Enantioselective Synthesis of Tetrahydroquinolines, Tetrahydroquinoxalines, and Tetrahydroisoquinolines via Pd-Catalyzed Alkene Carboamination Reactions

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Supporting Information

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General: Reactions were carried out under nitrogen in flame-dried glassware. Tris(dibenzylideneacetone)dipalladium and (S)-Siphos-PE were purchased from Strem Chemical Co. and used without further purification. 2-Allylbenzonitrile was prepared
according to a slight modification of a literature procedure (BuMgCl was used in place of BuMgBr).\(^1\) \((Z)\)-1-bromobut-1-ene\(^2\) and 3-(2-Bromophenyl)-\(N\)-methoxy-\(N\)-methylpropanamide\(^3\) were synthesized according to published procedures. All other reagents including all aryl and alkenyl bromides were purchased from commercial sources and used as received unless otherwise noted. Xylenes were purified by distillation over CaH\(_2\) prior to use in reactions. Methylene chloride and toluene were purified using a GlassContour solvent system. All yields refer to isolated compounds that are estimated to be \(\geq 95\%\) pure as judged by \(^1\)H NMR or GC analysis. The yields reported in the supporting information describe the result of a single experiment, whereas yields reported in Tables 1–4 and equations 1–3 are average yields of two or more experiments. Thus, the yields reported in the supporting information may differ from those shown in Tables 1–4 and equations 1–3.

1-Bromo-2-(3-methylbut-3-en-1-yl)benzene (S1a): A flame-dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen. 2-bromobenzyl bromide (1.25 g, 5.0 mmol), and THF (5 mL) were added to the flask and the resulting solution was cooled to 0 °C. 2-methylallylmagnesium chloride (20 mL, 10 mmol, 0.5 M solution in THF) was slowly added and the resulting mixture was moved into an oil bath and heated to 40 °C for 1.5 h. The mixture was then cooled to rt and quenched with 4 mL of 2M H\(_2\)SO\(_4\). Water (5 mL) and ether (15 mL) were added and the mixture was transferred to a separatory funnel. The layers were separated, the aqueous layer was extracted with diethyl ether (3x15 mL), and the organic layers were then combined, dried over anhydrous sodium sulfate, and concentrated in vacuo. The product was purified via flash chromatography on silica gel using hexanes as the eluent to afford 1.06 g (95\%) of the title compound as a clear oil. \(^1\)H NMR (700 MHz, CDCl\(_3\)) \(\delta\) 7.54 (d, \(J = 7.8\) Hz, 1 H), 7.25–7.21 (m, 1 H), 7.08–7.02 (m, 1 H), 4.79 (s, 1 H), 4.76 (s, 1 H), 2.91–2.86 (m, 2 H), 2.32 (t, \(J = 8.6\) Hz, 2 H), 2.81 (s, 3 H); \(^{13}\)C NMR (175 MHz, CDCl\(_3\)) \(\delta\)
145.1; 141.5; 132.9; 130.36; 127.7; 127.5; 124.6; 110.7; 38.1; 34.9; 22.7; IR (film) 2933, 1648, 1439 cm⁻¹.

**1-Bromo-4-methoxy-2-(3-methylbut-3-en-1-yl)benzene (S1b):** The conversion of 2-bromo-5-methoxybenzyl bromide (1.40 g, 5.0 mmol) was accomplished using a procedure analogous to that described above for the preparation of S1b except using 2.5 equiv of 2-methylallylmagnesium chloride. This procedure afforded 1.15 g (90%) of the title compound as a clear oil. ¹H NMR (700 MHz, CDCl₃) δ 7.40 (d, J = 8.7 Hz, 1H), 6.77 (d, J = 3.1 Hz, 1H), 6.62 (dd, J = 3.1, 8.7 Hz, 1H), 4.77 (s, 1H), 4.75 (s, 1H), 3.78 (s, 3H), 2.83–2.78 (m, 2H), 2.29 (t, J = 8.5 Hz, 2H), 1.79 (s, 3H); ¹³C NMR (175 MHz, CDCl₃) δ 159.1, 145.3, 142.6, 133.4, 116.1, 115.0, 113.2, 110.6, 55.6, 38.0, 35.1, 22.7; IR (film) 2933, 1571, 1471 cm⁻¹.

