Silicon-assisted Homopropargylic Transfer to Aldehydes

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Supporting Information

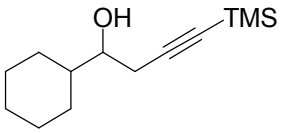
General

THF was distilled from sodium/benzophenone. Hexane and dichloromethane were distilled from CaH_2. TLC was carried out with pre-coated Merck 60 F_{254} plates. Silica gel 60 (Merck, 400-630 mesh) was used for column chromatography. Infrared spectra were recorded on a Bio-Rad FTS 165 FTIR spectrometer. Liquid samples were examined as film between NaCl salt plates. ^1H and ^{13}C NMR spectra were taken in $CDCl_3$ on Bruker DPX300 and Bruker AMX500 and referenced to internal tetramethylsilane ($SiMe_4$). Chemical shifts for ^1H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 7.2600, singlet). Multiplicities were given as: s (singlet); brs (broad singlet); d (doublet); t (triplet); q (quartet); dd (doublets of doublet); dt (doublets of triplet); dtq (doublets of triplets of quartet); or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a J value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 77, triplet).
Mass spectral analyses were carried out on a VG 7035 micromass mass spectrophotometer at a source temperature of 200 ºC and at an ion current of 70 eV. Mass spectral data were reported in units of mass to charge (m/z) and % intensity.

Experimental Section

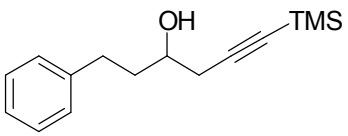
Crossover Reactions of Allenic Alcohols and Aldehydes in CH_2Cl_2 with $In(OTf)_3$; General Procedure:
The mixture of cyclohexanecarboxyldehyde (89 mg, 0.8 mmol, 4 equiv) and 1-cyclohexyl-2-trimethylsilanyl-buta-2,3-dien-1-ol (45 mg, 0.2 mmol, 1 equiv) was added to a solution of indium triflate (1 mg, 0.002 mmol, 0.01 equiv) in 6 mL dried CH_2Cl_2 at room temperature under an atmosphere of dry nitrogen. The mixture was stirred for 1 hour and finally quenched with saturated sodium bicarbonate. Purification through flash silica gel column chromatography provides 44.1 mg (98% yield) of 1-cyclohexyl-4-trimenthylsilanyl-but-3-yn-ol as a colourless oil.

1-Cyclohexyl-4-trimenthylsilanyl-but-3-yn-ol (Table 3, entry 1)



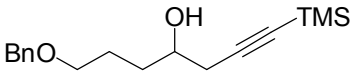
Yield%: 98%
R_f 0.67 (hexane : ethyl acetate 4:1)
^{1}H NMR: (300 MHz, $CDCl_3$)
δ 3.46-3.41 (m, 1H, C**H**OH), 2.43 (dd, *J* = 3.6, 16.71 Hz, 1H, C≡CC**H**H), 2.32 (dd, *J* = 7.65, 16.71 Hz, 1H, C≡CCH**H**), 2.12 (br, 1H, CHO**H**), 1.86-0.96 (m, 11H, c-C_6**H**$_{11}$), 0.11 [s, 9H, Si(C**H**$_3$)$_3$] ppm
^{13}C NMR: (125 MHz, $CDCl_3$)
δ 103.7 (**C**≡CSi), 87.3 (C≡**C**Si), 73.8 (**C**HOH), 42.6 (**C**HCHOH), 28.9, 28.1 , 26.3, 26.1, 26.0, 25.9 (**C**H$_2$), -0.01 (Si(**C**H$_3$)$_3$) ppm
FTIR: 3402, 2928, 2851, 2663, 2174, 1713, 1450, 1422, 1349, 1249, 1198, 1103, 1086, 1015, 842, 760, 698, 651 cm^{-1}
EIHRMS: Calcd for $C_{13}H_{24}OSi$: 224.1596, found : 224.1594.

