

Supplementary Information for

Anthranilamide (aam)-substituted arylboranes in direct carbon–carbon bond-forming reactions

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Table of Contents

General Remarks	S2
Synthesis of 2-Thienyl–B(aam)	S2
Synthesis of 2-Furyl–B(aam)	S2
Direct Suzuki–Miyaura Cross-Coupling of Ar–B(aam)	S3
Rhodium-Catalyzed 1,4-Addition of Ar–B(aam) to Cyclohexenone	S3
Characterization of Products	S4
References	S14
Copies of NMR	S15

General Remarks.

All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under an argon atmosphere. Nuclear magnetic resonance spectra were taken on a Varian System 500 (^1H , 500 MHz; ^{13}C , 125 MHz; ^{11}B , 186 MHz) spectrometer using residual chloroform (^1H , $\delta = 7.26$), CDCl_3 (^{13}C , $\delta = 77.16$), a residual proton in $\text{DMSO-}d_6$ (^1H , $\delta = 2.50$) and $\text{DMSO-}d_6$ (^{13}C , $\delta = 39.52$) as an internal standard, and boron trifluoride diethyl etherate (^{11}B , $\delta = 0.00$) as an external standard. ^1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration. High-resolution mass spectra were obtained with a Thermo Fisher Scientific LTQ Orbitrap XL spectrometer. Melting points were measured with Yanaco Micro Melting Point apparatus and uncorrected. Column chromatography was carried out using Merck Kieselgel 60. All microwave reactions (Biotage, Initiator+) were conducted in a sealed tube, and the reaction temperature was maintained by an external infrared sensor. Unless otherwise noted, commercially available reagents were used without purification. 1,4-Dioxane was distilled from CaH_2 , and other solvents were dried over activated molecular sieves 3Å.

Synthesis of 2-Thienyl-B(aam)

Pd-Catalyzed Borylation of 2-Thienyl Bromide: A Schlenk tube equipped with a magnetic stirring bar was charged with potassium acetate (0.450 mmol), (pin)B-B(aam) (0.150 mmol), tris(dibenzylideneacetone)dipalladium (3.75 μmol), 2-dicyclohexylphosphino-2',4',6'-triisopropylbiphenyl (0.0113 mmol), 1,4-dioxane (0.5 mL) and 2-thienyl bromide (0.225 mmol). After the mixture was stirred at 60 °C for 18 h, the mixture was diluted with ethyl acetate and the organic solution was washed with brine, dried over MgSO_4 , and evaporated. The product was isolated by Florisil-column chromatography (hexane:ethyl acetate = 1:1 as an eluent).

Condensation of 2-Thienyl Boronic Acid with Anthranilamide: A reaction vessel equipped with a magnetic stirring bar was charged with 2-thienyl boronic Acid (4.00 mmol), anthranilamide (4.00 mmol) and toluene (16 mL). After the mixture was refluxed for 1 h, the product was isolated by filtration.

Synthesis of 2-Furyl-B(aam)

A reaction vessel equipped with a magnetic stirring bar was charged with 2-furyl boronic Acid (8.67 mmol), anthranilamide (8.67 mmol) and toluene (35 mL). After the mixture was refluxed for 1 h, the product was isolated by filtration.

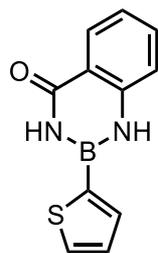
Direct Suzuki–Miyaura Cross-Coupling of Ar–B(aam): A General Procedure.

A reaction tube equipped with a magnetic stirring bar was charged with tripotassium phosphate (1.13 mmol), Ar–B(aam) (0.225 mmol), palladium(II) acetate (7.50 μ mol), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (0.0150 mmol), 1,4-dioxane (1.9 mL), H₂O (375 μ l) and an aryl bromide (0.150 mmol). After the mixture was stirred at 140 °C for 0.5 h under microwave irradiation, the mixture was diluted with ethyl acetate. The organic solution was washed with brine, dried over MgSO₄, and evaporated. The product was isolated by silica gel-column chromatography (hexane/ethyl acetate = 20:1 as an eluent).

Rhodium-Catalyzed 1,4-Addition of Ar–B(aam) to Cyclohexenone: A General Procedure.

