A Concise and Atom-Economical Suzuki-Miyaura Coupling Reaction Using Unactivated Trialkyl- and Triarylboranes with Aryl Halides

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General Information. Commercial reagents and anhydrous solvents were used without further purification. All reactions were conducted under N₂ atmosphere, and monitored by High Performance Liquid Chromatography (HPLC) (using Ascentis Express C18 2.7 μ m, 100 mm $\times$ 4.6 mm) or GC. Concentration *in vacuum* refers to removal of the solvent using a rotary evaporator at reduced pressure (10-20 torr) with water bath. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker Avance 400 or 500 NMR Spectrometer with chemical shifts reported in ppm relative to the residual CDCl₃. High resolving power accurate mass measurements were acquired by use of a Bruker Daltonics 7T Fourier transform ion cyclotron resonance (FTICR) mass spectrometer.

General Suzuki-Miyaura cross-coupling procedure with commercially available trialkyl or triarylboranes (A):

To a flask charged aryl bromide (1 equiv), trialkylborane in THF (1M, 0.40 equiv) or triphenylborane (0.40 equiv), Pd₂dba₃•DCM (0.0125 equiv), *n*BuAd₂P (0.05 equiv), K₃PO₄ (2 equiv) and toluene (10 volume) and degassed water (1 volume) under N₂, the reaction mixture were heated to 100 °C under N₂ and stirred until HPLC shows full consumption of aryl bromide. The reaction mixture was cooled to rt and added water (5 mL) and extracted with EtOAc (10 mL), cut the layer and back extract the aqueous layer once with EtOAc (10 mL). The combined organic layers were dried, then assay by HPLC with desired product standard for yield or concentrated down and purified by flash chromatography.

Compound **3a-3i**, **4b** and **4l** were prepared based on above procedure A with commercially available Et₃B, *n*-Bu₃B, *sec*-Bu₃B and Ph₃B. All spectral data of isolated coupling products matched that of the commercial reagent.

General Suzuki-Miyaura cross-coupling procedure with trialkylboranes generated in situ via hydroboration of terminal alkenes (B):

To a flask charged BH₃•THF (1M, 0.45 equiv) under N₂, cooled to 0 °C and added terminal alkene (1.2 equiv) dropwise, aged the mixture at rt for 1 h. Then degassed water (0.3 mL) was added to the flask to quench the excess amount of BH₃ in the reaction mixture. K₃PO₄ (2 equiv), toluene (3 mL), aryl halide (0.30 g, 1 equiv), Pd₂dba₃•DCM (0.0125 equiv) and Ruphos (0.05 equiv) were added under N₂, the reaction mixture were heated up to 100 °C and aged under N₂ for ~20 h. The reaction mixture was cooled down to rt and added water (10 mL) and extracted with EtOAc (20 mL). The aqueous phase was extracted again with EtOAc (10 mL). The combined organic layers were dried, concentrated and purified by flash chromatography.

General procedure for the kinetics studies (the experiments were set up and run in glove box):

To a 40 mL vial was charged bromobenzene (8.0 mmol, 1 equiv), K₃PO₄ (16.0 mmol, 2 equiv), 1 M tri-*n*-butylborane in THF solution (2.8 mmol, 0.35 equiv; or 5.6 mmol, 0.70 equiv) or *n*-butylboronic acid (8.4 mmol, 1.05 equiv), Pd(OAc)₂ (0.20 mmol, 0.025 equiv), *n*-BuAd₂P (0.40 mmol, 0.05 equiv), degassed water (1.6 mL). Then, toluene was charged to fill up the reaction mixture to a total of 16 mL, which was sealed with a rubber cap. The reaction mixture were stirred and heated to 90 °C. 50 μ L of solution was

taken from the reaction mixture for every 15 min or every 30 min. The solution was diluted with 5 mL of acetonitrile for HPLC.

Figure 1. Plot of conversion versus time for 0.35 equiv or 0.70 equiv of tri-*n*-butylborane or 1.05 equiv of *n*-butylboronic acid with 1.0 equiv of bromobenzene (0.5 M), 2.5 mol % of Pd(OAc)₂ and 5 mol % of *n*-BuAd₂P in 10:1 of toluene/water in the Suzuki-Miyaura reaction at 90 °C. The reaction was run in glove box instead of in a fume hood for a typical reaction.

Preparation of 1,3-diphenylpropane (**5a**):

5a was prepared based on above procedure B, purified by flash chromatography (eluting solvent: hexanes) to give title compound (X = Br, 0.32 g, yield = 86%; and X = Cl, 0.40 g, yield = 76%) as a colorless oil.ⁱ ¹H NMR (400 MHz, CDCl₃) δ: 7.32-7.26 (m, 4 H), 7.21-7.17 (m, 6 H), 2.67 (t, J = 7.7 Hz, 4 H), 1.98 (m, 2 H).

