O (3 × 50 mL). The combined organic layers were dried over Na_2SO_4, filtered and concentrated *in vacuo*. The remaining oil was purified by silica gel chromathography (25:1 hexane:Et_2O) to give the corresponding ketone. The structures of synthesized compounds were confirmed by GC–MS and NMR spectroscopy maching data reported in the literature: (*E*)-4-(trimethylsilyl)but-3-en-2-one (70% yield) [2a] and (*E*)-1-(trimethylsilyl)pent-1-en-3-one (75% yield) [20].

3. Results and discussion

During the course of our studies on the reactivity of bis(silyl) alkenes towards carbon electrophiles, we have unexpectedly found that (*E*)-1,2-bis(dimethylphenylsilyl)ethene **1**, in the reaction with 3 equivalents of propionic anhydride in the presence of rhodium carbonyl catalyst $[RhCl(CO)_2]_2$ forms exclusively (instead of the expected diketone–(*E*)-oct-4-ene-3,6-dione) *mono*-substitution product–(*E*)-1-(dimethylphenylsilyl)pent-1-en-3-one, with perfect stereoselectivity and good yield (Scheme 2).

Although *mono*-substitution of (*E*)-1,2-bis(silyl)ethenes containing differently-substituted silyl groups in $AlCl_3$-mediated desilylative



Scheme 2. Rhodium-catalyzed acylation of (*E*)-1,2-bis(dimethylphenylsilyl)ethene by propionic anhydride.

acylation with acyl chlorides has been previously reported [16], the selective acylation of symmetrical (*E*)-1,2-bis(dimethylphenylsilyl) ethene under Narasaka coupling conditions is unprecedented.

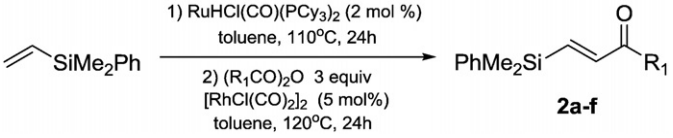
As was previously reported, the silylative coupling of dimethylphenylvinylsilane in the presence of ruthenium hydride catalyst $RuHCl(CO)(PCy_3)_2$ occurred stereoselectively to give (*E*)-1,2-bis (dimethylphenylsilyl)ethene in high yield [18]. The silylative coupling reaction of commercially available dimethylphenylvinylsilane was conducted in toluene, under argon atmosphere in Schlenk bomb flask fitted with a plug valve at 110 °C, to give (*E*)-1,2-bis (dimethylphenylsilyl)ethene **1** as a predominant product (*E*/*Z* = 98/2) after 24 h (GC yield > 99%). Pure compound **1** was isolated by evaporation of toluene and simple bulb-to-bulb distillation from the reaction mixture in 88% yield. Treatment of the isolated compound **1** with 3 equivalents of propionic anhydride in the presence of rhodium catalyst $[RhCl(CO)_2]_2$ (5 mol%) in dry 1,4-dioxane at 90 °C for 24 h under Ar atmosphere according to the method described by Narasaka and co-workers [14] allowed isolation of stereochemically pure (*E*)-1-(dimethylphenylsilyl)pent-1-en-3-one in 68% yield. Similar results were achieved using higher excess of the anhydride and an increase in the amount of propionic anhydride (from 1.5 to 3 equiv. per silyl group) did not affect the selectivity of this process. Thus, by sequencing the highly *E*-selective silylative coupling of dimethylphenylvinylsilane with a stereospecific acylation, the configuration of the product is preserved.

Since a single example of the efficient acylation of (*E*)-dimethylphenyl (4-phenylbut-1-en-1-yl)silane by acetic anhydride in toluene has been reported by Narasaka and co-workers [14], we tested this solvent for the desilylative acylation of bis(silyl)ethene **1**. After several attempts we have found that acylation of **1** by acetic anhydride in the presence of rhodium catalyst in dry toluene occurs more efficiently (90% GC yield) at higher temperature (120 °C) in closed reaction vessel without affecting the selectivity of the process. Since both reactions *i.e.* silylative coupling and desilylative acylation can be performed in toluene, this result prompted us to attempt the acylation step in one pot with silylative coupling without further purification of the (*E*)-1,2-bis(silyl)ethene intermediate. In a typical procedure, dimethylphenylvinylsilane and $RuHCl(CO)(PCy_3)_2$ catalyst (2 mol%) were dissolved in toluene (0.5 M concentration) and heated in Schlenk bomb flask fitted with a plug valve at 110 °C for 24 h under Ar atmosphere. Next, after cooling the reaction to room temperature, 3 equiv. of acetyl anhydride and 5 mol% of solid $[RhCl(CO)_2]_2$ were added and the reaction mixture was heated to 120 °C. Treatment of the silylative coupling product with anhydride caused mono-acylation in a stereospecific manner giving β-(dimethylphenylsilyl)-α,β-unsaturated ketones in high geometrical purity (*E*/*Z* = 97/3–99/1) within 24 h. Column chromatography of the resulting products (silica gel, eluent: hexane/diethyl ether 25:1) afforded pure ketones **2a**–**f** in 48–82% overall yield (Table 1). Unfortunately, when crotonic anhydride was applied as acylating agent, no reaction took place.