1-Phenyl-6-trimethylsilanyl-hex-5-yn-3-ol (Table 3, entry 2)



Yield%: 79%
R_f 0.54 (hexane : ethyl acetate 4:1)
^{1}H NMR: (500 MHz, $CDCl_3$)
δ 7.31-7.26 (m, 2H, phenyl), 7.22-7.18 (m, 3H, phenyl), 3.76 (quintet, *J* = 6.45 Hz, 1H, C**H**OH), 2.81 (dt, *J* = 13.9, 7.35 Hz, 1H, PhC**H**H), 2.71 (dt, *J* = 13.9, 8.35 Hz, 1H, PhCH**H**), 2.48 (dd, *J* = 16.65, 5.1 Hz, 1H, C≡CC**H**H), 2.40 (dd, *J* = 6.95, 16.63 Hz, 1H, C≡CCH**H**), 2.09 (brs, 1H, CHO**H**), 1.86 (td, *J* = 8.35, 6.0 Hz, 2H, PhCH$_2$C**H**$_2$), 0.17 [s, 9H, Si(C**H**$_3$)$_3$] ppm
^{13}C NMR: (125 MHz, $CDCl_3$)
δ 141.7, 128.3, 125.8 (phenyl), 102.9 (**C**≡CSi), 87.7 (C≡**C**Si), 69.0 (**C**HOH), 37.7 (**C**H$_2$CHOH), 31.8 (Ph**C**H$_2$), 28.9 (**C**H$_2$C≡C), 0.03 (Si(**C**H$_3$)$_3$) ppm
FTIR: 3434, 3020, 2961, 2170, 1718, 1492, 1454, 1411, 1251, 1216, 1040, 845, 758, 700, 669 cm^{-1}
EIHRMS: Calcd for $C_{15}H_{22}OSi$: 246.1440, found : 246.1396.

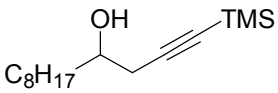
7-Benzyloxy-1-trimethylsilanyl-hept-1-yn-4-ol (Table 3, entry 3)



Yield%: 78%
R_f 0.40 (hexane : ethyl acetate 4:1)
^{1}H NMR: (500 MHz, $CDCl_3$)
δ 7.19-7.29 (m, 5H, phenyl), 4.44 (s, 2H, PhC**H**$_2$O), 3.70-3.66 (m, 1H, C**H**OH), 3.45 (t, *J* = 6 Hz, 2H, BnOC**H**$_2$), 2.60 (brs, 1H, CHO**H**), 2.36 (dd, *J* = 5.55, 16.87 Hz, 1H, C≡CC**H**H), 2.32 (dd, *J* = 6.5, 16.65 Hz, 1H, C≡CCH**H**), 1.73-1.47 (m, 4H, BnOCH$_2$(C**H**$_2$)$_2$), 0.09 (s, 9H, Si(C**H**$_3$)$_3$) ppm

^{13}C NMR: (125 MHz, $CDCl_3$)
δ 138.1, 128.3, 127.6, 127.5 (Phenyl), 103.4 (**C**≡CSi), 87.2 (C≡**C**Si), 72.9 (O**C**H$_2$), 70.2 (O**C**H$_2$), 69.7 (**C**HOH), 33.3 (**C**H$_2$CHOH), 28.7 (**C**H$_2$C≡C), 26.0 (BnOCH$_2$**C**H$_2$) 0.04 (Si(**C**H$_3$)$_3$) ppm
FTIR: 3409, 3088, 3064, 3031, 2957, 2929, 2902, 2859, 2174, 1605, 1454, 1362, 1249, 1096, 843, 734 cm^{-1}
EIHRMS: Calcd for $C_{17}H_{26}O_2Si$: 290.1702, found : 290.1701.