Preparation of 2-(3-phenylpropoxy)tetrahydro-2H-pyran (**5b**):

5a was prepared based on above procedure B, purified by flash chromatography (eluting solvents: MTBE/hexanes = 0-20%) to give title compound (0.41 g, yield = 98%) as a colorless oil.ⁱⁱ ¹H NMR (400 MHz, CDCl₃) δ: 7.30-7.26 (m, 2 H), 7.21-7.16 (m, 3 H), 4.58 (dd, J = 4.5, 2.9 Hz, 1 H), 3.88 (m, 1 H), 3.78 (m, 1 H), 3.50 (m, 1 H), 3.41 (1 H), 2.71 (m, 2 H), 1.93 (m, 2 H), 1.85 (m, 1 H), 1.72 (m, 1 H), 1.64-1.50 (m, 4 H).

Preparation of 5-(4-(1H-pyrrol-1-yl)phenyl)pentyl acetate (**5c**):

5c was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-30%) to give title compound (0.28 g, yield = 76%) as a colorless oil. ^1H NMR (500 MHz, CDCl_3) δ : 1.45 (m, 2 H), 1.72 (m, 4 H), 2.10 (s, 3 H), 2.67 (t, $J = 6.8$ Hz, 2 H), 4.10 (t, $J = 6.6$ Hz, 2 H), 6.39 (br s, 2 H), 7.10 (br s, 2 H), 7.25 (m, 2 H), 7.34 (m, 2 H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.0, 25.6, , 28.5, 31.0, 35.2, 64.4, 110.2, 119.4, 120.6, 129.4, 138.8, 139.9, 171.2 ppm. HRMS (ESI) calculated for $\text{C}_{17}\text{H}_{21}\text{NO}_2$ ($\text{M}+\text{H}$) $^+$ 272.1657, found 272.1645.

tert-Butyl 5-(5-acetoxypentyl)-1H-indole-1-carboxylate (**5d**):

5d was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-30%) to give title compound (0.33 g, yield = 94%) as a colorless oil. ^1H NMR (500 MHz, CDCl_3) δ : 1.41 (p, $J = 7.6$ Hz, 2 H), 1.68 (m, 13 H), 2.05 (s, 3 H), 2.72 (t, $J = 7.6$ Hz, 2 H), 4.07 (t, $J = 6.7$ Hz, 2 H), 6.52 (d, $J = 3.7$ Hz, 1 H), 7.15 (d, $J = 8.5$ Hz, 1 H), 7.36 (s, 1 H), 7.58 (s, 1 H), 8.06 (s, 1 H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.0, 25.5, 28.2, 28.5, 31.5, 35.6, 64.5, 83.5, 107.1, 114.9, 120.3, 124.9, 126.0, 130.8, 133.6, 136.8, 149.8, 171.2 ppm. HRMS (ESI) calculated for $\text{C}_{20}\text{H}_{27}\text{NO}_4$ ($\text{M}+\text{Na}$) $^+$ 368.1851, found 368.1832.

1-(4,4-Diethoxybutyl)-2-methoxybenzene (**5e**):

5e was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-30%) to give title compound (0.40 g, yield = 98%) as a colorless oil. ^1H NMR (500 MHz, CDCl_3) δ : 1.22 (t, $J = 7.1$ Hz, 6 H), 1.69-1.68 (m, 4 H), 2.66 (s, 2 H), 3.53-3.47 (m, 2 H), 3.68-3.62 (m, 2 H), 3.82 (s, 3 H), 4.53 (m, 1 H), 6.90-6.83 (m, 2 H), 7.20-7.14 (m, 2 H); ^{13}C NMR (125 MHz, CDCl_3) δ : 15.4, 25.0, 29.9, 33.4, 55.2, 60.9, 103.0, 110.2, 120.4, 126.9, 129.8, 130.7, 157.5 ppm. HRMS (ESI) calculated for $\text{C}_{15}\text{H}_{24}\text{O}_3$ ($\text{M}+\text{Na}$) $^+$ 275.1626, found 275.1618.

Ethyl 5-(4-methoxyphenyl)-2-methylpentanoate (5f):

5f was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-30%) to give title compound (0.35 g, yield = 88%) as a colorless oil. ^1H NMR (400 MHz, CDCl_3) δ : 1.14 (d, J = 7.0 Hz, 3 H), 1.24 (t, J = 7.1 Hz, 3 H), 1.48-1.40 (m, 1 H), 1.73-1.52 (m, 3 H), 2.45-2.40 (m, 1 H), 2.55 (t, J = 7.6 Hz, 2 H), 3.78 (s, 3 H), 4.12 (q, J = 7.1 Hz, 2 H), 6.85-6.81 (m, 2 H), 7.08 (d, J = 8.3 Hz, 2 H); ^{13}C NMR (100 MHz, CDCl_3) δ : 14.3, 17.1, 29.3, 33.4, 34.8, 39.5, 55.2, 60.1, 113.7, 129.2, 134.3, 157.8, 176.8 ppm. HRMS (ESI) calculated for $\text{C}_{15}\text{H}_{22}\text{O}_3$ (M) $^+$ 250.1585, found 250.1587.

