

### Alkyne Hydrosilylation

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# **Cobalt-Catalyzed Z-Selective Hydrosilylation of Terminal Alkynes**

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**Abstract:** A cobalt-catalyzed Z-selective hydrosilylation of alkynes has been developed relying on catalysts generated from bench-stable  $Co(OAc)_2$  and pyridine-2,6-diimine (PDI) ligands. A variety of functionalized aromatic and aliphatic alkynes undergo this transformation, yielding Z-vinylsilanes in high yields with excellent selectivities (Z/E ratio ranges from 90:10 to > 99:1). The addition of a catalytic amount of phenol effectively suppressed the Z/E-isomerization of the Z-vinyl-silanes that formed under catalytic conditions.

ransition-metal-catalyzed hydrosilylation of alkynes is the most straightforward and atom-economical approach to prepare synthetically valuable vinylsilanes.<sup>[1]</sup> The major issue in the hydrosilylation of alkynes is the control of the regio- and stereoselectivity because this reaction can afford multiple products. For example, the hydrosilylation of terminal alkynes can produce  $\alpha$ -, (E)- $\beta$ -, and (Z)- $\beta$ -vinylsilanes. Among these isomeric vinylsilanes, (E)- $\beta$ -vinylsilanes are usually produced with high selectivity in most of transitionmetal-catalyzed alkyne hydrosilylation reactions.<sup>[2]</sup> However, the stereoselective formation of (Z)- $\beta$ -vinylsilanes still remains highly challenging for the hydrosilylation of terminal alkynes. Furthermore, the isomerization of Z-vinylsilanes to thermodynamically favorable E-vinylsilanes under conditions for transition-metal-catalyzed alkyne hydrosilylation poses extra difficulty to the development of the Z-selective alkyne hydrosilylation.<sup>[3]</sup>

There are a few catalysts that can produce Z-vinylsilanes through hydrosilylation of terminal alkynes, but catalysts for this transformation have been limited to noble metals such as Ru,<sup>[4]</sup> Rh,<sup>[5]</sup> and Ir.<sup>[3,6]</sup> In addition, these catalysts show either low reactivity or substrate-dependent selectivity and often produce a mixture of  $\alpha$ -, (*E*)- $\beta$ -, and (*Z*)- $\beta$ -vinylsilanes. An iron catalyst has been studied for the *Z*-selective hydrosilylation of alkynes,<sup>[7]</sup> but the scope of alkynes has not been well established and only two terminal alkynes were reported for this reaction. Therefore, it is highly desirable to develop a base metal catalyst that can effectively catalyze *Z*-selective hydrosilylation of a wide range of functionalized terminal alkynes.

Recently, cobalt complexes have been emerging as active catalysts for hydrogenation,<sup>[8]</sup> hydroboration,<sup>[9]</sup> and hydrosilylation<sup>[10]</sup> of alkenes and alkynes. The cobalt-catalyzed

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hydrosilylation of terminal alkynes has also been reported and these reactions selectively produce either  $\alpha$ - or (*E*)- $\beta$ vinylsilanes (Scheme 1 a,b).<sup>[11]</sup> However, the Co-catalyzed alkyne hydrosilylation that can selectively produce (*Z*)- $\beta$ vinylsilanes still remains unknown. Very recently, we have shown that the combination of Co(acac)<sub>2</sub> and a pyridine-2,6diimine ligand selectively catalyzes Markovnikov hydrosilylation of aliphatic alkenes.<sup>[12]</sup> In this study, we tested this catalytic system for alkyne hydrosilylation. Herein, we report the first Co-catalyzed *Z*-selective anti-Markovnikov hydrosilylation of terminal aromatic and aliphatic alkynes (Scheme 1 c). Furthermore, we found that the addition of phenol can effectively suppress the isomerization of *Z*-vinylsilanes that would result in the thermodynamically more stable *E*-vinylsilanes after full conversion of alkynes.