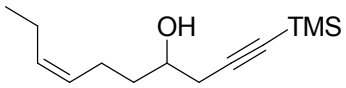
1-Trimethylsilanyl-dodec-1-yn-4-ol (Table 3, entry 4)



Yield%: 77%
R_f 0.68 (hexane : ethyl acetate 4:1)
^{1}H NMR: (300 MHz, $CDCl_3$)
δ 3.71 (quintet, *J* = 5.55 Hz, 1H, C**H**OH), 2.45 (dd, *J* = 4.89, 16.71 Hz, 1H, C≡CC**H**H), 2.33 (dd, *J* = 6.96, 16.71 Hz, 1H, C≡CCH**H**), 1.96 (brs, 1H, CHO**H**), 1.52-1.48 (m, 2H, C**H**$_2$CHOH), 1.27 [m, 12H, (C**H**$_2$)$_6$], 0.87 (t, *J* = 5.9 Hz, 3H, C**H**$_3$), 0.15 [s, 9H, Si(C**H**$_3$)$_3$] ppm
^{13}C NMR: (75 MHz, $CDCl_3$)
δ 103.4 (**C**≡CSi), 86.9 (C≡**C**Si), 70.0 (**C**HOH), 36.2 (**C**H$_2$CHOH), 31.9, 29.5, 29.3, 28.9, 25.6, 22.7 [(**C**H$_2$)$_6$CH$_3$], 14.1 (**C**H$_3$), 0.07 [Si(**C**H$_3$)$_3$] ppm
FTIR: 3365, 2957, 2926, 2855, 2176, 1465, 1250, 1084, 1005, 842, 760, 698, 648 cm^{-1}

EIHRMS: Calcd for $C_{15}H_{30}OSi$: 254.2066, found : 254.2067.

1-trimethylsilanyl-dec-7-en-1-yn-4-ol (Table 3, entry 5)



Yield%: 77%

R_f 0.64 (hexane : ethyl acetate 4:1)

^{1}H NMR: (500 MHz, $CDCl_3$)

δ 5.39 (m, 1H, **H**C=CH), 5.32 (m, 1H, HC=C**H**), 3.74 (quintet, *J* = 6 Hz, 1H, C**H**OH), 2.45 (dd, *J* = 4.65, 16.65 Hz, 1H, C≡CC**H**H), 2.35 (dd, *J* = 6.5, 16.87 Hz, 1H, C≡CCH**H**), 2.19-2.10 (m, 2H, C=CC**H**$_2$CH$_3$), 2.08-2.02 (m, 2H, C=CC**H**$_2$CH$_2$), 2.00 (brs, 1H, CHO**H**), 1.58 (td, *J* = 7.62, 6.5 Hz, 2H, C**H**$_2$CHOH), 0.96 (t, *J* = 7.65 Hz, 3H, CH$_2$C**H**$_3$), 0.15 (s, 9H, Si(C**H**$_3$)$_3$) ppm

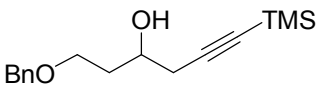
^{13}C NMR: (125 MHz, $CDCl_3$)

δ 132.4 (H**C**=CH), 128.1(HC=**C**H), 103.1 (**C**≡CSi), 87.6 (C≡**C**Si), 69.4 (**C**HOH), 36.1 (**C**H$_2$), 28.8 (**C**H$_2$C≡C), 23.3 (**C**H$_2$), 20.4 (**C**H$_2$), 14.2 (CH$_2$**C**H$_3$), 0.03 (Si(**C**H$_3$)$_3$) ppm

FTIR: 3392, 3007, 2962, 2933, 2875, 2856, 2176, 1654, 1453, 1249, 1067, 1033, 842, 760 cm^{-1}

EIHRMS: Calcd for $C_{13}H_{24}OSi$: 224.1596, found : 246.1600.

1-Benzyloxy-6-trimethylsilanyl-hex-5-yn-3-ol (Table 3, entry 6)



Yield%: 73%

R_f 0.46 (hexane : ethyl acetate 4:1)

^{1}H NMR: (500 MHz, $CDCl_3$)