Dimethyl(phenyl)((1R,2R)-2-(4-(4-(trifluoromethyl)phenyl)butyl)cyclopropyl)silane (5g):

5g was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-10%) to give title compound (X = Br, 0.46 g, yield = 92%; and X = Cl, 0.54 g, yield = 87%) as a colorless oil. ^1H NMR (400 MHz, CDCl_3) δ : -0.40 (dt, J = 9.8, 6.5 Hz, 1 H), 0.24 (s, 6 H), 0.54-0.37 (m, 2 H), 0.72-0.69 (m, 1 H), 0.97-0.91 (m, 1 H), 1.33-1.24 (m, 2 H), 1.47-1.48 (m, 1 H), 1.69 (br, 2 H), 2.70 (t, J = 7.7 Hz, 2 H), 7.32 (d, J = 7.9 Hz, 2 H), 7.41 (t, J = 3.2 Hz, 3 H), 7.63-7.58 (m, 4 H); ^{13}C NMR (100 MHz, CDCl_3) δ : -3.8, -3.7, 3.5, 9.2, 15.7, 29.5, 31.1, 35.7, 35.9, 123.4 (q, J = Hz), 125.2, 127.7, 128.2, 128.9, 133.8, 139.5, 147.0 ppm. HRMS (ESI) calculated for $\text{C}_{22}\text{H}_{28}\text{F}_3\text{Si}$ ($\text{M}+\text{H}$) $^+$ 377.1912, found 377.1907.

3-(4-((1R,2R)-2-(dimethyl(phenyl)silyl)cyclopropyl)butyl)pyridine (5h):

5h was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-20%) to give title compound (X = Br, 0.45 g, yield = 77%; and X = Cl, 0.52 g, yield = 63%) as a colorless oil. ^1H NMR (500 MHz, CDCl_3) δ : -0.44 (dt, J = 9.8, 6.5 Hz, 1 H), 0.20 (s, 6 H), 0.43 (d, J = 27.1 Hz, 2 H), 0.69-0.65 (m, 1 H), 1.31-1.23 (m, 1 H), 1.47-1.40 (m, 3 H), 1.68-1.59 (m, 2 H), 2.60 (t, J = 7.7 Hz, 2 H), 7.21 (dd, J = 7.8, 4.8 Hz, 1 H), 7.37 (t, J = 3.1 Hz, 3 H), 7.47 (d, J = 7.8 Hz, 1 H), 7.58-7.56 (m, 2 H), 8.46 (s, 2 H); ^{13}C NMR (125 MHz, CDCl_3) δ : -3.8,

-3.7, 3.4, 9.2, 15.6, 29.4, 31.0, 33.1, 35.6, 123.2, 127.7, 128.8, 133.8, 135.8, 137.9, 139.4, 147.2, 150.0 ppm. HRMS (ESI) calculated for $C_{20}H_{27}NSi$ ($M+H$)⁺ 310.1998, found 310.1986.

1-(3-(5-((1R,2R)-2-((*tert*-Butyldiphenylsilyl)oxy)cyclopentyl)pentyl)phenyl)ethan-1-one (**5i**):

5i was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-10%) to give title compound (X = Br, 0.64 g, yield = 83%; and X = Cl, 0.81 g, yield = 81%) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ: 0.94-0.85 (m, 1 H), 1.07 (m, 11 H), 1.22 (m, 5 H), 1.50-1.45 (m, 1 H), 1.62-1.54 (m, 4 H), 1.76-1.70 (m, 1 H), 1.82 (t, *J* = 7.5 Hz, 1 H), 1.93-1.87 (m, 1 H), 2.64-2.61 (m, 5 H), 3.85 (q, *J* = 4.9 Hz, 1 H), 7.42-7.36 (m, 8 H), 7.69-7.67 (m, 4 H), 7.80-7.78 (m, 2 H); ¹³C NMR (125 MHz, CDCl₃) δ: 19.2, 22.0, 26.7, 27.1, 27.8, 29.4, 31.3, 33.5, 34.4, 35.8, 48.2, 80.4, 125.9, 127.4, 128.1, 128.4, 129.41, 129.43, 133.3, 134.8, 134.9, 135.90, 135.92, 137.2, 143.5, 198.4 ppm. HRMS (ESI) calculated for $C_{34}H_{44}O_2Si$ ($M+H$)⁺ 513.3184, found 513.3183.

