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COMMUNICATION

Nickel-Catalyzed Decarboxylative C–Si Bond Formation: A Regioselective Cross-Coupling Between Trialkyl Silanes and α , β -Unsaturated Carboxylic Acids

Bharat Kumar Allam* | Sadaf Azeez* | Jeyakumar Kandasamy 🕩

Department of Chemistry, Indian Institute of Technology (BHU), Varanasi 221 005Uttar Pradesh, India

Correspondence

Email: jeyakumar.chy@iitbhu.ac.in

This report presents the first example of nickel-catalyzed mild decarboxylative cross-coupling reaction for the regioselective formation of C–Si bond. An easily accessible and significantly stable Ni $(dmg)_2$ owes the role of key promoter. This reaction is highly functional group tolerant and offers α,β -unsaturated silanes in synthetically useful yields. The reaction gives access to the successful utilization of otherwise difficult trialkyl silanes as coupling partners and operates at a moderate temperature, which is beneficial to deal with highly volatile silanes.

KEYWORDS

carboxylic acids, decarboxylation, nickel, silicon, synthetic methods

1 | INTRODUCTION

Silicon is the most abundant element in nature.^[1] It is often a major constituent element in sand and found only in traces in biological systems. Some living organisms can engineer their shell structures by utilizing silicaprecipitating proteins.^[2] Trypsin is reported to show promiscuous activity in the metabolism of alkoxysilanes. The family of silicate in enzymes from marine sponges is unique for catalyzing the formation of organosiloxane structures.^[3] Despite the limited availability of organosilicon compounds in living systems, synthetic chemistry has provided us straightforward access. The organic compounds containing silicon are important constituents for molecules of chemistry and material science interest.^[4] Silicon is a carbon isostere, the biocompatibility of silicon can be used to optimize and reconstituting of the pharmacology of bioactive molecules.^[5] The silicon supply in nature is more abundant, but the sustainable methods for synthesizing organosilicon compounds are limited. The affinity of silicon towards carbon to form a carbon-silicon (C-Si) bond is an interesting research

problem since the fascinating chemistry was introduced by the landmark report of Friedel and Crafts.^[6] The formation of C–Si bond has been reached to the extensive levels of sophistication by the efforts of Grubbs and Stolz,^[7] Hiyama,^[8] Hartwig,^[9] Oestreich,^[10] Huang,^[11] and Arnold.^[12]

The alkenylsilanes are important synthons that are frequently utilized in various synthetic transformations.^[13] They are key coupling partners in Hiyama coupling, Hiyama-Denmark coupling, and silvl-Heck reactions.^[14] Stork described alkenylsilanes as carbonyl precursors in annelation reactions.^[15] The reported methods for the synthesis of alkenylsilanes involves the reaction of chlorotriorganosilanes with organolithium/organoreagents,^[16] magnesium silyl-Heck reaction of halosilanes,^[17] hydrosilylation and silylmetalation of alkynes,^[18] and dehydrogenative silylation of alkenes.^[19] However, they require low temperatures, prolonged reaction times, lack of regio- and stereoselectivity, requirement of precious transition metal catalysts, generation of large quantity of halogenated waste, use of a huge excess of activated olefins, formation of polymeric side products, and moderate yields of trialkyl (alkenyl)silanes. So, the development of highly regioselective methods for

*Bharat Kumar Allam and Sadaf Azeez contributed equally to this work.

the synthesis of trialkyl (alkenyl/alkynyl)silanes using sustainable and economic starting materials are highly valuable and in constant demand.

