

Title: Direct Coupling of Alcohols with Alkenylsilanes Catalyzed by Indium Trichloride or Bismuth Tribromide

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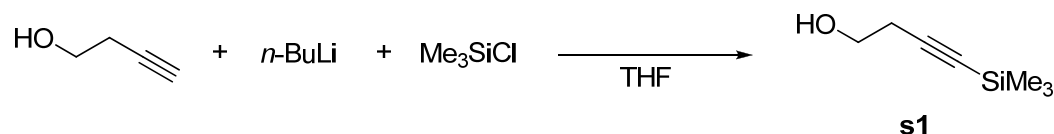
General.

IR spectra were recorded as thin films or as solids in KBr pellets on a HORIBA FT-720 spectrophotometer. ¹H (400 MHz) and ¹³C NMR (100 MHz) spectra were obtained with TMS as internal standard. Mass spectra were recorded on a JEOL JMS-DS303. Column chromatography was performed on silica gel (MERK C60). Bulb-to-bulb distillation (Kugelrohr) was accomplished in a Sibata GTO-250RS at the oven temperature and pressure indicated. Yields were determined by ¹H NMR using internal standards.

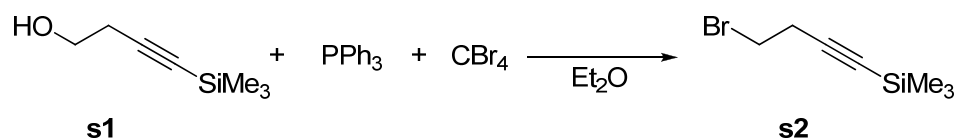
Materials.

Dichloromethane (stabilized with 2-methyl-2-butene, Wako Pure Chemical Industries, Ltd.) were used as obtained. 1,2-Dichloroethane was distilled from P₂O₅. The starting vinylsilane **4** and **6** were prepared and the experimental details are described below (These preparation methods were not optimized.). The starting alcohol **1j**¹ and the starting vinylsilane **2a**², **2b**³, **2c**³, **2d**³, **2e**², **2f**⁴, **2g**⁵ and **2h**² were prepared by known methods and these compounds was reported. All other starting alcohols are commercially available. All catalysts were commercially available.

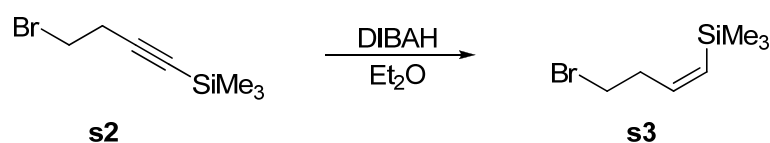
Synthesis of **4**.



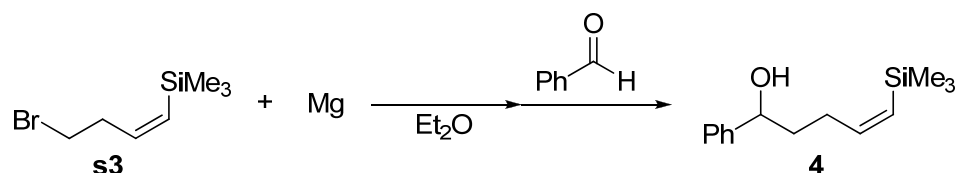
To a solution of 4-pentyn-1-ol (143 mmol) in THF (800 mL) was slowly added *n*-BuLi (210 mL, 1.6 M in hexane) at -78 °C and the reaction mixture was stirred for 30 min at -78 °C. Me₃SiCl (55 mL) was added and the reaction mixture was allowed to warm to room temperature and quenched with aqueous HCl (2M, 200 mL). After 48 h, the mixture was extracted with ether (50 x 3 mL), and the collected organic layer was dried (MgSO₄). The solvent was evaporated and the residue was purified by column chromatography and distillation to give the product **s1** (18.9 g, 93%). The compound **s1** was reported (ref. E. C. Davison, I. T. Forbes, A. B. Holmes and J. A. Warner, *Tetrahedron*, **1996**, 52, 11601.).