3-(5-((1R,2R)-2-((*tert*-Butyldiphenylsilyl)oxy)cyclopentyl)pentyl)quinoline (**5j**):

5j was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-20%) to give title compound (0.56 g, yield = 75%) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ: 0.88 (t, *J* = 8.7 Hz, 1 H), 1.04 (s, 9 H), 1.20-1.30 (m, 5 H), 1.47 (m, 1 H), 1.65-1.51 (m, 5 H), 1.70-1.67 (m, 1 H), 1.84-1.80 (m, 1 H), 1.90-1.87 (m, 1 H), 2.74 (t, *J* = 7.7 Hz, 2 H), 3.82 (d, *J* = 5.4 Hz, 1 H), 7.39-7.34 (m, 6H), 7.53 (t, *J* = 7.5 Hz, 1 H), 7.66 (d, *J* = 5.8 Hz, 5 H), 7.77 (d, *J* = 8.1 Hz, 1 H), 7.88 (s, 1 H), 8.09 (d, *J* = 8.4 Hz, 1 H), 8.76 (s, 1 H); ¹³C NMR (125 MHz, CDCl₃) δ: 19.2, 22.0, 27.1, 27.8, 29.3, 29.4, 31.0, 33.2, 33.5, 34.4, 48.2, 80.4, 126.5, 127.3, 127.4, 128.2, 128.5, 129.2, 129.42, 129.43, 134.1, 134.76, 134.84, 135.4, 135.9, 146.8, 152.2 ppm. HRMS (ESI) calculated for $C_{35}H_{43}NOSi$ ($M+H$)⁺ 522.3195, found 522.3187.

2-Benzyl-6,6-dimethylbicyclo[3.1.1]heptane (**5k**):

5k was prepared based on above procedure B, purified by flash chromatography (eluting solvent: hexanes) to give title compound (0.30 g, yield = 74%) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ: 0.90 (d, *J* = 9.6 Hz, 1 H), 1.20 (s, 3 H), 1.25 (s, 3 H), 1.65-1.56 (m, 1 H), 2.01-1.87 (m, 5 H), 2.36 (d, *J* = 9.7 Hz, 2 H), 2.78-2.69 (m, 2 H), 7.23-7.19 (m, 3 H), 7.35-7.30 (m, 2 H); ¹³C NMR

(125 MHz, CDCl₃) δ : 22.4, 23.5, 26.5, 28.3, 33.8, 38.8, 41.6, 43.3, 43.5, 45.3, 125.6, 128.1, 129.1, 142.0 ppm. HRMS (ESI) calculated for C₁₆H₂₂ (M)⁺ 214.1742, found 214.1742.

3-(((1R,2S,5R)-6,6-Dimethylbicyclo[3.1.1]heptan-2-yl)methyl)quinoline (**5I**):

5I was prepared based on above procedure B, purified by flash chromatography (eluting solvents: EtOAc/hexanes = 0-20%) to give title compound (0.31 g, yield = 82%; or 0.32 g, yield = 84% by RuPhos as ligand) as a colorless oil. ¹H NMR (500 MHz, CDCl₃) δ : 0.87 (d, *J* = 9.6 Hz, 1 H), 1.19 (s, 3 H), 1.21 (s, 3 H), 1.64-1.59 (m, 1 H), 1.93-1.84 (m, 4 H), 1.99 (t, *J* = 12.2 Hz, 1 H); 2.33-2.29 (m, 1 H), 2.42 (t, *J* = 8.6 Hz, 1 H), 2.90-2.81 (m, 2 H), 7.51 (t, *J* = 7.5 Hz, 1 H), 7.64 (t, *J* = 7.5 Hz, 1 H), 7.76 (d, *J* = 8.2 Hz, 1 H), 7.87 (s, 1 H), 8.08 (d, *J* = 8.4 Hz, 1 H), 8.76 (s, 1 H); ¹³C NMR (125 MHz, CDCl₃) δ : 22.3, 23.6, 26.4, 28.2, 33.7, 38.8, 40.7, 41.4, 43.1, 45.2, 126.5, 127.3, 128.5, 129.2, 134.8, 146.8, 152.5 ppm. HRMS (ESI) calculated for C₁₉H₂₃N (M+H)⁺ 266.1915, found 266.1903.

