



Supporting Information

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Consecutive Double 1,2-Migration of Two Different Groups in Silyldichloromethylcuprates

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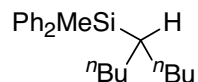
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Instrumentation and Materials

^1H NMR (300 MHz) and ^{13}C NMR (75.3 MHz) spectra were taken on a Varian GEMINI 300 spectrometer in CDCl_3 as a solvent, and chemical shifts were given in δ value with tetramethylsilane as an internal standard. IR spectra were determined on a SHIMADZU FTIR-8200PC spectrometer. Mass spectra were determined on a JEOL JMS-700 spectrometer. TLC analyses were performed on commercial glass plates bearing 0.25 mm layer of Merk Silica gel 60F₂₅₄. Column chromatography was done with silica gel (Wakogel 200 mesh). The analyses were carried out at the Elemental Analysis Center of Kyoto University. Tetrahydrofuran (THF) was freshly distilled from sodium benzophenone ketyl before use. Grignard reagents were prepared from the corresponding alkyl halide and Mg turning (Nacalai tesque, INC). Unless otherwise noted, materials obtained from commercial suppliers were used without further purification.

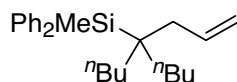
Experimental and Compound Data

General Procedure for Consecutive Double Alkylation



*n*BuLi (0.31 mL, 1.6 M solution in hexane, 0.50 mmol) was added to a solution of $\text{Ph}_2\text{MeSiCHCl}_2$ (**1**, 141 mg, 0.5 mmol) in THF (5 mL) at -78°C , and the mixture was stirred for 30 min. To the resulting solution at -78°C was added $n\text{Bu}_2\text{CuCN}(\text{MgBr})_2$ in THF, which was prepared by mixing *n*BuMgBr (1.25 mL, 1.0 M solution in THF, 1.25 mmol) and $\text{CuCN}\cdot 2\text{LiCl}$ (0.55 mL, 1.0 M solution in THF, 0.55 mmol) at 0°C . After stirring for 5 min, the mixture was allowed to warm gradually to 0°C . The mixture was stirred for 1 h at 0°C , and then the reaction was quenched with diluted aqueous HCl (20 mL). The mixture was extracted with hexane (10 mL $\times$ 3), and the organic layers were dried over anhydrous Na_2SO_4 and concentrated in vacuo. Purification by silica gel column chromatography provided 5-(methyldiphenylsilyl)nonane (127 mg, 0.39 mmol) in 78% yield as a colorless oil: $R_f = 0.78$ (hexane/ethyl acetate = 10/1); IR (neat) 3069, 2856, 1952, 1880, 1817, 1466, 1252, 1111, 787, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.58 (s, 3H), 0.81 (t, $J = 7.2$ Hz, 6H), 1.12–1.44 (m, 12H), 1.48–1.62 (m, 1H), 7.31–7.42 (m, 6H), 7.51–7.59 (m, 4H); ^{13}C NMR (CDCl_3) δ –5.06, 14.10, 23.02, 23.61, 29.65, 31.77, 127.54, 128.76, 134.63, 137.26. Found: C, 81.19; H, 9.71%. Calcd for $\text{C}_{22}\text{H}_{32}\text{Si}$: C, 81.41; H, 9.94%.

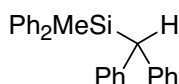
4-Butyl-4-(methyldiphenylsilyl)-1-octene



$R_f = 0.66$ (hexane/ethyl acetate = 10/1); IR (neat) 2957, 2858, 1636, 1427, 1254, 1105, 999, 910, 784, 737, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.66 (s, 3H), 0.78 (t, $J = 6.9$ Hz, 6H), 1.06–1.24 (m, 8H), 1.44–1.60 (m, 4H), 3.32 (d, $J = 7.5$ Hz, 2H), 4.88–4.98 (m, 2H), 5.75

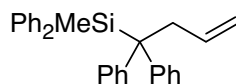
(ddt, $J = 9.3, 16.5, 7.5$ Hz, 1H), 7.30–7.38 (m, 6H), 7.60–7.66 (m, 4H); ^{13}C NMR (CDCl_3) δ –3.21, 13.83, 23.56, 26.64, 29.14, 35.75, 40.74, 116.69, 127.59, 128.78, 135.53, 136.21, 137.63. Found: C, 82.13; H, 9.72%. Calcd for $\text{C}_{25}\text{H}_{36}\text{Si}$: C, 82.35; H, 9.95%. HRMS (m/z) Found: 364.2571. Calcd for $\text{C}_{25}\text{H}_{36}\text{Si}$: 364.2586.