**Scheme 1.** Cobalt-catalyzed hydrosilylation of terminal alkynes.

We chose the reaction of phenylacetylene with PhSiH<sub>3</sub> to evaluate the reaction conditions. Cobalt catalysts were generated from bench-stable Co(acac)<sub>2</sub> or Co(OAc)<sub>2</sub> and pyridine-2,6-diimine ligands and activated in situ by the reaction with PhSiH<sub>3</sub>. Reactions were conducted with 1 mol% cobalt catalyst at room temperature with a 1:1 ratio of phenylacetylene and PhSiH<sub>3</sub>. Selected experiments are summarized in Table 1. The reaction catalyzed by Co(acac)<sub>2</sub> and <sup>mes</sup>PDI (L1) proceeded to modest conversion in 12 h and gave the vinylsilane (Z)-1a in 78% GC yield with good selectivity (98%, entry 1). Extending the reaction time to 24 h did not significantly improve the reaction (entry 2). However, the reaction conducted with Co(OAc)<sub>2</sub>/L1 occurred in full conversion in 12 h, but afforded a mixture of Z/E-isomers with a ratio of 65:34 (entry 3). Considering the potential isomerization of Z-vinylsilanes to E-vinylsilanes, we followed the selectivity of the reaction by GC analysis and found that the reaction selectively afforded (Z)-1a before phenylacetylene was fully consumed. Subsequently, the isomerization of

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**Table 1:** Evaluation of conditions for the Co-catalyzed hydrosilylation of phenylacetylene.<sup>[a]</sup>



[a] Reaction conditions: phenylacetylene (0.500 mmol), PhSiH<sub>3</sub>
(0.500 mmol), Co(OAc)<sub>2</sub> (5.0 μmol), ligand (5.0 μmol), solvent (1 mL).
[b] Determined by GC analysis with dodecane as the internal standard.
[c] Co(acac)<sub>2</sub> (5.0 μmol).

(Z)-1a to (E)-1a occurred when the reaction was not quenched after full conversion of phenylacetylene was reached. These results indicate that this Co-catalyzed alkyne hydrosilylation is Z-selective and the E-vinylsilane is generated via the isomerization of the Z-vinylsilane. For this reaction, it took 4.5 h to reach almost full conversion and (Z)-1a was formed selectively (entry 4).

As the time needed to achieve full conversion of alkynes is substrate-dependent, it is more desirable to develop a general procedure for the hydrosilylation of a variety of alkyne substrates. As such, we need to identify an additive that can suppress the Z/E-isomerization after the full conversion of alkynes is achieved. For this reason, we tested the hydrosilylation (reaction time: 48 h) with a variety of terminal alkynes that contain various functional groups. We found that the reaction of 3-ethynylphenol afforded the corresponding Z-vinylsilane with >99% selectivity [Eq. (1)]. This result suggested that addition of phenol could possibly suppress the Z/E-isomerization of Z-vinylsilanes. To test this, we subsequently conducted the hydrosilylation of phenol (see the Support-



ing Information). The reaction with the addition of  $10 \mod \%$  phenol afforded (Z)-1a in 95% yield with 98% selectivity (entry 5).

To determine the parameter that governs the regioselectivity of alkyne hydrosilylation, we conducted this Cocatalyzed hydrosilylation of phenylacetylene (entries 5–11 in Table 1) in the presence of a series of sterically diverse PDI ligands **L1–L3** and **L7**, oxazoline iminopyridine ligands **L4** and **L5**, and 'Pr-PyBox ligand **L6** (**L4–L6** have been used for Co-catalyzed  $\alpha$ -hydrosilylation of alkynes<sup>[11b,c]</sup>). The results of these experiments indicate that the steric properties of the ligands significantly influence the regioselectivity of the reaction, with sterically less hindered ligands (**L3–L6**: entries 7–10) favoring  $\alpha$ -selectivity and sterically more congested ligands (**L1** and **L7**: entries 5 and 11) favoring (*Z*)- $\beta$ selectivity (see the Supporting Information for a detailed discussion).

The scope of aromatic alkynes that undergo this Cocatalyzed Z-selective hydrosilylation is summarized in Table 2. In general, a wide range of ethynylarenes containing



[a] Reaction conditions: alkyne (0.500 mmol), PhSiH<sub>3</sub> (0.500 mmol), Co(OAc)<sub>2</sub> (5.0  $\mu$ mol), <sup>mes</sup>PDI (5.0  $\mu$ mol), phenol (0.050 mmol), THF (1 mL), RT, 6 h; yield of isolated product. [b] 50°C. [c] 3 mol% catalyst, phenol (0.150 mmol). [d] (L1)CoMe (5.0  $\mu$ mol), 1,4-dioxane (1 mL), 80°C, 6 h.