Ethyl (*Z*)-6-phenylhex-2-enoate (**5m-cis**) and Ethyl (*E*)-6-phenylhex-2-enoate (**5m-trans**)

Both of **5m-cis** and **5m-trans** were prepared based on above procedure B through the coupling reaction of ethyl (*Z*)-3-bromoacrylate with tris(3-phenylpropyl)borane to give *cis:trans* = 9:1. The mixture was separated by flash chromatography (eluting solvents: MTBE/hexanes = 0-5%) to give title compounds **5m-cis** (0.19 g), yield = 52% as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ : 1.28 (t, *J* = 7.1 Hz, 3 H), 1.82-1.74 (m, 2 H), 2.74-2.64 (m, 4 H), 4.16 (q, *J* = 7.1 Hz, 2 H), 5.80 (dt, *J* = 11.6, 1.8 Hz, 1 H), 6.23 (dt, *J* = 11.6, 7.6 Hz, 1 H), 7.20-7.16 (m, 3 H), 7.30-7.25 (m, 2 H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.2, 28.7, 30.8, 35.6, 59.8, 120.1, 125.8, 128.3, 128.4, 142.1, 149.8, 166.4 ppm. HRMS (ESI) calculated for C₁₄H₁₉O₂ (M+H)⁺ 219.1385, found 219.1386. **5m-trans** (0.018 g), yield = 5% as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ : 1.28 (t, *J* = 7.1 Hz, 3 H), 1.80 (m, 2 H), 2.34 (m, 2 H), 2.65 (t, *J* = 7.6 Hz, 2 H), 4.19 (q, *J* = 7.1 Hz, 2 H), 5.83 (dt, *J* = 15.6, 1.6 Hz, 1 H), 6.98 (dt, *J* = 15.6, 7.0 Hz, 1 H), 7.21-7.16 (m, 3 H), 7.30-7.25 (m, 2 H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.2, 29.6, 31.6, 35.2, 60.2, 121.7, 125.9, 128.3, 128.4, 141.7, 148.7, 166.7 ppm. HRMS (ESI) calculated for C₁₄H₁₉O₂ (M+H)⁺ 219.1385, found 219.1396.

(*E*)-Hex-4-en-1-ylbenzene (**5n**)

5n was prepared based on above procedure B through the coupling reaction of (*E*)-1-bromoprop-1-ene with tris(3-phenylpropyl)borane, purified by flash chromatography (eluting solvent: hexanes) to give title compound (0.24 g, yield = 60%) as a colorless oil. ^1H NMR (400 MHz, CDCl_3) δ : 1.74-1.67 (m, 5 H), 2.07-2.02 (m, 2 H), 2.63 (t, $J = 7.7$ Hz, 2 H), 5.19 (m, 2 H), 7.22-7.18 (m, 3 H), 7.31-7.28 (m, 2 H); ^{13}C NMR (100 MHz, CDCl_3) δ : 17.9, 31.3, 32.1, 35.4, 125.2, 125.6, 128.2, 128.4, 131.0, 142.6 ppm. HRMS (ESI) calculated for $\text{C}_{12}\text{H}_{17}$ ($\text{M}+\text{H}$) $^+$ 161.1330, found 161.1340.

((1*S*,2*S*)-2-(But-3-en-1-yl)cyclopropyl)dimethyl(phenyl)silane:

^1H NMR (500 MHz, CDCl_3) δ : -0.40 (dt, $J = 9.8, 6.6$ Hz, 1 H), 0.21 (s, 3 H), 0.24 (s, 3 H), 0.46 (d, $J = 14.0$ Hz, 2 H), 0.74-0.69 (m, 1 H), 1.35-1.26 (m, 1 H), 1.56-1.49 (m, 1 H), 2.18 (q, $J = 7.3$ Hz, 2 H), 4.97 (d, $J = 10.2$ Hz, 1 H), 5.05 (t, $J = 17.1$ Hz, 1 H), 5.88 (ddt, $J = 17.3, 10.2, 6.5$ Hz, 1 H), 7.39 (t, $J = 3.2$ Hz, 3 H), 7.60-7.58 (m, 2 H); ^{13}C NMR (125 MHz, CDCl_3) δ : -3.9, -3.6, 3.5, 9.1, 15.5, 34.1, 35.4, 114.1, 114.2, 127.7, 128.8, 133.8, 139.0, 139.4 ppm. HRMS (ESI) calculated for $\text{C}_{15}\text{H}_{23}\text{Si}$ ($\text{M}+\text{H}$) $^+$ 231.1569, found 231.1565.

tert-Butyl(((1*R*,2*R*)-2-(pent-4-en-1-yl)cyclopentyl)oxy)diphenylsilane:

^1H NMR (500 MHz, CDCl_3) δ : 0.95-0.88 (m, 1 H), 1.08 (m, 11 H), 1.28 (m, 3 H), 1.61-1.46 (m, 2 H), 1.76-1.69 (m, 1 H), 1.85-1.83 (m, 1 H), 1.99-1.86 (m, 3 H), 3.86 (q, $J = 5.03$ Hz, 1 H), 4.97-4.90 (m, 2 H), 5.75 (ddt, $J = 17.1, 10.2, 6.7$ Hz, 1 H), 7.42-7.34 (m, 6 H), 7.71-7.68 (m, 4 H); ^{13}C NMR (125 MHz, CDCl_3) δ : 19.2, 22.0, 27.1, 27.3, 29.3, 33.1, 33.9, 34.4, 48.1, 80.4, 114.1, 127.5, 127.6, 129.4, 134.7, 134.9, 135.6, 135.9, 139.1 ppm. HRMS (ESI) calculated for $\text{C}_{15}\text{H}_{22}\text{Si}$ (M) $^+$ 230.1487, found 230.1490.