(Methyldiphenylsilyl)diphenylmethane



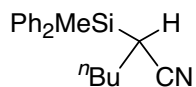
$R_f = 0.59$ (hexane/ethyl acetate = 10/1); IR (neat) 3024, 1958, 1886, 1821, 1597, 1493, 1427, 1254, 1111, 999, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.53 (s, 3H), 4.14 (s, 1H), 7.05–7.21 (m, 10H), 7.24–7.40 (m, 10H); ^{13}C NMR (CDCl_3) δ –3.58, 44.31, 125.23, 127.51, 128.06, 129.18, 129.26, 135.21, 135.62, 141.78. Found: C, 85.39; H, 6.77%. Calcd for $\text{C}_{26}\text{H}_{24}\text{Si}$: C, 85.66; H, 6.64%.

1-(Methyldiphenylsilyl)-1,1-diphenyl-3-butene



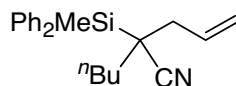
$R_f = 0.67$ (hexane/ethyl acetate = 10/1); IR (neat) 3053, 2957, 1599, 1493, 1427, 1254, 1105, 912, 791, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.46 (s, 3H), 3.24 (d, $J = 6.6$ Hz, 2H), 4.89 (dd, $J = 2.1, 10.5$ Hz, 1H), 4.95 (dd, $J = 2.1, 17.1$ Hz, 1H), 5.58 (ddt, $J = 10.5, 17.1, 6.6$ Hz, 1H), 7.11–7.23 (m, 10H), 7.23–7.28 (m, 4H), 7.28–7.41 (m, 6H); ^{13}C NMR (CDCl_3) δ –3.12, 40.63, 45.66, 117.11, 125.22, 127.39, 127.45, 127.51, 128.05, 128.98, 129.17, 129.25, 129.93, 134.87, 135.20, 135.85, 135.94, 141.77, 143.42. HRMS (m/z) Found: 404.1954. Calcd for $\text{C}_{29}\text{H}_{28}\text{Si}$: 404.1960.

General procedure for Consecutive Alkylation–Cyanation



*n*BuLi (0.31 mL, 1.6 M solution in hexane, 0.50 mmol) was added to a solution of Ph₂MeSiCHCl₂ (**1**, 141 mg, 0.5 mmol) in THF (5 mL) dropwise at –78 °C, and the mixture was stirred for 30 min. To the resulting solution at –78 °C was added a solution of *n*Bu₂CuLi•LiCN in THF, which was prepared by mixing *n*BuLi (0.78 mL, 1.6 M solution in THF, 1.25 mmol) and CuCN•2LiCl (0.55 mL, 1.0 M solution in THF, 0.55 mmol) at 0 °C. After stirring for 5 min, the mixture was allowed to warm gradually to 0 °C. The mixture was stirred for 1 h at 0 °C, and the reaction was quenched with diluted aqueous HCl (20 mL). The mixture was extracted with ethyl acetate (10 mL × 3), and the organic layers were dried over anhydrous Na₂SO₄ and concentrated in vacuo. Purification by silica gel column chromatography provided 2-(methyldiphenylsilyl)hexanenitrile (116 mg, 0.39 mmol) in 79% yield as a colorless oil: *R*_f = 0.38 (hexane/ethyl acetate = 10/1); IR (neat) 3072, 2932, 2860, 2222, 1429, 1258, 1115, 793, 729, 700 cm^{–1}; ¹H NMR (CDCl₃) δ 0.76 (s, 3H), 0.85 (t, *J* = 7.2 Hz, 3H), 1.12–1.72 (m, 6H), 2.30 (dd, *J* = 4.2, 10.8 Hz, 1H), 7.38–7.51 (m, 6H), 7.56–7.66 (m, 4H); ¹³C NMR (CDCl₃) δ –5.46, 13.90, 17.84, 22.01, 26.87, 32.01, 121.88, 128.06, 128.10, 130.16, 132.21, 132.50, 134.55. Found: C, 77.78; H, 7.80%. Calcd for C₁₉H₂₃NSi: C, 77.76; H, 7.90%.