To a solution of PPh₃ (140 mmol) and CBr₄ (140 mmol) in Et₂O (160 mL) at room temperature was added **s1** (127 mmol) and stirring continued at room temperature for 2 days. The resulting suspension was filtered, dried (MgSO₄) and the solvent removed in vacuo. The residue was purified by distillation to give the product **s2** (26.4 g, 98%). The compound **s2** was reported (ref. A. P. Dobbs, K. Jones and K. T. Veal, *Tetrahedron*, **1998**, *54*, 2149.).

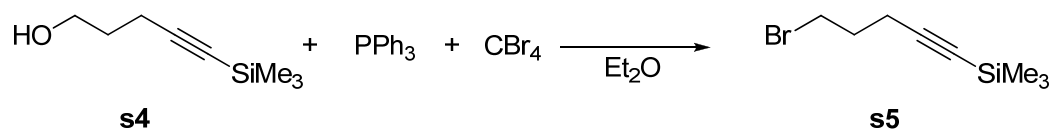


Hexane was removed from DIBALH (187 mL, 1 M in hexane) in vacuo, and Et₂O (340 mL) was added. To this solution of DIBALH in Et₂O was added **s2** at room temperature, and the reaction mixture was stirred at 40 °C. Then, the reaction was quenched at 0 °C with 3 N HCl, extracted with Et₂O (50 x 3 mL), washed with sat. NaHCO₃aq (50 x 3 mL) and brine (50 x 1 mL), dried (MgSO₄) and the solvent removed in vacuo. The residue was purified by distillation to give the product **s3**. The compound **s3** was reported (ref. E. Negishi, L. D. Boardman, H. Sawada, V. Bagheri, A. T. Stoll, J. M. Tour and C. L. Rand, *J. Am. Chem. Soc.*, **1988**, *110*, 5383.).

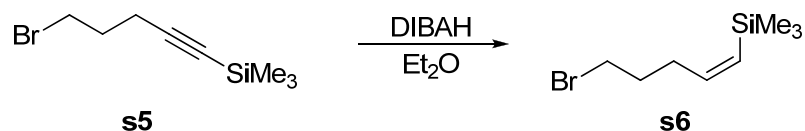


To a suspension of Mg (88 mmol) in THF (5 mL) was slowly added a solution of **s3** (82 mmol) in THF (10 mL). Then, the additional THF (30 mL) was added, and the reaction mixture was refluxed for 1 h. To the resulted mixture was slowly added a solution of benzaldehyde (90 mmol) in THF (10 mL) at 0 °C. Then, the additional THF (10 mL) was added, and the reaction mixture was stirred for 5 h at room temperature. The reaction was quenched with sat. NaHCO₃aq (70 mL) at 0 °C, extracted with Et₂O (50 x 3 mL), washed with sat. NaHCO₃aq (50 x 1 mL) and brine (50 x 1 mL), dried (MgSO₄) and the solvent removed in vacuo. The residue was purified by distillation to give the product **4** (5.9 g, 32%). The compound **4** was reported (ref. P. Claus, A. Brückner, C. Mohr and H. Hofmeister, *J. Am. Chem. Soc.*, **2000**, *122*, 11348.).

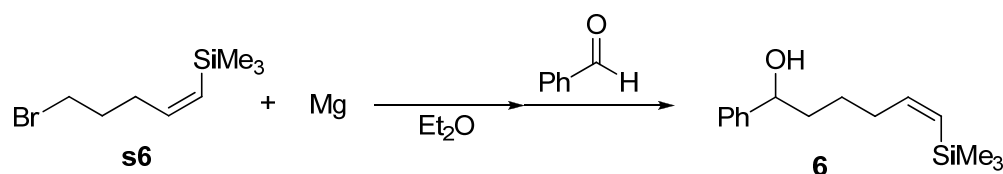
Synthesis of 6.