A reaction tube equipped with a magnetic stirring bar was charged with tripotassium phosphate (0.225 mmol), Ar–B(aam) (0.150 mmol), cyclooctadiene rhodium(I) chloride dimer (4.50 μ mol), 1,4-dioxane (0.50 mL), H₂O (0.13 mL) and cyclohexenone (0.300 mmol). After the mixture was stirred at 140 °C for 0.5 h under microwave irradiation, the mixture was diluted with ethyl acetate. The organic solution was washed with brine, dried over MgSO₄, and evaporated. The product was isolated by silica gel-column chromatography (hexane/diethyl ether = 5:1 as an eluent).

2-(thiophen-2-yl)-2,3-dihydrobenzo[d][1,3,2]diazaborinin-4(1H)-one



Isolated in 95% yield as a white solid: mp 210.6–213.9 °C

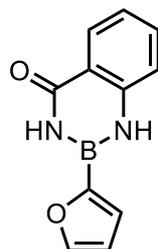
¹H NMR (400 MHz, DMSO-d₆) δ 7.09 (ddd, *J* = 8.1, 7.1, 1.2 Hz, 1H), 7.29 (dd, *J* = 4.7, 3.4 Hz, 1H), 7.42 (ddd, *J* = 8.2, 1.2, 0.5 Hz, 1H), 7.55 (ddd, *J* = 8.2, 7.1, 1.6 Hz, 1H), 7.93 (dd, *J* = 4.7, 0.9 Hz, 1H), 7.97 – 8.04 (m, 2H), 9.28 (brs, 1H), 9.74 (brs, 1H).

¹³C NMR (126 MHz, DMSO-d₆) δ 118.13, 118.78, 120.82, 127.93, 128.63, 132.49, 133.41, 136.36, 145.40, 166.01.

¹¹B NMR (160 MHz, DMSO) δ 28.60.

HRMS Calcd for C₁₁H₉BN₂OSNa: [M+Na]⁺, 251.0421 Found: *m/z* 251.0423

2-(furan-2-yl)-2,3-dihydrobenzo[d][1,3,2]diazaborinin-4(1H)-one



Isolated in 95% yield as a white solid: mp 197.3–199.2 °C

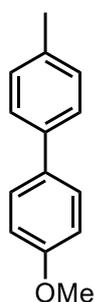
¹H NMR (400 MHz, DMSO-d₆) δ 6.61 (dd, *J* = 3.4, 1.6 Hz, 1H), 7.08 (ddd, *J* = 8.1, 7.1, 1.2 Hz, 1H), 7.43 (ddd, *J* = 8.2, 1.2, 0.5 Hz, 1H), 7.48 (dd, *J* = 3.4, 0.7 Hz, 1H), 7.54 (ddd, *J* = 8.2, 7.1, 1.6 Hz, 1H), 7.94 – 8.02 (m, 2H), 9.35 (s, 1H), 9.67 (s, 1H).

¹³C NMR (101 MHz, DMSO-d₆) δ 111.04, 118.22, 118.87, 120.83, 122.13, 127.95, 133.40, 145.38, 147.43, 165.92.

¹¹B NMR (160 MHz, DMSO) δ 26.43

HRMS Calcd for C₁₁H₉BN₂O₂Na: [M+Na]⁺, 235.0649 Found: *m/z* 235.0652

4-methoxy-4'-methyl-1,1'-biphenyl (2a)¹

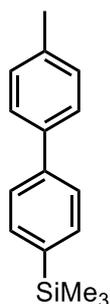


Isolated in 88% yield as a white solid: mp 114.3–118.4 °C

¹H NMR (400 MHz, Chloroform-d) δ 2.38 (s, 3H), 3.85 (s, 3H), 6.94 – 6.99 (m, 2H), 7.23 (dt, *J* = 7.9, 0.7 Hz, 2H), 7.42 – 7.48 (m, 2H), 7.48 – 7.54 (m, 2H).

¹³C NMR (101 MHz, Chloroform-d) δ 21.21, 55.50, 114.29, 126.73, 128.10, 129.57, 133.89, 136.50, 138.11, 159.06.

trimethyl(4'-methyl-[1,1'-biphenyl]-4-yl)silane (2b)²

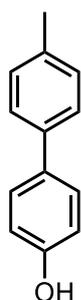


Isolated in 99% yield as a white solid: mp 63–66 °C

¹H NMR (500 MHz, Chloroform-d) δ 0.31 (s, 9H), 2.40 (s, 3H), 7.21 – 7.29 (m, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 7.55 – 7.64 (m, 4H).

¹³C NMR (126 MHz, Chloroform-d) δ -0.92, 21.27, 126.46, 127.14, 129.63, 133.93, 137.26, 138.43, 138.99, 141.68.

