Supporting Information

Iron Catalyzed Synthesis of (E)-β-Vinylsilanes via a Regio and Stereoselective Hydrosilylation from Terminal Alkynes

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1 General Information

Chromatography

Thin layer chromatography (TLC) was performed on Huanghai pre-coated glass-backed TLC plates and visualized by UV lamp (254 nm). Column chromatography on silica gel (300-400 mesh) was carried out using technical grade 60-90 °C petroleum ether (distilled prior to use) and analytical grade EtOAc (without further purification). Concentration under reduced pressure was performed by rotary evaporation. Purified compounds were further addressed under high vacuum (3-5 mmHg). Yields refer to chromatographically purified compounds.

Nuclear magnetic resonance spectra

$^1$H and $^{13}$C spectra were recorded on Bruker AV 400MHz and Bruker AV 500 MHz spectrometer. Chemical shifts were reported in ppm. $^1$H NMR spectra were referenced to CDCl$_3$ (7.28 ppm), and $^{13}$C-NMR spectra were referenced to CDCl$_3$ (77.0 ppm). All $^{13}$C-NMR spectra were measured with complete proton decoupling. Peak multiplicities were designated by the following abbreviations: s, singlet; d, doublet; t, triplet; m, multiplet and J, coupling constant in Hz.

IR spectra and Mass spectroscopy

IR spectra were recorded on a Nicolet AVATER FTIR360 spectrometer as a thin film. Absorptions were given in wavenumbers (cm$^{-1}$). Mass spectroscopy: HRMS spectra were recorded with Micromass QTOF2 Quadrupole/Time-of-Flight Tandem mass spectrometer using electron sprayionization.
2 General Procedure for the Synthesis of (E)-β-Vinylsilanes 3

In a nitrogen filled schlenk tube, FeCl₂ (2 mol%), Xantphos (2 mol%), NaBHEt₃ (4 mol%) and ether (2 mL) were added and the mixture was stirred at 50 °C for 10 minutes, then alkyne (1.0 mmol) and phenylsilane (1.1 mmol) were added under N₂. The reaction mixture was stirred at 50 °C. Upon completion, the solvent was removed by vacuum and the crude residue was purified by silica gel column chromatography to afford the corresponding products 3 (eluent: petroleum ether/EtOAc = 100/1).

Table S1. Scope of terminal alkynes for the Fe/Xantphos catalysed anti-Markovnikov hydrosilylation with PhSiH₃

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- Reaction conditions: 1a (1.0 mmol), PhSiH₃ (1.1 mmol), Et₂O (2 mL), FeCl₂ (2.0 mol%), Xantphos (2.0 mol%), NaBHEt₃ (4 mol%), 12 h, 50 °C in a schlenk tube.
- Yields of isolated products.
- The selectivity for product was determined by 1H NMR spectroscopy.
3 $^1$H NMR and $^{13}$C NMR Data of the Products

$(E)$-but-1-en-1-yl(phenyl)silane (3a):

![SiH$_2$Ph]

Colorless liquid (82%, 201.7mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.66-7.68 (m, 2H), 7.44-7.48 (m, 3H), 6.46 (dt, $J_1 = 18.5$Hz, $J_2 = 6.4$Hz, 1H), 5.79-5.85 (m, 1H), 4.65 (d, $J = 3.2$Hz, 2H), 2.25-2.31 (m, 2H), 1.49-1.54 (m, 2H), 1.38-1.41 (m, 10H), 0.99 (t, $J = 6.8$Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 154.2, 135.3, 132.4, 129.5, 128.0, 119.8, 36.9, 31.9, 29.4, 29.3, 28.4, 22.7, 14.1; IR (film): 3065, 2131, 1615 cm$^{-1}$; HRMS (ESI) m/z Calculated for C$_{16}$H$_{27}$Si$^+$ [M+H]$^+$ 247.1877, found: 247.1876.