To a solution of PPh₃ (133 mmol) and CBr₄ (133 mmol) in Et₂O (160 mL) at room temperature was added **s4** (120 mmol, commercially available) and stirring continued at room temperature for 2 days. The resulting suspension was filtered, dried (MgSO₄) and the solvent removed in vacuo. The residue was purified by column chromatography to give the product **s5** (19.7 g, 75%). The compound **s5** was reported (ref. L. -M. Yang, L. -F. Huang and T. -Y. Luh, *Org. Lett.*, **2004**, *6*, 1461.).



Hexane was removed from DIBALH (108 mL, 1 M in hexane) in vacuo, and Et₂O (90 mL) was added. To this solution of DIBALH in Et₂O was added **s5** at room temperature, and the reaction mixture was stirred at room temperature. Then, the reaction was quenched at 0 °C with 3 N HCl, extracted with Et₂O (50 x 3 mL), washed with sat. NaHCO₃aq (50 x 3 mL) and brine (50 x 1 mL), dried (MgSO₄) and the solvent removed in vacuo. The residue was purified by column chromatography to give the product **s6** (12.6 g, 63%). IR: (neat) 1608 (C=C), 1250 (HBrC-H) cm⁻¹; ¹H NMR: (400 MHz, CDCl₃) 6.23 (dt, *J* = 14.0, 7.6 Hz, 1H, 2-H), 5.56 (d, *J* = 14.0 Hz, 1H, 1-H), 3.42 (t, *J* = 6.8 Hz, 2H, 5-H₂), 2.27 (dt, *J* = 7.6, 6.8 Hz, 2H, 3-H₂), 1.95 (tt, *J* = 6.8, 6.8 Hz, 2H, 4-H₂), 0.13 (s, 9H, Si-CH₃ x 3); ¹³C NMR: (100 MHz, CDCl₃) 146.4 (d, C-2), 130.8 (d, C-1), 33.2 (t, C-5), 32.7 (t, C-4), 31.9 (t, C-3), 0.18 (q, Si-CH₃); MS: (EI, 70 eV) *m/z*, 207 (M⁺ + 2 - Me, 12), 205 (M⁺ - Me, 12), 139 (98), 137 (100); HRMS: (EI, 70 eV) Calculated (C₇H₁₄⁸¹BrSi) 207.0022 (M⁺ + 2 - Me) Found: 207.0029.



To a suspension of Mg (75 mmol) in THF (5 mL) was slowly added a solution of **s6** (60 mmol) in THF (10 mL). Then, the additional THF (30 mL) was added, and the reaction mixture was stirred for 2 h at room temperature. To the resulted mixture was slowly added a solution of benzaldehyde (60 mmol) in THF (10 mL) at 0 °C. Then, the additional THF (10 mL) was added, and the reaction mixture was stirred for 5 h at room temperature. The reaction was quenched with sat. NaHCO₃aq (70 mL) at 0 °C, extracted with Et₂O (50 x 3 mL), washed with sat. NaHCO₃aq (50 x 1 mL) and