nmr500c h-1

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PROCNO 1
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Current Data Parameters
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EXNO 11
PROCNO 1

F2 - Acquisition Parameters
Date 20141022
Time 10.02

INSTRUM spect
PROBHD 5 mm PABBI 1H-
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 1024
DS 4
SSNH 40322.581 Hz
FIDRES 0.307637 Hz
AQ 1.6253428 sec
RG 4096
DM 12.400 usec
DE 1453.0 usec
TE 295.0 K
D1 0.10000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 13C =====
NUC1 13C
P1 5.00 usec
PL1 -6.00 dB
SFO1 125.8206598 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 82.50 usec
PL2 120.00 dB
PL12 15.00 dB
SFO2 500.3325016 MHz

F2 - Processing parameters
SI 32768
SF 500.136438 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40

Current Data Parameters
 NAME HM-301865-123
 EXNO 10
 F2CNO 1

F2 - Acquisition Parameters

Date_ 20141022
 Time 10.10
 INSTRUM spect
 PROBHD 5 mm PAB12430
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 13020.83 Hz
 FIDRES 0.196882 Hz
 AQ 2.516323 sec
 RG 20.2
 DW 38.400 usec
 DE 653.0 usec
 TE 295.0 K
 D1 0.10000000 sec
 TDO 1

===== CHANNEL f1 =====

NUC1 1H
 P1 8.60 usec
 PL1 -1.00 dB
 SF01 500.330895 MHz
 F2 - Processing parameters
 SI 32768
 SF 500.330000 MHz
 WDW EX
 SSB 0
 GB 0 Hz
 PC 1.00

Current Data Parameters

NAME HM-301865-123
EXRNO 11
PROCNO 1

F2 - Acquisition Parameters

Date_ 20141022
Time_ 10.42
INSTRUM spect
PROBHD 5 mm PABBI 1H-
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 1024
DS 4
SWH 40322.582 Hz
FIDRES 0.307637 Hz
AQ 1.6253428 sec
RG 4096
RGW 4400
DE 12.50 usec
TE 295.0 K
D1 0.10000000 sec
D11 0.03000000 sec
ID0 1

CHANNEL F1

NUC1 13C
P1 5.00 usec
PL1 6.00 dB
SFO1 125.8206598 MHz

CHANNEL F2

NAME_ waltz16
CPDPRG2
NUC2 1H
P2 1.00 usec
PL2 120.00 dB
PL12 15.00 dB
SFO2 500.3325016 MHz

F2 - Processing parameters

SI 32768
SF 125.8080790 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Current Data Parameters
NAME hm-0301865-108
EXNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140930
Time 15.54
INSTRUM spect
PROBHD 5 mm PAXO 3H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SSBH 6578.94 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 32
DM 76.000 usec
DE 6.50 usec
TE 29.7 Ksec
D1 0.10000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 12.75 usec
PL1 -2.00 dB
PL1W 13.05791473 W
SF01 400.334722 MHz
F2 - Processing parameters
SI 16384
SF 400.330000 MHz
WDW EX
SSB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME hm-0301865-108
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140930
Time 16.09
PULPROG 5 TMR PIRN2
SOLVENT CDCl3
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DM 19.4100 usec
DE 6.50 usec
TE 293.0 K
D1 0.10000000 sec
D11 0.03000000 sec
D10 1

CHANNEL f1
NUC1 13C
P1 3.80 usec
PL1 6.00 dB
SFO1 127.000000 MHz
SFO2 100.6741319 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 120.00 dB
P1L2 14.12 dB
P1L2W 0.00000000 W
P1L2W 0.3196110 W
SFO2 400.326017 MHz
F2 - Processing parameters
SI 32768
SF 100.6630590 MHz
RG 65536
SSB 1.00 Hz
LB 0
GB 1.40
PC 1.40

Current Data Parameters
NAME hm-0301865-109
EXNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140930
Time 15.09
INSTRUM spect
PROBHD 5 mm PATXO 3H/1
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SSBH 6578.944 Hz
AQ 0.200774 Hz
RG 2.4904180 sec
FIDRES 32
DM 76.000 usec
DE 6.500 usec
TE 292.7 K
D1 0.10000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 12.75 usec
PL1 -2.00 dB
PL1W 13.05791473 W
SF01 400.334722 MHz
F2 - Processing parameters
SI 16384
SF 400.330000 MHz
WDW EX
SSB 0.30 Hz
LB 0
GB 0
PC 1.00