Carboxylic acids are the naturally prevalent functional groups found in organic molecules and serve as adaptable synthetic building blocks. Through the extrusion of CO₂, carboxylic groups can act as traceless reactivity guiding functionalities to construct simple to complex molecules. Gooßen and MacMillan most extensively explored the decarboxylative functionalizations for C-C bond formation.^[20] On the other hand, the decarboxylative C-X bond formation currently limited to the C–N,^[21] C–P,^[22] C–S,^[23] C-F,^[24] C-Br,^[25] and C-I^[26] bond creation. Particularly, the formation of C-Si bond, an analogues bond to the C-C bond, using decarboxylative functionalization has not been studied well.^[27] In this context, Liu et al. explored a copper-catalyzed decarboxylative C-Si bond formation between unsaturated carboxylic acids and silanes, for the first time.^{27a} In fact, it is the only method so far available in the literature for C-Si bond formation between unsaturated carboxylic acids and silanes. Hence, we believed that there is enough room for the development of mild, selective, economical and high yielding methods for the C-Si bond formation using sustainable and cheap silane feed stocks. Nickel is an indispensable metal in organic synthesis which is explored in many decarboxylative crosscoupling reactions.^[28] In this context, Rueping et al.^{27b} and Shi et al.^{27c} have recently demonstrated a nickel promoted decarbonylative silvlation of aryl esters. However, to best of knowledge, a decarboxylative C-Si bond formation between unsaturated acids and silanes is not explored with nickel catalysts.

Given all the above facts, here we have developed a nickel catalyzed decarboxylative silyl functionalization strategy *via* the reaction of trialkyl silanes with (*E*)-cinnamic acids/alkynoic acids, to deliver trialkyl (alkenyl/alkynyl)silanes (Scheme 1).

2 | EXPERIMENTAL SECTION

2.1 | General experimental information

Proton and carbon magnetic resonance spectra were recorded on a Bruker Avance 500 MHz NMR (¹H NMR

at 500 MHz, ¹³C NMR at 125 MHz). Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual deuterium in the solvent (¹H NMR: CDCl₃ at 7.26 ppm). Chemical shits for carbons are reported in parts per million downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent peak (¹³C NMR: CDCl₃ at 77.0 ppm). NMR data are represented as follows: chemical shift, multiplicity (s = singlet, d = doubletand m = multiplet), coupling constant (J) (Hz), and integration. HRMS was determined using Agilent 6530 Accurate-Mass Q-TOF instrument. Analytical thin layer chromatography (TLC) was performed on Merck Kieselgel 60 GF254 plates (thickness 0.25 mm). Visualization was performed with a 254 nm UV lamp and by staining in I2 chamber. Organic solutions were concentrated under reduced pressure using a Büchi rotary evaporator. Purification of the crude products was carried out by column chromatography using silica gel 100-200 mesh. All the reactions were carried out under an open atmosphere using oven-dried glassware. Yield refers to the isolated analytically pure material (See Supporting Information).

2.2 | General experimental procedure

Under air a mixture of α , β -unsaturated carboxylic acid (2 mmol), trialkyl silane (5 mmol), Ni (dmg)₂ (20 mol%), TBHP (6 mmol) were taken in a 10 ml glass vial contain a Teflon coated magnetic stirr bar and the reaction mixture was further heated up to 60 °C for 12 hr. After completion of the reaction (TLC) water was added. The reaction mixture was filtered and partitioned in between water and ethyl acetate. The organic layer was dried over Na₂SO₄, evaporated by using a rotary evaporator. The residue thus obtained was further subjected to silica gel column chromatography using a mixture of ethyl acetate and hexane as an eluent (See supporting Information).

3 | **RESULTS AND DISCUSSION**

To establish optimized reaction conditions, we initially screened a reaction between (*E*)-cinnamic acid (1a) and triethylsilane (2a) using 10 mol% of Ni (dmg)₂, 100 mol% of TBHP at 60 °C under solvent-free conditions (Table 1).



SCHEME 1 Decarboxylative C–Si bond formation.