brine (50 x 1 mL), dried (MgSO₄) and the solvent removed in vacuo. The residue was purified by column chromatography and distillation to give the product **6** (9.6 g, 64%). bp: 112-117 °C/0.2 Torr; IR: (neat) 1604 (C=C), 3348 (OH) cm⁻¹; ¹H NMR: (400 MHz, CDCl₃) 7.38-7.26 (m, 5H, aroma), 6.26 (dt, *J* = 14.0, 7.2 Hz, 1H, 5-H), 5.48 (d, *J* = 14.0 Hz, 1H, 6-H), 4.69-4.65 (m, 1H, 1-H), 2.15 (td, *J* = 8.4, 7.2 Hz, 2H, 4-H₂), 1.86-1.69 (m, 2H, 2-H₂), 1.83 (d, *J* = 3.2 Hz, 1H, OH, D₂O exchangeable) 1.58-1.33 (m, 2H, 3-H₂) 0.09 (s, 9H, Si-CH₃ x 3); ¹³C NMR: (100 MHz, CDCl₃) 148.5 (d, C-5), 144.7 (s, C-*i*), 129.4 (d, C-6), 128.5 (d, C-*m*), 127.6 (d, C-*p*), 125.9 (d, C-*o*), 74.6 (d, C-1), 38.6 (t, C-2), 33.2 (t, C-4), 25.9 (t, C-3), 0.2 (q, Si-CH₃); MS: (CI, 70 eV) *m/z*, 249 (M + 1, 4), 231 (M + 1 - H₂O, 28), 157 (M - H₂O - SiMe₃, 100), 73 (SiMe₃⁺, 71); HRMS: (CI, 70 eV) Calculated (C₁₅H₂₅OSi) 249.1675 (M + 1) Found: 249.1683; Analysis: C₁₅H₂₄OSi (248.44) Calcd: C 72.52, H 9.74. Found: C 72.36, H 9.87

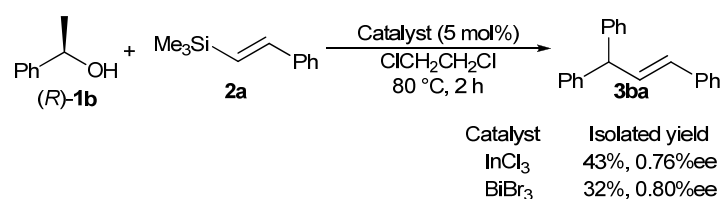
Typical Procedure for the coupling of **1a** with **2a** (Table 1, Entry 2)

To a mixture of InCl₃ (0.05 mmol) and benzhydrol (**1a**, 1.0 mmol) in dichloroethane (1 mL) was added *E*-2-phenyl-1-trimethylsilylethylene (**2a**, 2.0 mmol) under nitrogen. The reaction mixture was stirred under the reaction conditions noted in the text. The resulting mixture was poured into Et₂O (10 mL) and aqueous NaHCO₃ (10 mL). The solution was extracted with Et₂O and the organic layer was dried over MgSO₄. The evaporation of the ether solution gave the crude product which was analyzed by NMR. The details of further purification performed for the new compounds are described in Product Data.

Intramolecular cyclization reactions (Scheme 2)

To a mixture of InCl₃ (0.1 mmol) and **4** (1.0 mmol) in dichloromethane (200 mL) was stirred under the reaction conditions noted in the text. The resulting mixture was poured into Et₂O (10 mL) and aqueous NaHCO₃ (10 mL). The solution was extracted with Et₂O and the organic layer was dried over MgSO₄. The evaporation of the ether solution gave the crude product which was analyzed by NMR. The details of further purification performed for the new compounds are described in Product Data.

The coupling reaction using the optically active alcohol

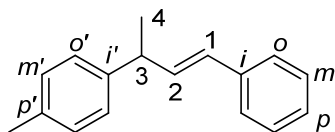


To a mixture of catalyst (0.05 mmol) and (*R*)-(+)-1-phenylethanol ((*R*)-**1b**, 1.0 mmol, Tokyo Chemical Industry Co., Ltd.) in 1,2-dichloroethane (1 mL) was added *E*-2-phenyl-1-trimethylsilyl ethylene (**2a**, 2.0 mmol) under nitrogen. The reaction mixture was stirred for 2 h at 80 °C. The resulting mixture was poured into Et₂O (10 mL) and aqueous NaHCO₃ (10 mL). The solution was extracted with Et₂O and the organic layer was dried over MgSO₄. The evaporation of the ether solution gave the crude product. The crude mixture was purified by column chromatography (hexane/ethyl acetate, 95/5) on silica gel (70-230 mesh) to give the pure product. Enantiomer excess was analyzed by HPLC (OD-H, hexane/isopropanol, 99.8/0.02, 0.9 mLmin⁻¹, 215 nm).