It has been found that one-pot sequential silylative coupling–desilylative acylation protocol can be also applied to the synthesis of (*E*)-β-(trimethylsilyl)-α,β-unsaturated ketones. Since silylative coupling of trimethylvinylsilane in the presence of ruthenium hydride catalyst $RuHCl(CO)(PCy_3)_2$ yielded a mixture of isomeric bis(silyl) ethenes, *i.e.* (*E*)-1,2-bis(trimethylsilyl)ethene and 1,1-bis(trimethylsilyl) ethene (*E*/*gem* = 9:1), the homo-coupling of trimethylvinylsilane was performed in the presence of $[RuCl_2(CO)_3]_2$ complex which has been previously reported as an efficient catalyst for the selective formation of (*E*)-1,2-bis(trimethylsilyl)ethene [19]. Thus, silylative coupling of trimethylvinylsilane was conducted in toluene (0.5 M concentration), in the presence of $[RuCl_2(CO)_3]_2$ (1 mol%) under argon atmosphere in a Schlenk bomb flask fitted with a plug valve at 110 °C, to give (*E*)-1,2-bis(trimethylsilyl)ethene after 48 h (GC yield 96%). Then, after cooling the reaction mixture to room temperature, 3 equiv. of acetyl anhydride and 5 mol% of solid $[RhCl(CO)_2]_2$ were added and the reaction mixture was heated to 120 °C. Treatment of (*E*)-1,2-bis(trimethylsilyl) ethene with acetic and propionic anhydrides caused mono-acylation in a stereospecific manner giving β-(trimethylsilyl)-α,β-unsaturated ketones in high geometrical purity (*E*/*Z* = 99/1) and good yield (80–85% measured by GC–MS) within 24 h (Scheme 3).

Efforts were made to further improve the procedure by examining tandem catalysis using one catalyst for both silylative coupling and acylation steps. Unfortunately, the silylative coupling of dimethylphenylvinylsilane in the presence of $[RhCl(CO)_2]_2$ was unsuccessful. Other rhodium precursors which are known to be active in the silylative coupling reaction, *e.g.*, $[RhCl(cod)]_2$, $[Rh(OSiMe_3)(cod)]_2$, and $RhH(CO)(PPh_3)_3$, also failed as potential Narasaka acylation catalysts.

Table 1
One-pot synthesis of (*E*)-β-(dimethylphenylsilyl)-α,β-unsaturated ketones from dimethylphenylvinylsilane.

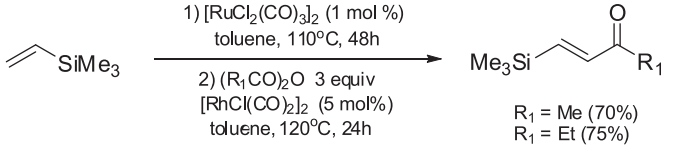


Compound	R_1	Yield (%)[a]	*E*/*Z*
2a	Me	68	99/1
2b	Et	82	99/1
2c	*n*-Pr	54	97/3
2d	*i*-Pr	60	98/2
2e	Ph	48	98/2
2f	$-C(=CH_2)Me$	70	98/2
2g	(*E*)-MeCH=CH–	0	–

Silylative coupling conditions: $[CH_2=CHSiMe_2Ph]:[RuHCl(CO)(PCy_3)_2] = 1:0.02$; toluene (0.5 M), 110 °C, 24 h.
Narasaka coupling conditions:$[CH_2=CHSiMe_2Ph]:[(R_1CO)_2O]:[[RhCl(CO)_2]_2] = 1:3:0.05$, toluene, 120 °C, 24 h.
[a] Isolated yields of products.

4. Conclusion

In conclusion, we have developed a new one-pot protocol for stereoselective preparation of (*E*)-β-silyl-α,β-unsaturated ketones *via* a highly selective catalytic silylative coupling/desilylative acylation sequence. Starting from easily available dimethylphenylvinylsilane or trimethylvinylsilane and carboxylic acid anhydrides the corresponding unsaturated β-silylketones are obtained in generally good yields and selectivities.



Scheme 3. One-pot synthesis of (*E*)-β-(trimethylsilyl)-α,β-unsaturated ketones from trimethylvinylsilane.

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