δ 7.30-7.27 (m, 5H, phenyl), 4.52 (s, 2H, PhC**H**$_2$O), 3.96 (tq, *J* = 9.03, 3.25 Hz, 1H, C**H**OH), 3.73 (ddd, *J* = 9.49, 7.88, 4.6 Hz, 1H, BnOC**H**H), 3.65 (ddd, *J* = 10.88, 7.65, 4.65 Hz, 1H, BnOCH**H**), 2.46 (dd, *J* = 6.05, 17.35 Hz, 1H, C≡CC**H**H), 2.41 (dd, *J* = 6.45, 16.85 Hz, 1H, C≡CCH**H**), 1.92 (dddd, *J* = 14.32, 6.48, 4.63, 3.25 Hz, 1H, BnOCH$_2$C**H**H), 1.86-1.79 (m, 1H, BnOCH$_2$CH**H**), 0.15 (s, 9H, Si(C**H**$_3$)$_3$) ppm

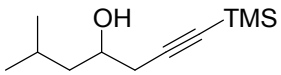
^{13}C NMR: (125 MHz, $CDCl_3$)

δ 137.8, 128.4, 127.7, 127.6 (Phenyl), 103.3 (**C**≡CSi), 87.0 (C≡**C**Si), 73.2 (O**C**H$_2$) 69.3 (**C**HOH), 68.4 (O**C**H$_2$), 35.3 (**C**H$_2$CHOH), 28.5 (**C**H$_2$C≡C), 0.04 (Si(**C**H$_3$)$_3$) ppm

FTIR: 3430, 3089, 3065, 3032, 2958, 2926, 2900, 2863, 2175, 1632, 1454, 1249, 1098, 843, 735 cm^{-1}

EIHRMS: Calcd for $C_{16}H_{23}O_2Si$ (M^+) : 275.1473, found : 275.1466.

6-methyl-1-trimethylsilanyl-hept-1-yn-4-ol (Table 3, entry 7)



Yield%: 73%

R_f 0.61 (hexane : ethyl acetate 4:1)

^{1}H NMR: (500 MHz, $CDCl_3$)

δ 3.80 (m, 1H, C**H**OH), 2.43 (dd, *J* = 4.65, 16.62 Hz, 1H, C≡CC**H**H), 2.32 (dd, *J* = 6.95, 16.62 Hz, 1H, C≡CCH**H**), 1.77 (m, 1H, C**H**$(CH_3)_2$), 1.45 (ddd, *J* = 8.66, 8.88, 13.99 Hz, 1H, $(CH_3)_2$CHC**H**H), 1.29 (ddd, *J* = 8.55, 8.88, 14.4 Hz, 1H, $(CH_3)_2$CHCH**H**), 0.91 (q, *J* = 3.45 Hz, 6H, CH(C**H**$_3)_2$), 0.15 (s, 9H, Si(C**H**$_3)_3$) ppm

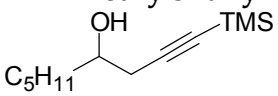
^{13}C NMR: (125 MHz, $CDCl_3$)

δ 103.3 (**C**≡CSi), 87.6 (C≡**C**Si), 68.0 (**C**HOH), 45.4 [$(CH_3)_2$CH**C**H_2], 29.3 (**C**H_2C≡C), 24.6 [**C**H$(CH_3)_2$], 23.25, (CH**C**H_3), 22.1 (CH**C**H_3) 0.05 (Si(**C**$H_3)_3$) ppm

FTIR: 3401, 2958, 2929, 2871, 2176, 1641, 1467, 1368, 1250, 1014, 842, 760 647 cm^{-1}

EIHRMS: Calcd for $C_{11}H_{22}OSi$: 198.1440, found : 198.1397.

1-Trimethylsilanyl-non-1-yn-4-ol (Table 3, entry 8)



Yield: 71%.