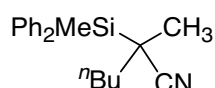
2-Butyl-2-(methyldiphenylsilyl)-pent-4-enenitrile



*R*_f = 0.41 (hexane/ethyl acetate = 10/1); IR (neat) 2936, 2214, 1962, 1890, 1827, 1639, 1429, 1259, 1113, 922, 793, 729 cm^{–1}; ¹H NMR (CDCl₃) δ 0.81 (s, 3H), 0.80 (t, *J* = 7.2 Hz, 3H), 1.13–1.30 (m, 2H), 1.30–1.50 (m, 2H), 1.50–1.72 (m, 2H), 2.29 (dd, *J* = 7.2, 14.1 Hz, 1H), 2.48 (dd, *J* = 7.2, 14.1 Hz, 1H), 5.02 (d, *J* = 16.8 Hz, 1H), 5.08 (d, *J* = 9.9 Hz,

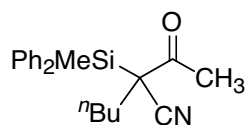
1H), 5.82 (ddt, $J = 9.9, 16.8, 7.2$ Hz, 1H), 7.38–7.49 (m, 6H), 7.70–7.76 (m, 4H); ^{13}C NMR (CDCl_3) δ –4.96, 13.82, 22.92, 27.39, 28.50, 32.71, 38.03, 118.74, 123.94, 128.03, 130.07, 132.50, 133.42, 134.61, 135.15, 135.19. Found: C, 79.34; H, 8.35%. Calcd for $\text{C}_{22}\text{H}_{27}\text{NSi}$: C, 79.22; H, 8.16%.

2-Methyl-2-(methyldiphenylsilyl)hexanenitrile



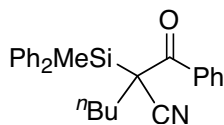
$R_f = 0.48$ (hexane/ethyl acetate = 5/1); IR (neat) 2936, 2214, 1429, 1259, 1113, 793, 729, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.76 (s, 3H), 0.86 (t, $J = 7.2$ Hz, 3H), 1.33 (s, 3H), 1.19–1.36 (m, 3H), 1.42–1.54 (m, 2H), 1.68–1.81 (m, 1H), 7.38–7.49 (m, 6H), 7.70–7.76 (m, 4H); ^{13}C NMR (CDCl_3) δ –6.27, 13.98, 19.28, 22.12, 22.83, 27.36, 34.08, 125.36, 128.03, 130.09, 132.11, 132.14, 135.22, 135.24. Found: C, 77.82; H, 8.22%. Calcd for $\text{C}_{20}\text{H}_{25}\text{NSi}$: C, 78.12; H, 8.19%.

2-Acetyl-2-(methyldiphenylsilyl)hexanenitrile



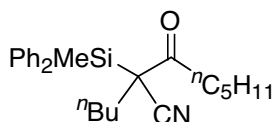
$R_f = 0.52$ (hexane/ethyl acetate = 5/1); IR (neat) 2930, 2205, 1632, 1429, 1286, 1121, 797, 738, 698 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.79 (s, 3H), 0.89 (t, $J = 7.2$ Hz, 3H), 1.24–1.38 (m, 3H), 1.39–1.51 (m, 2H), 2.02 (s, 3H), 2.21 (t, $J = 7.5$ Hz, 2H), 7.40–7.47 (m, 6H), 7.55–7.61 (m, 4H); ^{13}C NMR (CDCl_3) δ –1.49, 13.90, 18.92, 22.07, 22.23, 26.55, 30.26, 120.65, 128.11, 130.21, 130.50, 133.91, 134.17, 134.62, 162.85. HRMS (m/z) Found: 335.1702. Calcd for $\text{C}_{21}\text{H}_{25}\text{NOSi}$: 335.1705.