4'-methyl-[1,1'-biphenyl]-4-ol (2c)³



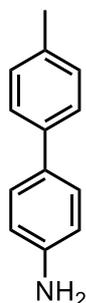
Isolated in 88% yield as a white solid: mp 150.6–154. 8°C

¹H NMR (500 MHz, Chloroform-d) δ 2.38 (s, 3H), 4.88 (brs, 1H), 6.86 – 6.93 (m, 2H), 7.19 –

7.25 (m, 2H), 7.40 – 7.50 (m, 4H).

^{13}C NMR (126 MHz, Chloroform-d) δ 21.20, 115.71, 115.74, 126.70, 128.33, 129.58, 134.12, 136.55, 138.04, 154.99.

4'-methyl-[1,1'-biphenyl]-4-amine (2d)⁴

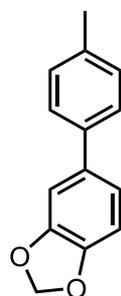


Isolated in 69% yield as a white solid: mp 97.5–100.3 °C

^1H NMR (500 MHz, Chloroform-d) δ 2.37 (s, 3H), 3.70 (brs, 2H), 6.71 – 6.78 (m, 2H), 7.18 – 7.23 (m, 2H), 7.36 – 7.43 (m, 2H), 7.43 (dd, J = 8.2, 2.0 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-d) δ 21.18, 115.53, 126.40, 127.96, 129.50, 131.75, 136.04, 138.44, 145.69.

5-(p-tolyl)benzo[d][1,3]dioxole (2e)⁵

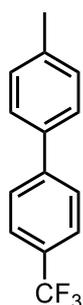


Isolated in 70% yield as a white solid: mp 62.3–66.0 °C

^1H NMR (500 MHz, Chloroform-d) δ 2.38 (s, 3H), 5.99 (s, 2H), 6.87 (dd, J = 8.0, 0.8 Hz, 1H), 7.01 – 7.08 (m, 2H), 7.18 – 7.24 (m, 2H), 7.41 (d, J = 7.5 Hz, 1H).

^{13}C NMR (126 MHz, Chloroform-d) δ 21.20, 101.22, 107.69, 108.68, 120.51, 126.87, 129.59, 135.74, 136.82, 138.23, 146.96, 148.19.

4-methyl-4'-(trifluoromethyl)-1,1'-biphenyl (2f)⁶

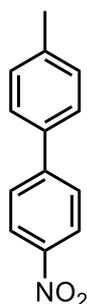


Isolated in 71% yield as a white solid: mp 121.0–125.9 °C

¹H NMR (500 MHz, Chloroform-d) δ 2.42 (s, 3H), 7.27 – 7.31 (m, 2H), 7.48 – 7.53 (m, 2H), 7.68 (s, 4H).

¹³C NMR (126 MHz, Chloroform-d) δ 21.30, 123.41 (t, *J* = 271.8 Hz), 125.81 (q, *J* = 3.7 Hz), 127.25, 127.31, 129.05 (q, *J* = 32.4 Hz), 129.85, 137.01, 138.29, 144.79.

4-methyl-4'-nitro-1,1'-biphenyl (2g)⁷

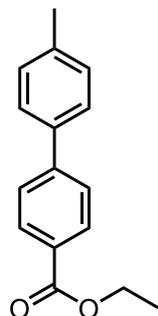


Isolated in 64% yield as a white solid: mp 137.5–141.3 °C

¹H NMR (500 MHz, Chloroform-d) δ 2.42 (s, 3H), 7.31 (dt, *J* = 7.8, 0.7 Hz, 1H), 7.51 – 7.55 (m, 2H), 7.70 – 7.74 (m, 2H), 8.26 – 8.30 (m, 2H).

¹³C NMR (126 MHz, Chloroform-d) δ 21.37, 124.25, 127.37, 127.63, 130.03, 136.01, 139.23, 147.00, 147.73.

ethyl 4'-methyl-[1,1'-biphenyl]-4-carboxylate (2h)⁸

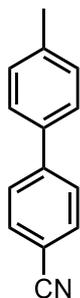


Isolated quantitatively as a white solid: mp 76.8–80.6 °C

¹H NMR (500 MHz, Chloroform-d) δ 1.41 (t, *J* = 7.1 Hz, 3H), 2.41 (s, 3H), 4.40 (q, *J* = 7.1

Hz, 2H), 7.25 – 7.31 (m, 2H), 7.51 – 7.55 (m, 2H), 7.62 – 7.66 (m, 2H), 8.07 – 8.12 (m, 2H).
¹³C NMR (126 MHz, Chloroform-d) δ 14.53, 21.31, 61.08, 126.89, 127.25, 129.09, 129.79, 130.18, 137.30, 138.22, 145.61, 166.72.