$(E)$-but-1-en-1-yl(phenyl)silane (3b):

![SiPhH$_2$]

Colorless liquid (89%, 169.1mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.63-7.66 (m, 2H), 7.40-7.47 (m, 3H), 6.43 (dt, $J_1 = 18.3$Hz, $J_2 = 6.3$Hz, 1H), 5.76-5.82 (m, 1H), 4.61 (d, $J = 3.2$Hz, 2H), 2.23-2.28 (m, 2H), 1.45-1.52 (m, 2H), 1.34-1.37 (m, 2H), 0.98 (t, $J = 7.2$Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 154.2, 135.4, 132.4, 129.6, 128.0, 119.8, 36.6, 30.6, 22.2, 13.9; IR (film): 3063, 2131, 1615 cm$^{-1}$; HRMS (ESI) m/z Calculated for C$_{12}$H$_{19}$Si$^+$ [M+H]$^+$ 191.1251, found: 191.1252.

$(E)$-pent-1-en-1-yl(phenyl)silane (3c):

![SiPhH$_2$]

Colorless liquid (68%, 156.6mg); $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.58-7.60 (m, 2H), 7.36-7.41 (m, 3H), 6.38 (dt, $J_1 = 18.5$Hz, $J_2 = 6.3$Hz, 1H), 5.73-5.76 (m, 1H), 4.55 (d, $J = 3.3$Hz, 2H), 2.16-2.21 (m, 2H), 1.44-1.51 (m, 2H), 0.94 (t, $J = 7.4$Hz, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 153.9, 135.4, 132.4, 129.6, 128.0, 120.1, 39.0, 21.6, 13.7; IR (film): 3063, 2132, 1618 cm$^{-1}$; HRMS (ESI) m/z Calculated for C$_{11}$H$_{17}$Si$^+$ [M+H]$^+$ 177.1095, found: 177.1094.

$(E)$-phenyl(4-phenylbut-1-en-1-yl)silane (3d):

![SiPhH$_2$]

Colorless liquid (64%, 152.3mg); $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.52-7.53 (m, 2H), 7.34-7.39 (m, 3H), 7.26-7.28 (m, 2H), 7.17-7.20 (m, 3H), 6.39 (dt, $J_1 = 18.4$Hz, $J_2 = 6.2$Hz, 1H), 5.74-5.78 (m, 1H), 4.52 (d, $J = 3.1$Hz, 2H), 2.75 (t, $J = 7.5$Hz, 2H), 2.48-2.53 (m, 2H). $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 152.7, 141.6, 135.4, 132.1, 129.6, 128.4, 128.3, 128.0, 125.9, 120.9, 38.5, 34.8; IR (film): 3065, 2133, 1614 cm$^{-1}$; HRMS (ESI) m/z Calculated for C$_{16}$H$_{19}$Si$^+$ [M+H]$^+$ 239.1251, found: 239.1252.
(E)-(5-methylhex-1-en-1-yl)(phenyl)silane (3e):

\[ \text{SiPh}_2 \]

Colorless liquid (64%, 130.6 mg); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.56-7.58 (m, 2H), 7.35-7.40 (m, 3H), 6.37 (dt, \(J_1 = 18.4\) Hz, \(J_2 = 6.3\) Hz, 1H), 5.71-5.75 (m, 1H), 4.53 (d, \(J = 3.1\) Hz, 2H), 2.17-2.22 (m, 2H), 1.53-1.61 (m, 1H), 1.30-1.34 (m, 2H), 0.90 (d, \(J = 6.6\) Hz, 6H); \(^1^3\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 154.3, 135.4, 132.4, 129.6, 128.0, 119.6, 37.6, 34.8, 27.6, 22.5 Hz; IR (film): 3068, 2132, 1619 cm\(^{-1}\); HRMS (ESI) m/z Calculated for C\(_{13}\)H\(_{21}\)Si\(^+\) [M+H]\(^+\) 205.1408, found: 205.1406.

(\(E\))-(3,3-dimethylbut-1-en-1-yl)(phenyl)silane (3f):

\[ \text{SiPh}_2 \]

Colorless liquid (58%, 110.2 mg); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.56-7.58 (m, 2H), 7.35-7.40 (m, 3H), 6.39 (d, \(J = 18.8\) Hz, 1H), 5.63 (m, 1H), 4.55 (s, 2H), 1.04 (s, 9H); \(^1^3\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 164.1, 135.3, 132.5, 129.5, 128.0, 113.4, 35.7, 28.9; IR (film): 3063, 2130, 1613 cm\(^{-1}\); HRMS (ESI) m/z Calculated for C\(_{12}\)H\(_{19}\)Si\(^+\) [M+H]\(^+\) 191.1251, found: 191.1250.