TABLE 1 Optimization of various parameters for decarboxylative silylation^a

		Et DH + H—Si—Et	Catalyst	Et Si Et	
	1a	Ét 2a	T °C, 12 h 3	a	
S. No	Catalyst (mol%)	Accelerator (mol%)	T(°C)	Solvent	Yield (%) ^b
1.	Ni (dmg) ₂ (10)	TBHP (100)	60 °C	-	45
2.	Ni (dmg) ₂ (10)	TBHP (200)	60 °C	-	53
3.	Ni (dmg) ₂ (10)	TBHP (300)	60 °C	-	60
4.	Ni (dmg) ₂ (10)	TBHP (400)	60 °C	-	40
5.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	-	87 (42) ^c
6.	Ni (dmg) ₂ (30)	TBHP (300)	60 °C	-	82
7.	Ni (dmg) ₂ (20)	TBHP (300)	40 °C	_	50
8.	Ni (dmg) ₂ (20)	TBHP (300)	rt	-	trace
9.	Ni (dmg) ₂ (20)	TBHP (300)	80 °C	-	80
10.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	CH ₃ CN	55
11.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	^t BuOH	79
12.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	DCE	71
13.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	1,4-dioxane	65
14.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	DMSO	50
15.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	DMF	32
16.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	THF	60
17.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	H ₂ O	45
18.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	C_6H_6	80
19.	Ni (dmg) ₂ (20)	TBHP (300)	60 °C	Toluene	57

^a**1a** (2 mmol), **2a** (5 mmol), for 12 hr.

^bIsolated yield after column chromatography.

^cReaction conducted with 70% TBHP (in water).

The reason behind the selection of nickel (II) dimethylglyoxime complex as catalyst was that, Ni $(dmg)_2$ is a bench stable compound which is most commonly available in research laboratories. Ni (dmg)₂ is often used for electrode surface modifications, as catalyst for oxygen reduction, and as an active site to promote hydrogen generation.^[29] However, Ni (dmg)₂ is less explored as catalysts for synthetic organic transformations.^[30] Hence, we have initially assessed the catalytic efficiency of Ni (dmg)₂ in C-Si bond forming reaction. To our delight, we observed the formation of (E)-triethyl (styryl)silane (3a) in 45% yield (entry 1). Later we raised the mol% of TBHP to 300, which provided 53% yield of the product (entry 2). But a further variation of TBHP concentration to 400 mol% did not provided a higher yield, surprisingly it diminished the product yield (entry 3). As an obvious alternative, we increased the Ni (dmg)₂ to 20 mol%, which provided a maximum yield of 87% at 60 °C (entry 5). However further increase of catalyst mol% slightly diminished the reaction yield (entry 6). Also, altering the reaction temperature from 60 °C, delivered the desired product in diminished yield (entries 7 & 9). The reaction provided only a trace of product at room temperature (entry 8). Various polar and non-polar solvents, Viz., CH₃CN, ^tBuOH, DCE, 1,4-dioxane, DMSO, DMF, THF, H₂O, C₆H₆, and toluene were screened for their proficiency, and none match the efficiency of solvent-free conditions (entries 10-19). Various salts of nickel, other metals, metal-free conditions, and accelerators were screened, and found that Ni $(dmg)_2$ and TBHP combination is the best (See the supporting information). It was interesting to mention here that 20 mol% BMIM[BF₄] along with 300 mol% TBHP promoted the reaction and delivered the product in 83% yield (Cf. Supporting information).

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After all, the combination of Ni $(dmg)_2$ and TBHP was crucial and necessary for the decarboxylative silylation

reaction. The optimized reaction conditions [20 mol% Ni $(dmg)_2$, 3 equivalents of TBHP at 60 °C for 12 hr under solvent-free conditions], proved to apply to a wide range of cinnamic/alkynoic acids in combination with trialky-lsilanes having linear or branched alkyl chains (Table 2).

Initially, the reaction was tested between triethylsilane and cinnamic acids bearing electron-donating and withdrawing groups under optimized conditions. It was observed that cinnamic acids having electron-donating groups such as methyl and methoxy, gave the desired products in excellent yields, *i.e.* 82–90% (Table 2, **3b-3d**). Nevertheless, the electron-withdrawing groups such as nitro, fluoro and chloro functionalized cinnamic acids were also participated in these coupling reactions with similar reactivity and provided the desired products in comparable yields (Table 2, **3e-3 g**). However, the *ortho*substituent on the cinnamic acids has a profound effect on lowering the reaction yield, perhaps, due to the steric hindrance. For instance, the installation of methyl or chloro substituent at ortho position of cinnamic acid lead the formation of desired products in slightly lower yields (Table 2, 3 h-3j). It is worth noting that a highly sensitive thiophene or furan containing α,β -unsaturated carboxylic acids were also fruitfully utilized as coupling partners (Table 2, 3 k-3 l). Similar to cinnamic acids, a decarboxylative coupling of naphthyl acrylic acid with triethylsilane was successfully accomplished under optimized condition in 73% yield. It is also interesting to note that the hydroxyl group present on the substrate found to be intact during the coupling reaction, which demonstrate the mildness of the reaction conditions. Having explored the substrate scope with different cinnamic acids, coupling reactions were investigated with different silanes. To our delight, similar to triethylsilane, tri-