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A)

electronically and sterically varied aryl groups reacted smoothly in the presence of  $1 \mod \%$  of  $Co(OAc)_2$  and mesPDI (L1) at room temperature, affording the corresponding Z-vinylsilanes (1a-1ab) in high yields with excellent stereoselectivities (Z/E = 96:4 to > 99:1).

This Z-selective alkyne hydrosilylation shows good functional group tolerance and a wide range of reactive groups, such as ether (1d, 1i, and 1k), trifluoromethyl ether (1e), halogen (11-1p), unprotected phenolic and alcoholic hydroxyl (1q and 1r), unprotected primary aniline (1s and 1t), ketone (1u), ester (1v), amide (1w), aldehyde (1x), and imine (1y) moieties, are compatible with the reaction conditions. In addition, nitrogen- and sulfur-containing heteroaromatic alkynes (1z, 1aa, and 1ab) reacted in good yields and excellent selectivities. However, ethynylarenes containing nitro or carboxylic acid moieties do not react under the identified conditions. In addition, the alkyne hydrosilylation was also tested with secondary hydrosilanes and the hydrosilvlation of 2-ethynylanisole with  $PhSiH_2R$  (R = Me, Ph) afforded the corresponding Z-vinylsilanes (1ac and 1ad) in good yields and high selectivity. The results of hydrosilylation reactions on a 5 mmol scale conducted without using a dry box as well as further transformations of Z-vinylsilanes are included in the Supporting Information.

Table 3 summarized the scope of aliphatic alkynes for this Co-catalyzed Z-selective hydrosilylation (see the Supporting Information for the evaluation of the reaction conditions). These reactions were conducted with 3 mol% catalyst under slightly modified conditions with <sup>iPr</sup>PDI ligand (L7 in Table 1) at 70°C. Z-Vinylsilanes (2a-21) were obtained in modest to high yields with good to excellent selectivities. A variety of functional groups, such as silvlether (2g), cyano (2h), chloride (2i), bromide (2j), ketone (2k), and ester (2l), were tolerated. Aliphatic alkynes containing aldehyde, aliphatic alcohol, and nitro moieties do not react under the identified conditions.

Table 3: Scope of aliphatic alkynes.<sup>[a]</sup>



[a] Reaction conditions: alkyne (0.500 mmol), PhSiH<sub>3</sub> (0.500 mmol), Co(OAc)<sub>2</sub> (15.0 µmol), <sup>iPr</sup>PDI (15.0 µmol), phenol (0.150 mmol), THF (1 mL), 70°C; yield of isolated product.

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For a preliminary understanding of this Co-catalyzed Z-

selective alkyne hydrosilylation, we conducted a series of

deuterium-labeling experiments (Scheme 2). Hydrosilylation

of 4-tert-butylphenylacetylene- $d_1$  and 1-octyne- $d_1$  with PhSiH<sub>3</sub>

in the presence of phenol proceeded with excellent stereose-

lectivity (Z/E = 99:1, Scheme 2 A,B) and afforded vinylsilane



Scheme 2. Deuterium-labeling experiments.

The kinetic behavior was assessed for phenylacetylene hydrosilylation with PhSiH<sub>3</sub> catalyzed by (<sup>mes</sup>PDI)CoMe in the presence of phenol at room temperature. The plots of the initial rate of the reaction vs. the concentration of (mesPDI)CoMe, PhSiH<sub>3</sub>, and phenylacetylene are shown in Figures S1-S3 in the Supporting Information. The kinetic analysis showed that this catalytic reaction is first order in the cobalt catalyst and zero order in phenylacetylene and silane.