R_f: 0.64 (Hexane : Ethyl Acetate = 4:1)

^{1}H NMR (500 MHz, $CDCl_3$):

δ 3.72 (quintet, *J* = 6.45 Hz, 1H, C**H**OH), 2.44 (dd, *J* = 4.60, 16.87 Hz, 1H, C≡CC**H**H), 2.44 (dd, *J* = 6.95, 16.62 Hz, 1H, C≡CCH**H**), 1.53-1.49 (m, 2H, C**H**$_2$), 1.33-1.27 (m, 6H, (C**H**$_2)_3$), 0.87 (t, *J* = 6.95 Hz, 3H, C**H**$_3$), 0.15 (s, 9H, Si(C**H**$_3)_3$) ppm

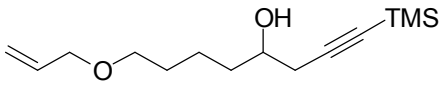
^{13}C NMR (125 MHz, $CDCl_3$):

δ 103.3 (**C**≡CSi), 87.5 (C≡**C**Si), 69.9 (**C**HOH), 36.2 (**C**H_2CHOH), 31.7, 28.8, 25.2, 22.5 (**C**$H_2)_4$CH$_3$), 13.9 $(CH_2)_4$**C**H_3), 0.04 (Si(**C**$H_3)_3$) ppm

FTIR: 3394, 2958, 2932, 2873, 2859, 2176, 1421, 1249, 1020, 840, 760 cm^{-1}

EIHRMS: Calcd for $C_{12}H_{24}OSi$: 212.1596, found : 212.1596.

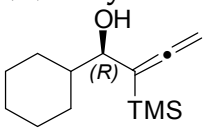
8-Allyloxy-1-trimethylsilanyl-oct-1-yn-4-ol (Table 3, entry 9)



Yield%: 70%
R$_f$ 0.42 (hexane : ethyl acetate 4:1)
^{1}H NMR: (500 MHz, $CDCl_3$)
δ 5.89 (tdd, J = 11.47, 10.53, 5.55 Hz, 1H, C**H**=CH_2), 5.25 (ddt, *J* = 9.71, 3.25, 1.85 Hz, 1H, CH=C**H**H), 5.15 (ddt, *J* = 10.27, 3, 1.4 Hz, 1H, CH=CH**H**), 3.95 (dt, J = 6, 1.4 Hz, 2H, CH_2=CHC**H**$_2$O), 3.72 (quintet, *J* = 6 Hz, 1H, C**H**OH), 3.42 (t, *J* = 6.5 Hz, 2H, AllyOC**H**$_2$), 2.44 (dd, *J* = 5.1, 16.62 Hz, 1H, C≡CC**H**H), 2.34 (dd, *J* = 6.95, 16.62 Hz, 1H, C≡CCH**H**), 2.07 (brs, 1H, CHO**H**), 1.37-1.66 (m, 6H, AllyOCH_2(C**H**$_2$)$_3$), 0.14 (s, 9H, Si(C**H**$_3$)$_3$) ppm
^{13}C NMR: (125 MHz, $CDCl_3$)
δ 134.9 (**C**H=CH_2), 116.7 (CH=**C**H$_2$), 103.2 (**C**≡CSi), 87.5 (C≡**C**Si), 71.8 (O**C**H$_2$), 70.1 (O**C**H$_2$), 69.7 (**C**HOH), 35.9 (**C**H$_2$CHOH), 29.5 (CH_2), 28.8 (**C**H$_2$C≡C), 22.2 (**C**H$_2$) 0.03 (Si(**C**H$_3$)$_3$) ppm
FTIR: 3427, 3081, 2939, 2863, 2175, 1420, 1347, 1249, 1103, 1010, 922, 843, 760 cm^{-1}
ESIHRMS: Calcd for $C_{14}H_{26}O_2SiNa$: 277.1600, found : 277.1599.

Chiral resolution of **1** was carried out with *S*-(+)-α-acetoxyphenylacetic acid followed by hydrolysis. For the standard procedure, refer to Whitesell, J.K.; Reynolds, D. *J. Org. Chem.* **1983**, *48*, 3548. (Note: the separation of the *S*-(+)-α-acetoxyphenylacetic-ester of **1** was done by silica gel column chromatography with Hexane:Acetone = 250:0.5 as mobile phase).
Enantiomeric excess for (-)-1-cyclohexyl-2-trimethylsilanyl-buta-2,3-dien-1-ol was 92 % by NMR (500 MHz, $CDCl_3$) analysis.
The configurations were determined based on literature:
Brown, H.C.; Khire, U.R.; Narla, G. *J. Org. Chem.* **1995**, *60*, 8130-8131.
Compain, P.; Gorè, J.; Vatèle, J.M. *Tetrahedron* **1996**, *52*(31), 10405-10416.