TABLE 2 The substrate scope for the decarboxylative C-Si bond formation^{a,b}



^a1a (2 mmol), 2a (5 mmol), Ni (dmg)₂ (20 mol%), TBHP (decane) (6 mmol) heated up to 60 °C for 12 hr.
 ^bIsolated yield after column chromatography.

isopropyl and dimethylphenyl silanes also participated in the coupling reactions with different cinnamic acids providing the desired products **3n-3v** in 46–84% yields.

Similar to cinnamic acids, alkynoic acids also partook as excellent coupling partners in the decarboxylative coupling reactions with different linear and branched trialkylsilanes under optimized conditions. These reactions provided the desired products 3w-3y in 65–85% yields. To our surprise coupling of triphenylsilane with cinnamic acids was resulted in no yield while with 3-phenylpropiolic acid provided 65% yield. It was quite unfortunate to report that all our efforts to utilize trialkylgermanes as coupling partners failed. Over all, under optimized condition various functional groups, such as nitro, ether, halo, and hydroxy were well tolerated in these reactions.

Control experiments were conducted to further understand and envision the reaction mechanism using cinnamic acid (1a) and triethylsilane (2a) (See the supporting information). The free radical trapping reagents BHT and TEMPO have completely seized the reaction indicating a radical type reaction mechanism. A blank experiment was undertaken with triethylsilane in the absence of cinnamic acid (1a) under standard reaction conditions, and we did not find the formation of 1,1,1,2,2,2-hexaethyldisilane indicating the possibility of forming a nickel (III)Si complex which efficiently transfers the triethylsilyl group to the unsaturated carboxylic acid. We have also conducted a reaction between cinnamic acid and 1.1.1.2.2.2-hexaethyldisilane under standard reaction conditions, which only provided a trace amount of product. To identify the role of light, the reaction was investigated in dark and isolated the desired product (3a) in 85% yield. It suggests that the light have no significant effect on the reaction outcome/vield. Further, we have investigated the reaction under inert (N_2) atmosphere. This reaction provides the desired product



SCHEME 2 Plausible reaction mechanism.

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in 75% yield suggesting that there is only a minimum effect of oxygen in the reaction.

Based on the above control experiments and reported literatures,^{27a, 31} a plausible mechanistic pathway is outlined in Scheme 2. We believe that the reaction proceeds through a Ni (III)Si complex (I) formed from the reaction of Et_3SiH (2a) with Ni (dmg)₂ in the presence of TBHP. The Ni (III)Si complex efficiently delivers trialkyl silyl radical, which undergo addition to cinnamic acid double bond and provides the intermediate II. Further, the intermediate (II) undergoes a decarboxylation via intermediate (II) in the presence of hydroxyl ion to deliver the desired (*E*)-trialkyl (alkenyl)silanes.^{27a}

4 | CONCLUSIONS

In conclusion, we have a developed a nickel catalyzed decarboxylative silylation strategy under mild conditions. The application of dmg as a ligand tunes the Lewis acidity of the nickel and safeguard the α , β -unsaturated silanes from further nickel catalyzed C–Si bond cleavage/ decomposition. The method efficiently used the otherwise difficult and less reactive trialkyl silanes to deliver trialkyl (alkenyl/alkynyl)silanes. This method opens windows to explore various transformations using aliphatic silanes. Further studies to expand the scope of chemistry with trialkylgermanes using various metal complexes are underway in our laboratory.

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ORCID

Jeyakumar Kandasamy D https://orcid.org/0000-0003-3285-971X

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

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