Product Data.

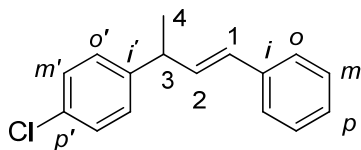
The spectral data of **3aa**⁶, **3ba**⁶, **3ca**⁷, **3fa**⁸, **3ga**⁸, **3ha**⁷, **3ia**⁹, **3ja**¹⁰, **3ka**¹¹, **3la**¹², **3ma**¹³, **3af**¹⁴, **5**¹⁵ and **7**¹⁶ were in an excellent agreement with the reported data. The spectral data for the products **3da**, **3ea**, **3ab**, **3ac**, **3ad** and **3ag** are shown below.

***E*-3-(4-Methylphenyl)-1-phenyl-1-butene (3da)**



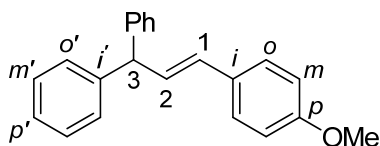
According to the typical procedure, this compound was produced from InCl_3 , 1-(4-methylphenyl)-ethanol and *E*-2-phenyl-1-trimethylsilylethene to give the pure product after column chromatography (hexane/ethyl acetate, 95/5) on silica gel (70-230 mesh). IR: (neat) 1647 ($\text{C}=\text{C}$) cm^{-1} ; ^1H NMR: (400 MHz, CDCl_3) 7.35 (d, $J = 7.7$ Hz, 2H, *o*-H), 7.28 (dd, $J = 7.7, 7.7$ Hz, 2H, *m*-H), 7.24-7.11 (m, 5H, aroma), 6.41 (d, $J = 16.8$ Hz, 1H, 1-H), 6.36 (dd, $J = 16.8, 6.0$ Hz, 1H, 2-H), 3.60 (qd, $J = 7.2, 6.0$ Hz, 1H, 3-H), 2.33 (s, 3H, Ar- CH_3), 1.45 (d, $J = 7.2$ Hz, 3H, 4- H_3); ^{13}C NMR: (100 MHz, CDCl_3) 142.6 (s, C- i'), 137.6 (s, C- i), 135.7 (s, C- p'), 135.4 (d, C-2), 129.1 (d, C- m'), 128.4 (d, C- m), 128.3 (d, C-1), 127.1 (d, C- o'), 127.0 (d, C- p), 126.1 (d, C- o), 42.1 (d, C-3), 21.2 (q, C-4), 21.0 (Ar- CH_3); MS: (EI, 70 eV) m/z , 222 (M^+ , 58), 207 ($\text{M}^+ - \text{Me}$, 100), 129 (43), 115 (42), 91 (Me-Ph^+ , 23); HRMS: (EI, 70 eV) Calculated ($\text{C}_{17}\text{H}_{18}$) 222.1409 (M^+) Found: 222.1411; Analysis: $\text{C}_{17}\text{H}_{18}$ (222.32) Calcd: C 91.84, H 8.16. Found: C 91.57, H 8.18.