Current Data Parameters
NAME hm-0301865-109
EXNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140930
Time 15.24

INSTRUM spect
PULPROG zgpg30
TD 65536

SOLVENT CDCl₃
NS 512

SMH 26315.789 Hz
FIDRES 0.401547 Hz

AQ 1.2452340 sec
RG 8192

DM 19.4100 usec
DE 6.450 usec

TE 293.0 K
D1 0.10000000 sec

D11 0.03000000 sec
ID0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 3.80 usec

PL1 6.00 dB
PL12 127.000000 Hz

PL14 100.6741319 MHz
SFO1

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H

P2 80.00 usec
PL2 120.00 dB

PL12 14.12 dB
PL12W 0.00000000 W

PL12W 0.3196110 W
SFO2 400.326017 MHz

F2 - Processing parameters
SI 32768

SF 100.6630590 MHz
WDW EM

SSB 0
LB 1.00 Hz

GB 0
PC 1.40

146.96
139.46
133.79
128.88
128.73
127.70
125.25
125.22
125.18
125.14

35.91
35.72
31.10
29.52
15.66
9.15
3.42
3.70
3.80

nmr500c h-1

Current Data Parameters
 NAME 301865-069
 EXNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20100709
 Time 11.12
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 32
 DS 4
 SFO 13020.84 Hz
 FIDRES 0.198682 Hz
 AQ 2.516707 sec
 RG 64
 DW 38.400 usec
 DE 4.50 usec
 TE 300.0 K
 D1 0.10000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 12.00 usec
 PL1 -4.00 dB
 SFO1 500.130885 MHz
 F2 - Processing parameters
 SI 32768
 SF 500.130000 MHz
 WDW EM
 SSB 0
 GB 0.30 Hz
 PC 1.00

nmr500c c-13

Current Data Parameters
NAME 301865-069
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100709
Time 11.15

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 131072
SOLVENT CDCl3

NS 11
DS 4
SWH 40322.582 Hz
FIDRES 0.307637 Hz
AQ 1.6253552 sec
RG 1625.5
RM 12.410
DM 12.450 usec
TE 300.1 K
D1 0.10000000 sec
d11 0.03000000 sec
ID0 40

===== CHANNEL f1 =====
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SFO1 125.7703648 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
ECG2 80.10 usec
PL2 11.50 dB
P2 120.00 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

150.00
147.23
139.40
137.87
135.75
133.75
128.84
127.67
123.22

77.35
77.09
76.84

35.65
33.10
30.98
29.42

15.64

9.15

3.43

-3.70
-3.80

Current Data Parameters
NAME 301865-093
EXNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100804
Time 9.18
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 13020.83 Hz
FIDRES 0.196882 Hz
AQ 2.516707 sec
RG 64
DW 38.400 usec
DE 4.50 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 500.130885 MHz

F2 - Processing parameters
SI 32768
SF 500.130000 MHz
WDW EM
SSB 0
GB 0
PC 1.00

nmr500c c-13

Current Data Parameters
NAME 301865-093
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100804
Time 9.23

INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30

TD 131072
SOLVENT CDCl3

NS 180
DS 4

SWH 40322.582 Hz
FIDRES 0.307637 Hz

AQ 1.6253552 sec
RG 1628.5

RM 12.400 usec
DE 6.450 usec

TE 300.0 K
D1 0.10000000 sec

d11 0.03000000 sec
ID0 40

CHANNEL f1
NUC1 13C
P1 2.50 usec

PL1 0.00 dB
SFO1 125.770348 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
P2 10.00 usec

PL2 11.50 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz

WDW EM
SSB 0

GB 1.00 Hz
PC 1.40

nmr500c h-1

Current Data Parameters
NAME 301865-067
EXNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100629
Time 8:59
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 13020.833 Hz
FIDRES 0.1019884 Hz
AQ 2.516707 sec
RG 362
DW 38.400 usec
DE 3.00 usec
DI 0.1000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 500.130085 MHz
F2 - Processing parameters
SI 32768
SF 500.130085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

5j

nmr500c h-1

Current Data Parameters
NAME 301865-079
EXNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100716
Time 16.42
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SSNH 13020.843 Hz
FIDRES 0.198682 Hz
AQ 2.516707 sec
RG 64
DW 38.400 usec
DE 6.50 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 -4.00 dB
SFO1 500.130885 MHz
F2 - Processing parameters
SI 32768
SF 500.130000 MHz
WDW EM
SSB 0
GB 0.30 Hz
PC 1.00

nmr500c c-13

Current Data Parameters
NAME 301865-079
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100716
Time 16.46

INSTRUM spect
PROBHD 5 mm QNP 125
PULPROG zgpg30
TD 131072
SOLVENT CDCl3
NS 105
DS 4
SWH 40322.582 Hz
FIDRES 0.307637 Hz
AQ 1.6253552 sec
RG 1149.4
RM 12.450 usec
DM 6.450 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
ID0 40