2-Benzoyl-2-(methyldiphenylsilyl)hexanenitrile



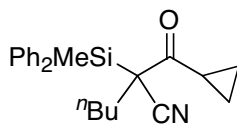
R_f = 0.41 (hexane/ethyl acetate = 5/1); IR (neat) 2959, 2206, 1746, 1429, 1258, 1115, 795, 698 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.40 (s, 3H), 0.88 (t, J = 7.2 Hz, 3H), 1.23–1.36 (m, 2H), 1.38–1.51 (m, 2H), 2.29 (dd, J = 7.5, 8.1 Hz, 2H), 7.32–7.38 (m, 6H), 7.43–7.50 (m, 4H); ^{13}C NMR (CDCl_3) δ -2.06, 13.91, 22.32, 27.80, 30.18, 97.32, 120.08. HRMS (m/z) Found: 397.1860. Calcd for $\text{C}_{26}\text{H}_{27}\text{NOSi}$: 397.1862.

2-Butyl-2-(methyldiphenylsilyl)-3-oxooctanenitrile



R_f = 0.66 (hexane/ethyl acetate = 5/1); IR (neat) 2959, 2206, 1728, 1626, 1429, 1261, 1121, 795, 737, 698 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.76 (s, 3H), 0.80 (t, J = 7.2 Hz, 3H), 0.86 (t, J = 7.2 Hz, 3H), 1.04–1.46 (m, 10H), 2.14 (t, J = 7.8 Hz, 2H), 2.29 (t, J = 7.5 Hz, 2H), 7.38–7.49 (m, 6H), 7.55–7.62 (m, 4H); ^{13}C NMR (CDCl_3) δ -1.88, 13.88, 13.97, 22.19, 22.33, 26.69, 27.20, 30.25, 31.07, 35.54, 95.51, 120.45, 128.04, 130.47, 134.00, 134.21, 167.05. HRMS (m/z) Found: 391.2332. Calcd for $\text{C}_{25}\text{H}_{33}\text{NOSi}$: 391.2331.

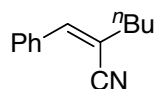
2-Cyclopropanecarbonyl-2-(methyldiphenylsilyl)hexanenitrile



R_f = 0.53 (hexane/ethyl acetate = 5/1); IR (neat) 2959, 2205, 1692, 1612, 1429, 1377, 1259, 1119, 793, 729, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.64–0.76 (m, 4H), 0.77 (s, 3H), 0.77 (t, J = 3.6 Hz, 3H), 1.01–1.15 (m, 2H), 1.23–1.36 (m, 2H), 1.87–1.94 (m, 1H), 1.97 (t, J = 7.8 Hz, 2H), 7.35–7.49 (m, 6H), 7.53–7.59 (m, 4H); ^{13}C NMR (CDCl_3) δ -2.14, 6.90, 13.82,

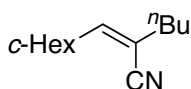
15.99, 22.15, 27.68, 30.22, 95.23, 120.64, 128.03, 130.39, 133.77, 134.53, 166.44. HRMS (m/z) Found: 361.1858. Calcd for $C_{23}H_{27}NOSi$: 361.1862.

(Z)-2-Butyl-3-phenylacrylonitrile



R_f = 0.54 (hexane/ethyl acetate = 10/1); IR (neat) 2959, 2208, 1730, 1448, 926, 750, 692 cm^{-1} ; 1H NMR ($CDCl_3$) δ 0.96 (t, J = 7.2 Hz, 3H), 1.33–1.47 (m, 2H), 1.65 (quint, J = 7.8 Hz, 2H), 2.41 (t, J = 7.8 Hz, 2H), 6.93 (s, 1H), 7.34–7.46 (m, 3H), 7.68–7.78 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 13.85, 21.92, 30.41, 36.04, 111.54, 118.75, 128.41, 128.66, 129.69, 133.72, 143.09. Found: C, 84.06; H, 8.20%. Calcd for $C_{13}H_{15}N$: C, 84.28; H, 8.16%. The stereochemistry was assigned by NOESY experiments. NOE (1H difference spectrum, 300MHz, $CDCl_3$) irradiation of δ 2.41 (CH_2)—enhancement of signals at δ 6.93 (CH, 1.6%); irradiation of δ 6.93 (CH)— enhancement of signals at δ 2.41 (CH_2 , 0.9%), δ 7.68–7.78 (Ph, 0.7%)

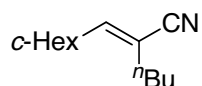
(Z)-2-Butyl-3-cyclohexylacrylonitrile (Z/E = 55/45)