***E*-3-(4-Chlorophenyl)-1-phenyl-1-butene (3ea)**



According to the typical procedure, this compound was produced from InCl_3 , 1-(4-chlorophenyl)-ethanol and *E*-2-phenyl-1-trimethylsilylethene to give the pure product after column chromatography (hexane/ethyl acetate, 95/5) on silica gel (70-230 mesh). IR: (neat) 1647 ($\text{C}=\text{C}$), 1095 (Ar-Cl) cm^{-1} ; ^1H NMR: (400 MHz, CDCl_3) 7.36-7.15 (m, 9H, aroma), 6.40 (d, $J = 15.9$ Hz, 1H, 1-H), 6.32 (dd, $J = 15.9, 6.4$ Hz, 1H, 2-H), 3.62 (qd, $J = 7.2, 6.4$ Hz, 1H, 3-H), 1.44 (d, $J = 7.2$ Hz, 3H, 4- H_3); ^{13}C NMR: (100 MHz, CDCl_3) 144.0 (s, C- i'), 137.3 (s, C- i), 134.5 (d, C-2), 131.9 (s, C- p'), 128.9 (d), 128.7 (d, C-1), 128.55 (d), 128.51 (d), 127.2 (d), 126.1 (d, C- o), 41.9 (d, C-3), 21.1 (q, C-4); MS: (EI, 70 eV) m/z , 244 ($\text{M}^+ + 2$, 21), 242 (M^+ , 66), 229 (29), 227 ($\text{M}^+ - \text{Me}$, 88), 207 ($\text{M}^+ - \text{Cl}$, 100), 192 (54), 149 (34), 129 (35), 115 (48), 91 (33); HRMS: (EI, 70 eV) Calculated ($\text{C}_{16}\text{H}_{15}\text{Cl}$) 242.0862 (M^+), Found: 242.0862; Analysis: $\text{C}_{16}\text{H}_{15}\text{Cl}$ (242.74) Calcd: C, 79.17; H, 6.23; Cl, 14.61. Found: C, 78.94; H, 6.22; Cl, 14.51.

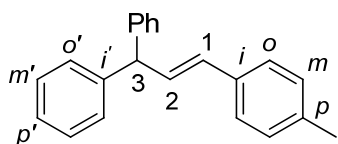
***E*-1-(4-Methoxyphenyl)-3,3-diphenyl-1-propene (3ab)**



According to the typical procedure, this compound was produced from InCl_3 , benzhydrol and

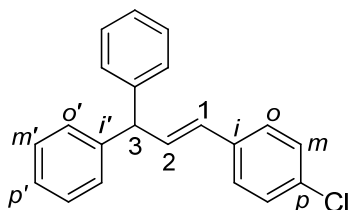
E-2-(4-methoxyphenyl)-1-trimethylsilylene to give the pure product after column chromatography (hexane/ethyl acetate, 95/5) on silica gel (70-230 mesh). Further purification was performed by GPC. mp: 91-93 °C; IR: (KBr) 1643 (C=C), 1246 (Ar-OMe) cm^{-1} ; ^1H NMR: (400 MHz, CDCl_3) 7.33-7.22 (m, 12H, aroma), 6.83 (d, $J = 8.8$ Hz, 2H, *m*-H), 6.53 (dd, $J = 15.6, 7.6$ Hz, 1H, 2-H), 6.29 (d, $J = 15.6$ Hz, 1H, 1-H), 4.87 (d, $J = 7.6$ Hz, 1H, 3-H), 3.80 (s, 3H, OCH_3); ^{13}C NMR: (100 MHz, CDCl_3) 159.0 (s, C-*p*), 143.7 (s, C-*i'*), 130.8 (d, C-1), 130.4 (d, C-2), 130.1 (s, C-*i*), 128.6 (d, C-*o'*), 128.4 (d, C-*m'*), 127.4 (d, C-*o*), 126.3 (d, C-*p'*), 113.9 (d, C-*m*), 55.3 (q, C- OCH_3), 54.2 (d, C-3); MS: (EI, 70 eV) m/z , 301 (23), 300 (M^+ , 100), 299 (33), 223 (24), 222 (25), 209 (26), 192 (50), 191 (40), 178 (26), 121 (30), 115 (27); HRMS: (EI, 70 eV) Calculated ($\text{C}_{22}\text{H}_{20}\text{O}$) 300.1514(M^+) Found: 300.1516; Analysis: $\text{C}_{22}\text{H}_{20}\text{O}$ (300.39) Calcd: C 87.96, H 6.71. Found: C 87.69, H 6.67.