===== CHANNEL f1 =====
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SFO1 125.7703648 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
ECG2 80.10 usec
PL2 11.50 dB
P12 120.00 dB
SFO2 500.1325007 MHz

F2 - Processing parameters
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Current Data Parameters
 NAME 301865-118
 EXNO 10
 FRCNO 1
 F2 - Acquisition Parameters
 Date_ 20141008
 Time 13.20
 INSTRUM spect
 PROBHD 5 mm PAXX-230
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 4
 SWH 6578.947 Hz
 FIDRES 0.200774 Hz
 AQ 2.4904180 sec
 RG 45.3
 DW 76.000 usec
 DE 6.500 usec
 TE 292.7 K
 D1 0.10000000 sec
 TD0 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 12.75 usec
 PL1 -2.00 dB
 PL1W 13.05791473 W
 SF01 400.3324722 MHz
 F2 - Processing parameters
 SI 16384
 SF 400.3300000 MHz
 MDW 4K
 SFS 0.30 Hz
 LBS 0
 GB 0
 PC 1.00

Current Data Parameters

NAME 301865-118

EXNO 1

PROCNO 1

F2 - Acquisition Parameters

Date_ 20141008

Time 13.35

INSTRUM spect

PROBHD 5 mm PAKO 3H/

PULPROG zgpg

TD 65536

SOLVENT CDCl3

NS 312

DS 4

SWH 26315.789 Hz

FIDRES 0.401547 Hz

AQ 1.2452340 sec

RG 8192

RW 39400 usec

DE 6.450 usec

TE 293.0 K

D1 0.10000000 sec

D11 0.03000000 sec

ID0 1

CHANNEL f1

NUC1 13C

P1 3.80 usec

PL1 6.00 dB

PR1 127.000000 MHz

SFO1 100.6741319 MHz

CHANNEL f2

waltz16

PCPD2 80.00 usec

PL2 120.00 dB

PL12 14.12 dB

PL2W 0.00000000 W

PL2W 0.31306110 W

SFO2 400.3350117 MHz

F2 - Processing parameters

SI 32768

SF 100.6630590 MHz

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

152.51
146.72
134.84
134.48
129.13
128.51
128.14
127.32
126.50

45.15
43.10
41.39
40.63
38.80
33.70
28.17
26.35
23.59
22.26

10.40	usec
0.00	dB
24710	MHz
32768	
00097	MHz
EM	
0	
0.30	Hz
0	
1.00

nmr500c h-1

NAME 0322653-suzuki-cysm
EXPNO 1
PROCNO 1
Date_ 20111107
Time 13.32
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 13020.833 Hz
FIDRES 0.198682 Hz
AQ 0.198682 Hz
RG 32768
AQ 0.198682 Hz
DE 38.400 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL 14.00 dB
SFO1 500.130000 MHz
SF 500.130000 MHz
WDW EX
SSB 0
LB 0.30 Hz
GB 1.40
PC 1.40

nmr500c h-1

NAME 0322653-suzuki-cycpe
EXPNO 1
PROCNO 1
Date_ 2011107
Time 13.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 13020.833 Hz
FIDRES 0.198682 Hz
AQ 2.516707 sec
RG 64
DE 38.400 usec
TE 300.0 K
D1 0.10000000 sec
ID0 1
===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -4.00 dB
SFO1 500.130853 MHz
SF 500.130853 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

nmr500c c-13

NAME 0322653-suzuki-cycp
EXENO 2
PROCNO 1
Date_ 2011107
Time_ 13:44
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
ID 131072
SOLVENT CDCl3
NS 1
DS 4
SWH 40322.582 Hz
FIDRES 0.307637 Hz
AQ 1.625352 sec
RG 383
DE 12.400 usec
TE 300.2 K
D1 0.1000000 sec
D11 0.0300000 sec
TD 40

139.09
135.94
135.91
135.60
134.90
134.74
129.44
129.41
127.61
127.46
127.44

114.14

80.38

48.10

34.42
33.94
33.10
29.29
27.34
27.06
21.96
19.22

===== CHANNEL f1 =====
NUC1 13C
P1 2.50 usec
PL1 0.00 dB
SFO1 125.7703648 MHz

===== CHANNEL f2 =====
CEPPRG2 waltz16
NUC2 13C
PCPD2 80.00 usec
PL2 120.00 dB
PL12 11.50 dB
SFO2 500.1325007 MHz
SI 65536
WDW EX
SSB 0
LB 1.00 Hz
GB 0
FC 1.40

ⁱ Matched the commercial available compound.

ⁱⁱ Known compound, see: Oriyama, T.; Yatabe, K.; Sugawara, S.; Machiguchi, Y.; Koga, G. *Synlett*. **1996**, 523-525.