4'-methyl-[1,1'-biphenyl]-4-carbonitrile (2i)⁹

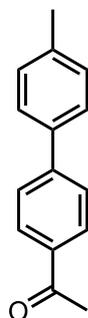


Isolated in 95% yield as a white solid: mp 102.4–105.4 °C

¹H NMR (500 MHz, Chloroform-d) δ 2.42 (s, 3H), 7.27 – 7.32 (m, 2H), 7.47 – 7.51 (m, 2H), 7.64 – 7.69 (m, 2H), 7.69 – 7.73 (m, 2H).

¹³C NMR (126 MHz, Chloroform-d) δ 21.33, 110.67, 119.18, 127.19, 127.60, 129.97, 132.70, 136.41, 138.89, 145.75.

1-(4'-methyl-[1,1'-biphenyl]-4-yl)ethan-1-one (2j)¹⁰

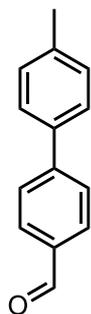


Isolated in 85% yield as a white solid: mp 118.3–122.0 °C

¹H NMR (500 MHz, Chloroform-d) δ 2.41 (s, 3H), 2.63 (s, 3H), 7.28 (dd, *J* = 7.8, 0.7 Hz, 2H), 7.53 (dd, *J* = 8.2, 2.0 Hz, 2H), 7.64 – 7.70 (m, 2H), 7.99 – 8.05 (m, 2H).

¹³C NMR (126 MHz, Chloroform-d) δ 21.32, 26.80, 127.10, 127.25, 129.05, 129.83, 135.74, 137.10, 138.39, 145.88, 197.93.

4'-methyl-[1,1'-biphenyl]-4-carbaldehyde (2k)¹¹

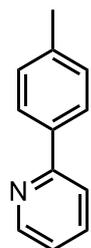


Isolated in 73% yield as a white solid: mp 104.5–108.6 °C

¹H NMR (500 MHz, Chloroform-d) δ 2.42 (s, 3H), 7.29 (dt, *J* = 7.9, 0.7 Hz, 2H), 7.52 – 7.58 (m, 2H), 7.72 – 7.78 (m, 2H), 7.91 – 7.97 (m, 2H), 10.05 (s, 1H).

¹³C NMR (126 MHz, Chloroform-d) δ 21.32, 127.10, 127.25, 129.05, 129.83, 135.74, 137.10, 138.39, 145.88, 197.93.

2-(p-tolyl)pyridine (2l)¹²

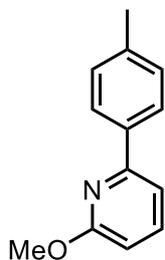


Isolated in 40% yield as a white solid: mp 128.7–132.5 °C

¹H NMR (400 MHz, Chloroform-d) δ 2.41 (s, 3H), 7.20 (ddd, *J* = 6.7, 4.8, 2.0 Hz, 1H), 7.26 – 7.32 (m, 1H), 7.66 – 7.78 (m, 2H), 7.89 (d, *J* = 8.2 Hz, 2H), 8.68 (dt, *J* = 4.9, 1.5 Hz, 1H).

¹³C NMR (101 MHz, Chloroform-d) δ 21.42, 120.39, 121.93, 126.90, 129.62, 136.77, 136.80, 139.08, 149.75, 157.62.

2-methoxy-6-(p-tolyl)pyridine (2m)¹³

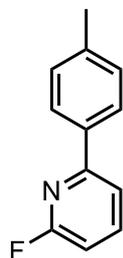


Isolated in 65% yield as a white solid: mp 74.2–78.4 °C

¹H NMR (500 MHz, Chloroform-d) δ 2.41 (s, 3H), 4.04 (s, 3H), 6.66 (d, *J* = 8.1 Hz, 1H), 7.26 (d, *J* = 7.9 Hz, 1H), 7.32 (d, *J* = 7.4 Hz, 1H), 7.61 (t, *J* = 7.8 Hz, 1H), 7.95 (d, *J* = 8.2 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 21.43, 53.32, 108.94, 112.56, 126.71, 129.45, 136.46, 138.94, 139.24, 154.86, 163.80.