R_f = 0.62 (hexane/ethyl acetate = 10/1); IR (neat) 2855, 2214, 1591, 1429, 1259, 1124, 858, 796, 698 cm^{-1} ; 1H NMR ($CDCl_3$) δ 0.91 (t, J = 7.5 Hz 3H), 1.20–1.24 (m, 4H), 1.24–1.42 (m, 4H), 1.45–1.55 (m, 2H), 1.62–1.78 (m, 6H), 2.18 (dt, J = 1.5, 6.9 Hz, 2H), 2.44–2.60 (m, 1H), 5.95 (d, J = 9.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 13.80, 21.77, 25.36, 25.71, 30.23, 32.28, 33.92, 40.72, 112.34, 117.84, 152.73. HRMS (m/z) Found: 191.1677. Calcd for $C_{13}H_{21}N$: 191.1674. The stereochemistry was assigned by NOESY experiments. NOE (1H difference spectrum, 300MHz, $CDCl_3$) irradiation of δ 2.18 (CH_2)—enhancement of signals at δ 5.95 (CH, 1.2%); irradiation of δ 5.95 (CH)— enhancement of signals at

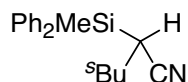
δ 2.18 (CH₂, 0.8%)

(*E*)-2-Butyl-3-cyclohexylacrylonitrile (*Z/E* = 55/45)



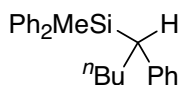
R_f = 0.55 (hexane/ethyl acetate = 10/1); IR (neat) 2855, 2214, 1591, 1429, 1259, 1124, 858, 797, 697 cm⁻¹; ¹H NMR (CDCl₃) δ 0.92 (t, J = 7.2 Hz, 3H), 1.04–1.43 (m, 8H), 1.43–1.80 (m, 8H), 2.19 (dt, J = 1.2, 7.2 Hz, 2H), 2.24–2.39 (m, 1H), 6.14 (d, J = 10.2 Hz, 1H); ¹³C NMR (CDCl₃) δ 13.87, 22.09, 25.39, 25.66, 28.39, 30.44, 31.98, 37.64, 113.12, 120.27, 152.78. HRMS (m/z) Found: 191.1680. Calcd for C₁₃H₂₁N: 191.1674. The stereochemistry was assigned by NOESY experiments. NOE (¹H difference spectrum, 300MHz, CDCl₃) irradiation of δ 6.14 (CH₂)— no enhancement of signals at δ 2.19 (CH₂, 0.9%)

2-(Methyldiphenylsilyl)-3-methylpentanenitrile



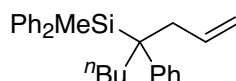
R_f = 0.37 (hexane/ethyl acetate = 10/1); IR (neat) 2964, 2220, 1429, 1115, 793, 732, 700 cm⁻¹; ¹H NMR (CDCl₃) δ 0.77 (t, J = 7.4 Hz, 3H), 0.80 (s, 3H), 0.81 (s, 3H), 0.86 (t, J = 7.4 Hz, 3H), 0.93 (d, J = 6.6Hz, 3H), 1.02 (d, J = 6.6 Hz, 3H), 1.20–1.77 (m, 6H), 2.38 (d, J = 4.5 Hz, 1H), 2.53 (d, J = 3.0 Hz, 1H), 7.36–7.51 (m, 12H), 7.56–7.62 (m, 4H), 7.63–7.69 (m, 4H); ¹³C NMR (CDCl₃) δ -4.41, -4.11, 11.70, 11.76, 18.12, 20.31, 23.60, 26.03, 27.78, 30.88, 33.21, 33.36, 120.33, 120.79, 128.07, 128.11, 128.14, 130.07, 130.12, 130.15, 132.65, 132.84, 133.39, 133.52, 134.50, 134.55. Found: C, 77.87; H, 8.08%. Calcd for C₁₉H₂₃NSi: C, 77.76; H, 7.90%.