***E*-1-(4-Methylphenyl)-3,3-diphenyl-1-propene (3ac)**



According to the typical procedure, this compound was produced from InCl_3 , benzhydrol and *E*-2-(4-methylphenyl)-1-trimethylsilylene to give the pure product after column chromatography (hexane/ethyl acetate, 95/5) on silica gel (70-230 mesh). IR: (neat) 1650 (C=C) cm^{-1} ; ^1H NMR: (400 MHz, CDCl_3) 7.33-7.09 (m, 14H, aroma), 6.62 (dd, $J = 16.8, 7.6$ Hz, 1H, 2-H), 6.32 (d, $J = 16.8$ Hz, 1H, 1-H), 4.88 (d, $J = 7.6$ Hz, 1H, 3-H), 2.32 (s, 3H, Ar- CH_3); ^{13}C NMR: (100 MHz, CDCl_3) 143.6 (s, C-*i'*), 137.1 (s, C-*p*), 134.4 (s, C-*i*), 131.5 (d, C-2), 131.2 (d, C-1), 129.2 (d, C-*m*), 128.6 (d, C-*o'*), 128.4 (d, C-*m'*), 126.4 (d, C-*p'*), 126.2 (d, C-*o*), 54.1 (d, C-3), 21.2 (q, Ar- CH_3); MS: (EI, 70 eV) m/z , 285 ($\text{M}^+ + 1$, 22), 284 (M^+ , 97), 283 (43), 282 (88), 270 ($\text{M}^+ + 1 - \text{Me}$, 21), 269 ($\text{M}^+ - \text{Me}$, 91), 207 (21), 206 (48), 205 (29), 193 (44), 192 (100), 191 (99), 179 (29), 178 (47), 165 (46) 115 (40), 91 (Me-Ph^+); HRMS: (EI, 70 eV) Calculated ($\text{C}_{22}\text{H}_{20}$) 284.1565 (M^+) Found: 284.1569.

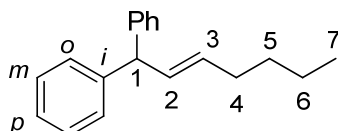
***E*-1-(4-Chlorophenyl)-3,3-diphenyl-1-propene (3ad)**



According to the typical procedure, this compound was produced from BiBr_3 , benzhydrol and *E*-2-(4-chlorophenyl)-1-trimethylsilylene to give the pure product after column chromatography (hexane/ethyl acetate, 90/10) on silica gel (70-230 mesh). Further purification was performed by GPC. mp: 66-69 °C; IR: (KBr) 1650 (C=C), 1095 (Ar-Cl) cm^{-1} ; ^1H NMR: (400 MHz, CDCl_3) 7.34-7.22 (m, 14H, aroma), 6.65 (dd, $J = 16, 7.6$ Hz, 1H, 2-H), 6.29 (d, $J = 16$ Hz, 1H, 1-H), 4.89 (d,

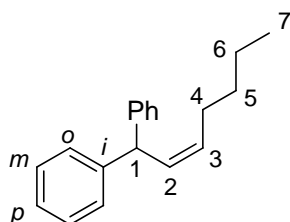
$J = 7.6$ Hz, 1H, 3-H); ^{13}C NMR: (100 MHz, CDCl_3) 143.2 (s, C- i'), 135.7 (s, C- i), 133.3 (d, C-2), 132.9 (s, C- p), 130.2 (d, C-1), 128.63 (d, aroma), 128.61 (d, C- o'), 128.5 (d, aroma), 127.5 (d, C- o), 126.5 (d, aroma), 54.1 (d, C-3); MS: (EI, 70 eV) m/z , 306 ($\text{M}^+ + 2$, 34), 305 ($\text{M}^+ + 1$, 26), 304 (M^+ , 100), 269 ($\text{M}^+ - \text{Cl}$, 67), 226 (47), 192 (67), 191 (89), 189 (22), 179 (36), 178 (49), 165 (45), 115 (23), 91 (26); HRMS: (EI, 70 eV) Calculated ($\text{C}_{21}\text{H}_{17}\text{Cl}$) 304.1019 (M^+) Found: 304.1016; Analysis: $\text{C}_{21}\text{H}_{17}\text{Cl}$ (304.81) Calcd: C 82.75, H 5.62, Cl 11.63. Found: C 82.94, H 5.70, Cl, 11.49.