(\(E\))-(2-cyclopropylvinyl)(phenyl)silane (3g):

\[ \text{SiPh}_2 \]

Colorless liquid (57%, 99.2 mg); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.57-7.58 (m, 2H), 7.35-7.41 (m, 3H), 5.73-5.844 (m, 2H), 4.53 (d, \(J = 2.5\) Hz, 2H), 1.52-1.59 (m, 1H), 0.78-0.82 (m, 2H), 0.49-0.52 (m, 2H); \(^1^3\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 157.4, 135.3, 132.4, 129.6, 128.0, 116.4, 17.8, 7.6; IR (film): 3065, 2133, 1617 cm\(^{-1}\); HRMS (ESI) m/z Calculated for C\(_{11}\)H\(_{15}\)Si\(^+\) [M+H]\(^+\) 175.0938, found: 175.0936.

(\(E\))-(2-(cyclohex-1-en-1-yl)vinyl)(phenyl)silane (3h):

\[ \text{SiPh}_2 \]

Colorless liquid (47%, 100.6 mg); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.58-7.60 (m, 2H), 7.35-7.40 (m, 3H), 6.79 (d, \(J = 18.8\) Hz, 1H), 5.88 (s, 2H), 5.73 (dt, \(J_1 = 18.8\) Hz, \(J_2 = 3.0\) Hz, 1H), 4.61 (d, \(J = 3.2\) Hz, 2H), 2.16-2.17 (m, 4H), 1.66-1.70 (m, 2H), 1.60-1.63 (m, 2H); \(^1^3\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 153.1, 137.3, 135.4, 132.9, 132.4, 129.6, 128.0, 114.1, 26.0, 23.9, 22.5, 22.4; IR (film): 3067, 2133, 1616 cm\(^{-1}\); HRMS (ESI) m/z Calculated for C\(_{14}\)H\(_{19}\)Si\(^+\) [M+H]\(^+\) 215.1251, found: 215.1253.
(E)-(6-chlorohex-1-en-1-yl)(phenyl)silane (3i):

![Chemical Structure](image)

colorless liquid (54%, 113.4mg); $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.56-7.58 (m, 2H), 7.36-7.41 (m, 3H), 6.34 (dt, $J_1 = 18.5$Hz, $J_2 = 6.3$Hz, 1H), 5.75-5.78 (m, 1H), 4.54 (d, $J = 3.1$Hz, 2H), 3.55 (t, $J = 6.7$Hz, 2H), 2.21-2.25 (m, 2H), 1.77-1.83 (m, 2H), 1.57-1.63 (m, 2H). $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 152.9, 135.4, 132.1, 129.6, 128.0, 120.9, 44.9, 36.0, 32.0, 25.6; IR (film): 3066, 2133, 1617 cm$^{-1}$; HRMS (ESI) m/z Calculated for C$_{12}$H$_{18}$ClSi$^+$ [M+H]$^+$ 225.0861, found: 225.0863.

(E)-6-(phenylsilyl)hex-5-enenitrile (3j):

![Chemical Structure](image)

colorless liquid (44%, 88.4mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.56-7.58 (m, 2H), 7.36-7.44 (m, 3H), 6.28 (dt, $J_1 = 18.5$Hz, $J_2 = 6.3$Hz, 1H), 5.81-5.88 (m, 1H), 4.55 (d, $J = 3.0$Hz, 2H), 3.32-3.38 (m, 4H), 1.77-1.85 (m, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 150.3, 135.3, 131.6, 129.8, 128.1, 123.1, 119.4, 35.3, 24.1, 16.5; IR (film): 3065, 2234, 2132, 1615 cm$^{-1}$; HRMS (ESI) m/z Calculated for C$_{12}$H$_{16}$NSi$^+$ [M+H]$^+$ 202.1047, found: 202.1045.

(E)-trimethyl(2-(phenylsilyl)vinyl)silane (3k):

![Chemical Structure](image)

colorless liquid (57%, 117.4mg); $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.57-7.58 (m, 2H), 7.36-7.41 (m, 3H), 6.95 (d, $J = 22.3$Hz, 1H), 6.64 (dt, $J_1 = 22.3$Hz, $J_2 = 2.7$Hz, 1H), 4.57 (d, $J = 2.6$Hz, 2H), 0.09 (s, 9H). $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 160.8, 141.8, 137.3, 133.5, 131.5, 129.8, 0.0; IR (film): 3067, 2131, 1620; HRMS (ESI) m/z Calculated for C$_{11}$H$_{19}$Si$_2^+$ [M+H]$^+$ 207.1021, found: 207.1023.