Based on the results of the deuterium-labeling experiments and the kinetic experiments and the precedent for the cobalt-catalyzed hydrosilylation and hydrogenation of alkenes and alkynes, we proposed a silvlmetalation pathway with a Co<sup>I</sup> silvl intermediate for this Co-catalyzed Z-selective alkyne hydrosilylation (Scheme 3).<sup>[13]</sup> Migratory insertion of the alkyne into the Co-Si bond forms a cobalt vinyl species (Z)-I, followed by the rate-limiting isomerization to a cobalt vinyl species (E)-I through a zwitterionic or metallacyclopropene intermediate.<sup>[14]</sup> Subsequent turnover with PhSiH<sub>3</sub> releases Z-vinylsilane and regenerates the Co-silyl species. The relief of steric repulsion between the ligand and the silyl group in (Z)-I may account for the isomerization of the cobalt species (Z)-I to (E)-I.

We subsequently studied the Z/E-isomerization of (Z)-1a under two sets of conditions that can generate cobalt hydride

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Scheme 3. Proposed catalytic cycle for this Z-selective hydrosilylation.



species because cobalt hydride species have been shown to catalyze the isomerization of alkenes.<sup>[8c,15]</sup> Indeed, the Z/E-isomerization of (Z)-1a occurred under both sets of conditions [Eq. (2)]. The isomerization of aliphatic vinylsilane (Z)-2b was also tested [Eq. (3)]. In addition to the Z/E-isomer-



ization, the formation of *E*-allylsilane was detected. These results suggest the *Z*/*E*-isomerization of *Z*-vinylsilanes occurs via alkene insertion into the Co–H bond followed by  $\beta$ -H elimination.<sup>[8c,15]</sup>

As the addition of phenol can effectively suppress the isomerization of Z-vinylsilanes, we propose that phenol can quench the Co-H species generated from the reaction mixture after the alkyne substrates are fully consumed. To test this hypothesis, we conducted the reaction of 4-methoxyphenol with the Co-H species "(mesPDI)CoH", generated in situ from the reaction of (mesPDI)CoMe with 1 equivalent of pinacolborane (HBpin), and this reaction formed a cobalt phenolate complex (<sup>mes</sup>PDI)Co(OC<sub>6</sub>H<sub>4</sub>-4-OMe) (3).<sup>[16]</sup> Alternatively, compound 3 can be prepared by the reaction of (mesPDI)CoMe with 4-methoxyphenol [Eq. (4)]. As a reference reaction, the addition of 4-methoxyphenol can also suppress the Z/E-isomerization of Z-vinylsilane [Eq. (5)]. With complex 3 in hand, we tested it as a cobalt precursor for the hydrosilylation of phenylacetylene with PhSiH<sub>3</sub> and found that it can effectively catalyze this hydrosilylation reaction with high Z-selectivity [Eq. (6)]. As expected, compound 3 does not catalyze the Z/E-isomerization of Z-vinylsilanes. These results suggest that phenol suppresses the isomerization of Z-vinylsilanes by quenching the Co–H species to form a cobalt phenolate and is not directly involved in the catalytic production of Z-vinylsilanes.

In summary, we have developed the first Co-catalyzed Z-selective anti-Markovnikov hydrosilylation of terminal alkynes with catalysts generated from bench-stable Co(OAc)<sub>2</sub> and pyridine-2,6-diimine ligands and activated in situ by the reaction with PhSiH<sub>3</sub>. A broad range of ethynylarenes and aliphatic alkynes underwent this reaction to afford (Z)- $\beta$ -vinylsilanes in high yields with excellent stereoselectivities. The addition of a catalytic amount of phenol is crucial to achieve high Z-selectivities because phenol suppresses the isomerization of Z-vinylsilane products that would form *E*-vinylsilanes after the full conversion of alkynes has been achieved. Further studies to determine the detailed mechanism of this transformation and to apply this transformation to the synthesis of complex molecules will be the subject of further work.

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#### Conflict of interest

The authors declare no conflict of interest.

**Keywords:** alkynes · cobalt · homogeneous catalysis · hydrosilylation · vinylsilanes

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which was generated from bench-stable  $Co(OAc)_2$  and a pyridine-2,6-diimine derivative and activated in situ by PhSiH<sub>3</sub>, promotes the highly regio- and Z-selective hydrosilylation of terminal aromatic

and aliphatic alkynes. This reaction tolerates a variety of functional groups such as halide, aldehyde, ketone, ester, amide, cyano, and free hydroxy and amine functionalities.

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