2-fluoro-6-(*p*-tolyl)pyridine (2n)¹⁴

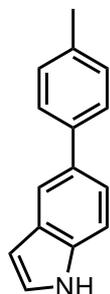


Isolated in 68% yield as a white solid: mp 38–40 °C

^1H NMR (400 MHz, Chloroform- d) δ 2.41 (s, 3H), 6.83 (ddd, $J = 8.1, 3.1, 0.7$ Hz, 1H), 7.28 (dd, $J = 8.7, 0.8$ Hz, 2H), 7.60 (ddd, $J = 7.6, 2.7, 0.7$ Hz, 1H), 7.82 (q, $J = 8.0$ Hz, 1H), 7.90 (d, $J = 8.2$ Hz, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 21.46, 107.38 (d, $J = 38.1$ Hz), 117.06 (d, $J = 4.6$ Hz), 126.94, 129.67, 134.91, 139.88, 141.67 (d, $J = 7.8$ Hz), 156.49 (d, $J = 13.4$ Hz), 163.52 (d, $J = 237.9$ Hz).

5-(*p*-tolyl)-1H-indole (2o)¹⁵



Isolated in 45% yield as a white solid: mp 66.9–70.3 °C

^1H NMR (500 MHz, Chloroform- d) δ 2.40 (s, 3H), 6.61 (dd, $J = 3.2, 2.0$ Hz, 1H), 7.23 – 7.25 (m, 2H), 7.45 (d, $J = 1.3$ Hz, 2H), 7.55 (d, $J = 8.1$ Hz, 2H), 7.82 – 7.87 (m, 1H), 8.16 (brs, 1H).

^{13}C NMR (126 MHz, Chloroform- d) δ 21.21, 103.15, 111.28, 119.15, 122.00, 124.88, 127.37, 128.52, 129.50, 133.54, 135.31, 136.08, 139.82.

2-(p-tolyl)thiophene (2p)¹⁶

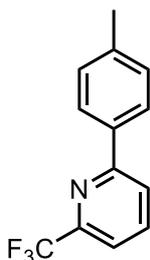


Isolated in 60% yield as a white solid: mp 60–64 °C

¹H NMR (500 MHz, Chloroform-d) δ 2.37 (s, 3H), 7.07 (dd, J = 5.1, 3.6 Hz, 1H), 7.19 (dd, J = 8.5, 0.8 Hz, 2H), 7.24 (dd, J = 5.1, 1.2 Hz, 1H), 7.27 (dd, J = 3.6, 1.2 Hz, 1H), 7.48 – 7.53 (m, 2H).

¹³C NMR (126 MHz, Chloroform-d) δ 21.32, 122.71, 124.40, 126.01, 128.06, 129.68, 131.78, 137.47, 144.72.

2-(p-tolyl)-6-(trifluoromethyl)pyridine (2q)¹⁷

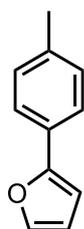


Isolated in 81% yield as a white solid: mp 37–41 °C

¹H NMR (400 MHz, Chloroform-d) δ 2.42 (s, 3H), 7.30 (d, J = 8.0 Hz, 2H), 7.57 (t, J = 4.3 Hz, 1H), 7.88 (d, J = 4.9 Hz, 1H), 7.96 (d, J = 8.2 Hz, 1H).

¹³C NMR (101 MHz, Chloroform-d) δ 21.48, 118.30 (q, J = 2.8 Hz), 121.75 (q, J = 274.1 Hz), 122.57, 127.13, 129.76, 135.17, 138.06, 140.10, 148.23 (q, J = 34.5 Hz), 157.96.

2-(p-tolyl)furan (2r)¹⁸

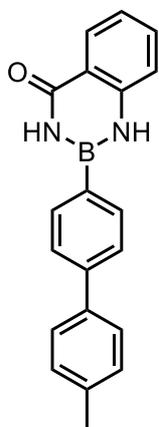


Isolated in 72% yield as a white solid: mp 38–42 °C

¹H NMR (400 MHz, Chloroform-d) δ 2.36 (s, 3H), 6.45 (dd, J = 3.3, 1.5 Hz, 1H), 6.59 (dd, J = 3.3, 0.6 Hz, 1H), 7.19 (dd, J = 7.9, 1.2 Hz, 2H), 7.44 (dd, J = 1.8, 0.7 Hz, 1H), 7.55 – 7.58 (m, 2H).