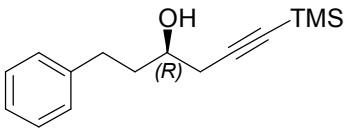
(*R*)-1-Cyclohexyl-2-trimethylsilanyl-buta-2,3-dien-1-ol (1)



Selectivity: 92 % *ee*
Opt. Rot.: $[\alpha]_D^{25}$ -16.95 (c = 7.8 in $CHCl_3$)
R$_f$ 0.75 (hexane : ethyl acetate 4:1)

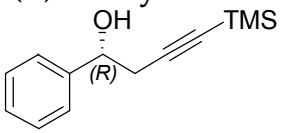
^{1}H NMR: (500 MHz, $CDCl_3$)
δ 4.54 (dd, *J* = 2.3, 11.08 Hz, 1H, C=C=C**H**H), 4.50 (dd, *J* = 2.3, 11.1 Hz, 1H, C=C=CH**H**), 3.91 (m, 1H, C**H**OH), 1.85-0.96 (m, 11H, c-C_6**H**$_{11}$), 0.14 [s, 9H, Si(C**H**$_3$)$_3$] ppm
^{13}C NMR: (125 MHz, $CDCl_3$)
δ 207.5 (C=**C**=CH_2), 99.5 (**C**=C=CH_2), 75.1 (**C**HOH), 71.5 (C=C=**C**H_2), 43.6 (**C**HCHOH), 30.4, 26.8, 26.4, 26.3, 26.0 (**C**H_2), -0.8 [Si(**C**H_3)$_3$] ppm
FTIR: 3436, 2929, 2855, 1925, 1716, 1666, 1449, 1250, 1019, 843 757 cm^{-1}.
EIHRMS: Calcd for $C_{13}H_{24}OSi$: 224.1596, found : 224.1592.

(*R*)-Cyclohexyl-4-trimenthylsilanyl-but-3-yn-ol (Table 4, entry 1)



Selectivity: 84 % *ee*
Opt. Rot.: $[\alpha]_D^{25}$ +15.8 (c = 0.9 in $CHCl_3$)
HPLC analysis employing a Daicel Chiracel OD column
(Hexane: *i*-propanol 99:1, 0.3 mL/min: t_1 = 14.08 t_2 = 19.72.
^{1}H NMR: (500 MHz, $CDCl_3$)
δ 7.30-7.27 (m, 2H, phenyl), 7.21-7.17 (m, 3H, phenyl), 3.75 (quintet, *J* = 6.0 Hz, 1H, C**H**OH), 2.81 (dt, *J* = 13.4, 7.85 Hz, 1H, PhC**H**H), 2.70 (dt, *J* = 13.85, 8.35 Hz, 1H, PhCH**H**), 2.48 (dd, *J* = 16.62, 4.65 Hz, 1H, C≡CC**H**H), 2.38 (dd, *J* = 16.65, 6.05 Hz, 1H, C≡CCH**H**), 1.98 (brs, 1H, CHO**H**), 1.85 (td, *J* = 7.7, 6.5 Hz, 2H, PhCH_2C**H**$_2$), 0.16 [s, 9H, Si(C**H**$_3$)$_3$] ppm
^{13}C NMR: (125 MHz, $CDCl_3$)
δ 141.7, 128.4, 125.8 (phenyl), 102.9 (**C**≡CSi), 87.8 (C≡**C**Si), 69.0 (**C**HOH), 37.7 (**C**H_2CHOH), 31.8 (Ph**C**H_2), 28.9 (**C**H_2C≡C), 0.05 (Si(**C**H_3)$_3$) ppm
FTIR: 3434, 3020, 2961, 2170, 1718, 1492, 1454, 1411, 1251, 1216, 1040, 845, 758, 700, 669 cm^{-1}
EIHRMS: Calcd for $C_{15}H_{22}OSi$: 246.1440, found : 246.1396.