Preparation of 1-(Methyldiphenylsilyl)-1-phenylpentane (**10**)



*n*BuLi (0.31 mL, 1.6 M solution in hexane, 0.5 mmol) was added to a solution of Ph₂MeSiCHCl₂ (**1**, 141 mg, 0.5 mmol) in THF (5 mL) dropwise at –78 °C, and the mixture was stirred for 30 min. To the resultant solution at –78 °C was added a solution of *n*BuCu in THF, which was prepared with *n*BuLi (0.34 mL, 1.6 M solution in THF, 0.55 mmol) and CuI•LiI (0.55 mL, 1.0 M solution in THF, 0.55 mmol) at –78 °C. After stirring for 10 min and an addition of PhMgBr (0.75 mL, 1.0 M solution in THF, 0.75 mmol), the mixture was allowed to warm gradually to –10 °C. The reaction was quenched with diluted aqueous HCl (20 mL). The mixture was extracted with ethyl acetate (10 mL × 3), and the organic layers were dried over anhydrous Na₂SO₄ and concentrated in vacuo. Purification by silica gel column chromatography provided 1-(methyldiphenylsilyl)-1-phenylpentane (**10**, 93 mg, 0.27 mmol) in 54% yield as a colorless oil: *R*_f = 0.72 (hexane/ethyl acetate = 10/1); IR (neat) 2957, 1956, 1882, 1818, 1599, 1427, 1252, 1111, 789, 700 cm^{–1}; ¹H NMR (CDCl₃) δ 0.42 (s, 3H), 0.77 (t, *J* = 6.6 Hz, 3H), 1.06–1.34 (m, 4H), 1.76–1.87 (m, 2H), 2.65 (dd, *J* = 5.1, 10.5 Hz, 1H), 6.86–6.92 (m, 2H), 7.04–7.19 (m, 4H), 7.24–7.49 (m, 6H), 7.50–7.58 (m, 3H); ¹³C NMR (CDCl₃) δ –5.22, 14.02, 22.45, 29.78, 31.43, 35.02, 124.48, 127.06, 127.43, 127.59, 127.78, 127.96, 128.36, 128.64, 128.91, 129.16, 134.54, 134.77, 135.16, 136.21, 142.34. HRMS (*m/z*) Found: 344.1963. Calcd for C₂₄H₂₈Si: 344.1960.

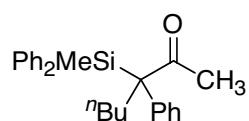
4-(Methyldiphenylsilyl)-4-phenyl-1-octene



*R*_f = 0.68 (hexane/ethyl acetate = 20/1); IR (neat) 2957, 1427, 1254, 1107, 784, 700 cm^{–1}; ¹H NMR (CDCl₃) δ 0.59 (s, 3H), 0.81 (t, *J* = 7.2 Hz, 3H), 1.16–1.33 (m, 4H), 1.94–2.14

(m, 2H), 2.73–2.90 (m, 2H), 4.93 (dd, $J = 1.5, 10.2$ Hz, 1H), 5.03 (dd, $J = 1.5, 17.1$ Hz, 1H), 5.65–5.81 (m, 1H), 6.88–6.94 (m, 2H), 7.06–7.18 (m, 3H), 7.22–7.31 (m, 4H), 7.32–7.42 (m, 6H); ^{13}C NMR (CDCl_3) δ –4.42, 14.12, 23.69, 25.94, 33.53, 36.34, 38.48, 116.52, 124.35, 127.25, 128.15, 128.88, 135.36, 135.58, 136.10, 143.95. HRMS (m/z) Found: 384.2258. Calcd for $\text{C}_{27}\text{H}_{32}\text{Si}$: 384.2273.

3-(Methyldiphenylsilyl)-3-phenylheptan-2-one



$R_f = 0.48$ (hexane/ethyl acetate = 10/1); IR (neat) 2957, 1659, 1429, 1217, 1119, 986, 793, 700 cm^{-1} ; ^1H NMR (CDCl_3) δ 0.56 (s, 3H), 0.80 (t, $J = 7.2$ Hz, 3H), 1.13–1.37 (m, 6H), 1.99 (s, 3H), 6.84–6.90 (m, 2H), 7.13–7.19 (m, 3H), 7.22–7.36 (m, 5H), 7.37–7.42 (m, 3H), 7.56–7.60 (m, 2H); ^{13}C NMR (CDCl_3) δ –3.49, 13.85, 23.65, 28.05, 33.46, 59.01, 125.75, 127.23, 127.37, 127.66, 128.62, 129.02, 134.78, 135.47, 135.89, 136.01, 139.61, 212.33. HRMS (m/z) Found: 386.2071. Calcd for $\text{C}_{26}\text{H}_{30}\text{OSi}$: 386.2066.