^{13}C NMR (126 MHz, Chloroform- d) δ 21.32, 104.12, 111.05, 123.97, 128.32, 129.53, 137.10, 141.96, 154.02.

2-(4'-methyl-[1,1'-biphenyl]-4-yl)-2,3-dihydrobenzo[d][1,3,2]diazaborinin-4(1H)-one (3)



Isolated in 84% yield as a white solid: mp 275.7–278.9 °C

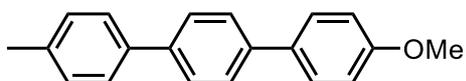
^1H NMR (400 MHz, DMSO- d_6) δ 2.36 (s, 3H), 7.11 (ddd, J = 8.1, 7.1, 1.2 Hz, 1H), 7.30 (d, J = 7.3 Hz, 2H), 7.44 (dd, J = 8.3, 1.1 Hz, 1H), 7.58 (ddd, J = 8.6, 7.1, 1.6 Hz, 1H), 7.65 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 8.3 Hz, 2H), 8.02 (dd, J = 8.0, 1.6 Hz, 1H), 8.13 (d, J = 8.3 Hz, 2H), 9.37 (brs, 1H), 9.75 (brs, 1H).

^{13}C NMR (101 MHz, DMSO- d_6) δ 20.76, 118.18, 118.80, 120.86, 125.76, 126.63, 127.98, 129.62, 133.45, 134.02, 136.91, 137.20, 141.97, 145.53, 166.36.

^{11}B NMR (160 MHz, DMSO) δ 30.83.

HRMS Calcd for $\text{C}_{20}\text{H}_{18}\text{BN}_2\text{O}$: $[\text{M}+\text{H}]^+$, 313.1507 Found: m/z 313.1509

4-methoxy-4''-methyl-1,1':4',1''-terphenyl (4)¹⁹

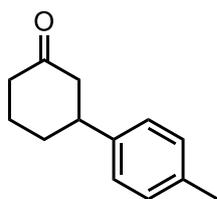


Isolated in 86% yield as a white solid: mp 238.7–242.4 °C

^1H NMR (400 MHz, Chloroform- d) δ 2.39 (s, 3H), 3.85 (s, 3H), 6.96 – 7.00 (m, 2H), 7.51 – 7.54 (m, 2H), 7.54 – 7.58 (m, 2H), 7.61 (d, J = 2.6 Hz, 4H).

^{13}C NMR (101 MHz, Chloroform- d) δ 21.28, 55.51, 114.37, 126.96, 127.01, 127.15, 127.40, 128.17, 129.66, 133.42, 137.17, 138.01, 139.57, 159.29.

3-(*p*-tolyl)cyclohexan-1-one (5a)²⁰

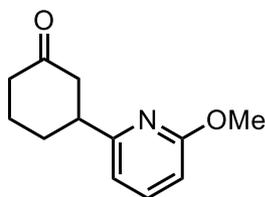


Isolated in 81% yield as colorless oil

¹H NMR (400 MHz, Chloroform-d) δ 1.65 – 1.92 (m, 2H), 2.01 – 2.21 (m, 2H), 2.33 (s, 3H), 2.34 – 2.65 (m, 1H), 2.88 – 3.08 (m, 1H), 7.05 – 7.31 (m, 4H).

¹³C NMR (126 MHz, Chloroform-d) δ 21.07, 25.64, 32.97, 41.27, 44.47, 49.15, 126.50, 129.41, 136.32, 141.47, 211.41.

3-(6-methoxypyridin-2-yl)cyclohexan-1-one (5b)²¹



Isolated in 39% yield as colorless oil

¹H NMR (500 MHz, Chloroform-d) δ 1.78 – 2.03 (m, 4H), 2.02 – 2.17 (m, 2H), 2.36 – 2.48 (m, 2H), 2.50 – 2.63 (m, 1H), 2.78 (dd, J = 14.4, 10.3 Hz, 1H), 3.08 – 3.23 (m, 1H), 3.89 (s, 3H), 6.57 (dd, J = 8.2, 0.7 Hz, 1H), 6.69 (dd, J = 7.2, 0.7 Hz, 1H), 7.48 (dd, J = 8.3, 7.2 Hz, 1H).

¹³C NMR (126 MHz, Chloroform-d) δ 24.70, 31.45, 41.31, 45.63, 46.58, 53.45, 108.48, 113.97, 139.15, 160.76, 163.74, 211.71.

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