***E*-1,1-Diphenyl-2-heptene (3ag)**



According to the typical procedure, this compound was produced from InCl_3 , benzhydrol and *E*-1-trimethylsilyl-1-hexene to give the pure product after column chromatography (hexane/ethyl acetate, 95/5) on silica gel (70-230 mesh). Further purification was performed by GPC. IR: (neat) 1685 ($\text{C}=\text{C}$) cm^{-1} ; ^1H NMR: (400 MHz, CDCl_3) 7.29 (t, $J = 7.2$ Hz, 4H, m -H), 7.21-7.17 (m, 6H, aroma), 5.88 (dd, $J = 15.2, 7.6$ Hz, 1H, 2-H), 5.43 (dt, $J = 15.2, 6.8$ Hz, 1H, 3-H), 4.67 (d, $J = 7.6$ Hz, 1H, 1-H), 2.07 (td, $J = 7.2, 6.8$ Hz, 2H, 4- H_2), 1.54-1.30 (m, 4H, 5- H_2 and 6- H_2) 0.89 (t, $J = 7.2$ Hz, 7- H_3); ^{13}C NMR: (100 MHz, CDCl_3) 144.3 (s, C- i), 132.5 (d, C-3), 132.2 (d, C-2), 128.5 (d, C- o), 128.3 (d, C- m), 126.1 (d, C- p), 54.0 (d, C-1), 32.3 (t, C-4), 31.5 (t), 22.3 (t), 13.9 (q, C-7); MS: (EI, 70 eV) m/z , 250 (M^+ , 41), 193 ($\text{M}^+ - \text{Bu}$, 100), 180 (27), 115 (34), 91 (22); HRMS: (EI, 70 eV) Calculated ($\text{C}_{19}\text{H}_{22}$) 250.1722 (M^+) Found: 250.1721.

***Z*-1,1-Diphenyl-2-heptene (3ah)**



According to the typical procedure, this compound was produced from InCl_3 , benzhydrol and *Z*-1-trimethylsilyl-1-hexene to give the pure product after column chromatography (hexane/ethyl acetate, 95/5) on silica gel (70-230 mesh) and distillation. bp: 138-140 $^\circ\text{C}/0.5$ mmHg; IR: (neat) 1655 ($\text{C}=\text{C}$) cm^{-1} ; ^1H NMR: (400 MHz, CDCl_3) 7.28 (t, 7.2 Hz, 4H, m -H), 7.20-7.17 (m, 6H, aroma), 5.85 (dd, $J = 10.6, 9.7$ Hz, 1H, 2-H), 5.69 (dt, $J = 10.6, 7.00$, 1H, 3-H), 5.00 (d, $J = 9.7$ Hz, 1H, 1-H), 2.16 (, $J = 7.0, 7.0$ Hz, 2H, 4- H_2), 1.54-1.30 (m, 4H, 5- H_2 and 6- H_2), 0.87 (t, 3H, $J = 7.24$ Hz, 7- H_3); ^{13}C NMR: (100 MHz, CDCl_3) 144.7 (s, C- i), 131.8 (d, C-2), 130.7 (d, C-3), 128.4 (d), 128.3 (d), 126.1 (d, C- p), 48.4 (d, C-1), 31.7 (t), 27.1 (t, C-4), 22.4 (t), 13.7 (q, C-7); MS: (EI, 70 eV) m/z , 250 (M^+ , 29), 193 ($\text{M}^+ - \text{Bu}$, 100), 180 (21), 115 (37), 91 (25); HRMS: (EI, 70 eV) Calculated ($\text{C}_{19}\text{H}_{22}$)

250.1722 (M⁺) Found: 250.1733.

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