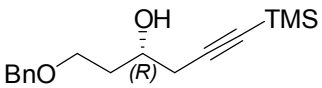
(*R*)-Phenyl-4-trimethylsilanyl-but-3-yn-1-ol (Table 4, entry 2)



Yield: 44%.
Selectivity: 89 % *ee*
Opt. Rot.: $[\alpha]_D^{25}$ +43.6 (c = 0.5 in $CHCl_3$)
HPLC analysis employing a Daicel Chiracel OD-H column

(Hexane: *i*-propanol 99:1, 1 mL/min: t_1 = 10.67 t_2 = 14.00.
R_f: 0.51 (Hexane : Ethyl Acetate = 4:1)
^{1}H NMR (300 MHz, $CDCl_3$):
δ 7.38-7.26 (m, 5H, phenyl), 4.84 (t, *J* = 6.27 Hz, 1H, PhC**H**OH), 2.65 (d, *J* = 6.27 Hz, 2H, C**H**$_2$C≡CSi), 2.45 (brd, 1H, CHO**H**), 0.17(s, 9H, Si(C**H**$_3$)$_3$) ppm
^{13}C NMR (75 MHz, $CDCl_3$):
δ142.5, 128.9, 127.8, 125.7, 103.0, 87.8 (CH$_2$**C**≡CSi), 72.3 (**C**HOH), 31.0 (**C**HCH$_2$C), 0.06 (Si(**C**H$_3$)$_3$) ppm
FTIR: 3396, 3033, 2960, 2902, 2177, 1724, 1604, 1494, 1454, 1250, 1202, 1039, 846, 759, 700 cm^{-1}
EIHRMS: Calcd for $C_{13}H_{18}OSi$: 218.1127, found: 218.1125.

(*R*)-Benzyloxy-6-trimethylsilanyl-hex-5-yn-3-ol (Table 4, entry 3)



R_f 0.46 (hexane : ethyl acetate 4:1)
Selectivity: 81 % *ee*
Opt. Rot.: $[\alpha]_D^{26}$ +13 (c = 0.05 in $CHCl_3$)
HPLC analysis employing a Daicel Chiracel OD-H column
(Hexane: *i*-propanol 99:1, 0.3 mL/min: t_1 = 27.64 t_2 = 30.71.
^{1}H NMR: (500 MHz, $CDCl_3$)
δ 7.30-7.27 (m, 5H, phenyl), 4.52 (s, 2H, PhC**H**$_2$O), 3.96 (tq, *J* = 9.03, 3.25 Hz, 1H, C**H**OH), 3.73 (ddd, *J* = 9.49, 7.88, 4.6 Hz, 1H, BnOC**H**H), 3.65 (ddd, *J* = 10.88, 7.65, 4.65 Hz, 1H, BnOCH**H**), 2.46 (dd, *J* = 6.05, 17.35 Hz, 1H, C≡CC**H**H), 2.41 (dd, *J* = 6.45, 16.85 Hz, 1H, C≡CCH**H**), 1.92 (dddd, *J* = 14.32, 6.48, 4.63, 3.25 Hz, 1H, BnOCH$_2$C**H**H), 1.86-1.79 (m, 1H, BnOCH$_2$CH**H**), 0.15 (s, 9H, Si(C**H**$_3$)$_3$) ppm
^{13}C NMR: (125 MHz, $CDCl_3$)
δ 137.8, 128.4, 127.7, 127.6 (Phenyl), 103.3 (**C**≡CSi), 87.0 (C≡**C**Si), 73.2 (O**C**H$_2$) 69.3 (**C**HOH), 68.4 (O**C**H$_2$), 35.3 (**C**H$_2$CHOH), 28.5 (**C**H$_2$C≡C), 0.04 (Si(**C**H$_3$)$_3$) ppm
FTIR: 3430, 3089, 3065, 3032, 2958, 2926, 2900, 2863, 2175, 1632, 1454, 1249, 1098, 843, 735 cm^{-1}
EIHRMS: Calcd for $C_{16}H_{24}O_2Si$: 276.1546, found : 276.1506.