(E)-tert-butyldimethyl((6-(phenylsilyl)hex-5-en-1-yl)oxy)silane (3l):

![Chemical Structure](image)

colorless liquid (70%, 224mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.58-7.61 (m, 2H), 7.36-7.42 (m, 3H), 6.38 (dt, $J_1 = 18.4$Hz, $J_2 = 6.3$Hz, 1H), 5.73-5.79 (m, 1H), 4.55 (d, $J = 3.2$Hz, 2H), 3.64 (t, $J = 6.3$Hz, 2H), 2.22-2.27 (m, 2H), 1.50-1.57 (m, 4H), 0.92 (s, 9H), 0.07 (s, 6H). $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 153.8, 135.3, 132.3, 129.6, 128.0, 120.1, 63.0, 36.6, 32.3, 26.0, 24.7, 18.4, -5.3; IR (film): 3062, 2132, 1615 cm$^{-1}$; HRMS (ESI) m/z Calculated for C$_{18}$H$_{33}$OSi$_2^+$ [M+H]$^+$ 321.2065, found: 321.2067.
(E)-6-(phenylsilyl)hex-5-en-1-yl 4-methylbenzenesulfonate (3m):  

![Chemical Structure](image)

colorless liquid (47%, 169.2mg); ¹H NMR (500 MHz, CDCl₃) δ 7.78-7.79 (m, 2H), 7.54-7.55 (m, 2H), 7.32-7.40 (m, 5H), 6.26 (dt, J₁ = 18.5Hz, J₂ = 6.2Hz, 1H), 5.67-5.71 (m, 1H), 4.51 (d, J = 3.1Hz, 2H), 4.03 (t, J = 6.4Hz, 2H), 2.44 (s, 3H), 2.11-2.16 (m, 2H), 1.63-1.69 (m, 2H), 1.43-1.49 (m, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 152.5, 144.7, 135.3, 133.2, 132.0, 129.8, 129.7, 128.0, 127.9, 121.1, 70.3, 35.9, 28.3, 24.1, 21.6; IR (film): 3068, 2132, 1617 cm⁻¹; HRMS (ESI) m/z Calculated for C₁₀H₁₅O₃Si⁺ [M+H]⁺ 361.1289, found: 361.1291.

(E)-phenyl(styryl)silane (3n):  

![Chemical Structure](image)

colorless liquid (65%, 136.5mg); ¹H NMR (500 MHz, CDCl₃) δ 7.61-7.63 (m, 2H), 7.45-7.46 (m, 2H), 7.28-7.40 (m, 6H), 7.16 (d, J = 19.0Hz, 1H), 6.51 (dt, J₁ = 19.0Hz, J₂ = 3.1Hz, 1H), 4.70 (d, J = 3.1Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 149.3, 137.8, 135.5, 131.7, 129.8, 128.7, 128.6, 128.1, 126.7, 119.4; IR (film): 3066, 2137, 1604 cm⁻¹; HRMS (ESI) m/z Calculated for C₁₄H₁₅Si⁺ [M+H]⁺ 211.0938, found: 211.0939.

(E)-(4-ethylstyryl)(phenyl)silane (3o):  

![Chemical Structure](image)

colorless liquid (64%, 152.3mg); ¹H NMR (500 MHz, CDCl₃) δ 7.62-7.64 (m, 2H), 7.37-7.42 (m, 5H), 7.13-7.19 (m, 3H), 6.45 (d, J = 19.0Hz, 1H), 4.69 (s, 2H), 2.65 (q, J = 7.5Hz, 2H), 1.24 (t, J = 7.6Hz, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 149.3, 145.1, 135.5, 135.4, 131.9, 129.8, 128.1, 126.7, 118.0, 28.7, 15.5; IR (film): 3065, 2132, 1599 cm⁻¹; HRMS (ESI) m/z Calculated for C₁₆H₁₉Si⁺ [M+H]⁺ 239.1251, found: 239.1252.

(E)-(4-methoxystyryl)(phenyl)silane (3p):  

![Chemical Structure](image)

colorless liquid (60%, 144mg); ¹H NMR (400 MHz, CDCl₃) δ 7.65-7.70 (m, 2H), 7.39-7.49 (m, 5H), 7.18 (d, J = 18.9 Hz, 1H), 6.89-6.94 (m, 2H), 6.40 (dt, J₁ = 19.0Hz, J₂ = 3.3Hz, 1H), 4.77 (d, J = 3.2Hz, 2H), 3.85 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 160.1, 148.8, 135.5, 132.0, 130.8, 129.7, 128.1, 128.0, 116.4, 114.0, 55.3; IR (film): 3068, 2135, 1601 cm⁻¹; HRMS (ESI) m/z Calculated for C₁₅H₁₇OSi⁺ [M+H]⁺ 241.1044, found: 241.1045.
(E)-(4-fluorostyryl)(phenyl)silane(3q):

\[
\begin{align*}
\text{F} & \quad \text{SiH}_2\text{Ph} \\
\end{align*}
\]

colorless liquid (45%, 102.6mg); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.63-7.65 (m, 2H), 7.39-7.45 (m, 5H), 7.10 (d, \(J=18.9\)Hz, 1H), 7.02-7.06 (m, 2H), 6.43 (dt, \(J_1=18.9\)Hz, \(J_2=3.2\)Hz, 1H), 4.70 (d, \(J=3.1\)Hz, 2H). 

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 163.0 (d, \(J=248.8\)Hz), 148.0, 135.5, 134.1, 131.5, 129.9, 128.3 (d, \(J=8.2\)Hz), 128.2, 119.2 (d, \(J=2.0\)Hz), 115.5 (d, \(J=22.1\)Hz); IR (film): 3068, 2137, 1601 cm\(^{-1}\); HRMS (ESI) m/z Calculated for C\(_{14}\)H\(_{14}\)FSi\(^{+}\) [M+H]\(^{+}\) 229.0844, found: 229.0843.
4 $^1$H NMR and $^{13}$C NMR Spectra of the Products

(E)-but-1-en-1-yl(phenyl)silane (3a):

$^1$H NMR in CDCl$_3$ (400 MHz)

$^{13}$C NMR in CDCl$_3$ (125 MHz)
(E)-but-1-en-1-yl(phenyl)silane (3b):
(E)-pent-1-en-1-yl(phenyl)silane (3c):
(E)-phenyl(4-phenylbut-1-en-1-yl)silane (3d):
(E)-(5-methylhex-1-en-1-yl)(phenyl)silane (3e):

$^1$H NMR in CDCl$_3$ (500 MHz)

$^{13}$C NMR in CDCl$_3$ (125 MHz)
(E)-(3,3-dimethylbut-1-en-1-yl)(phenyl)silane (3f):

$\text{H NMR in CDCl}_3 (600 \text{ MHz})$

$^{13}$C NMR in CDCl$_3$ (125 MHz)
(E)-(2-cyclopropylvinyl)(phenyl)silane (3g):

$^1$H NMR in CDCl$_3$ (600 MHz)

$^{13}$C NMR in CDCl$_3$ (150 MHz)
*(E)-(2-(cyclohex-1-en-1-yl)vinyl)(phenyl)silane (3h):*

1 H NMR in CDCl3 (500 M)

13 C NMR in CDCl3 (125 M)
(E)-(6-chlorohex-1-en-1-yl)(phenyl)silane (3i):
(E)-6-(phenylsilyl)hex-5-enenitrile (3j):

1H NMR in CDCl3 (400 MHz)

13C NMR in CDCl3 (100 MHz)
(E)-trimethyl(2-(phenylsilyl)vinyl)silane (3k):

$^1$H NMR in CDCl$_3$ (500 MHz)

$^{13}$C NMR in CDCl$_3$ (125 MHz)
(E)-tert-butyldimethyl((6-(phenylsilyl)hex-5-en-1-yl)oxy)silane (3l):

1 H NMR in CDCl3 (400 M):

13 C NMR in CDCl3 (125 M):
(E)-6-(phenylsilyl)hex-5-en-1-yl 4-methylbenzenesulfonate (3m):
(E)-phenyl(styryl)silane (3n):
(E)-(4-ethylstyril)(phenyl)silane (30):
(E)-(4-methoxystyryl)(phenyl)silane (3p):
(E)-(4-fluorostyryl)(phenyl)silane (3q):