**1-Bromo-2-(3-methylbut-3-en-1-yl)naphthalene (S1c):** The conversion of 1-bromo-2-(bromomethyl)naphthalene (0.60 g, 2.0 mmol) was accomplished using a procedure analogous to that described above for the preparation of S1a except using 2.5 equiv of 2-methylallylmagnesium chloride and a reaction temperature of 45 °C instead of 40 °C for the heated segment of the reaction. This procedure afforded 1.15 g (80%) of the title compound as a clear oil. ¹H NMR (700 MHz, CDCl₃) δ 8.32 (d, J = 8.5 Hz, 1H), 7.80 (d, J = 8.0 Hz, 1H), 7.74 (d, J = 8.3 Hz, 1H), 7.58 (t, J = 7.0 Hz, 1H), 7.48 (t, J = 8.0 Hz, 1H), 7.36 (d, J = 8.3 Hz, 1H), 4.78 (d, J = 5.1 Hz, 2H), 3.12 (t, J = 8.8 Hz, 2H), 2.39 (t, J
= 8.9 Hz, 2 H), 1.85 (s, 3 H); $^{13}$C NMR (175 MHz, CDCl$_3$) δ 145.4, 139.8, 133.4, 132.8, 128.19, 128.18, 127.7, 127.5, 127.4, 126.0, 123.8, 110.7, 38.3, 36.2, 22.8; IR (film) 2916, 1603, 1494 cm$^{-1}$.

**General procedure A: addition of a Grignard reagent to 3-(2-bromophenyl)-N-methoxy-N-methylpropanamide.** A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with 3-(2-bromophenyl)-N-methoxy-N-methylpropanamide$^3$ (1.0 equiv) and diethyl ether (0.40 M) was added to the flask and it was cooled to 0 °C. The appropriate Grignard reagent (1.5 equiv) was added dropwise and the reaction mixture was allowed to slowly warm to rt and stir for 5 hours. The reaction mixture was then cooled to 0 °C and quenched with saturated ammonium chloride (1mL/mmol substrate) and then water (5mL/mmol substrate) was added. This mixture was transferred to a separatory funnel and extracted with EtOAc. The combined organic layers were dried with sodium sulfate and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel.

3-(2-Bromophenyl)-1-phenylpropan-1-one (**S2a**).$^4$ General procedure A was used for the reaction of 3-(2-bromophenyl)-N-methoxy-N-methylpropanamide (2.72 g, 10 mmol), with phenylmagnesium bromide (1 M THF, 15 mL). This procedure afforded 1.92 g (66%) of the title compound as a clear oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.98 (d, $J = 8.1$ Hz, 2 H), 7.57–7.52 (m, 2H), 7.45 (t, $J = 7.8$ Hz, 2 H), 7.31 (d, $J = 6.9$ Hz, 1 H), 7.24 (t, $J = 8.1$ Hz, 1 H), 7.07 (t, $J = 7.3$ Hz, 1 H), 2.82 (t, $J = 7.3$ Hz, 2 H), 2.45 (t, $J = 7.8$ Hz, 2 H); $^{13}$C NMR (125 MHz, CDCl$_3$) δ 199.0, 140.6, 136.8, 133.2, 132.9, 130.9, 128.7, 128.1, 128.0, 127.7, 124.4, 38.7, 30.1.
1-(2-Bromophenyl)pentan-3-one (S2b). General procedure A was used for the reaction of 3-(2-bromophenyl)-N-methoxy-N-methylpropanamide (1.10 g, 4.04 mmol), with ethylmagnesium bromide (3 M THF, 2.02 mL). This procedure afforded 775 mg (80%) of the title compound as a clear oil. $^1$H NMR (700 MHz, CDCl$_3$) $\delta$ 7.47 (d, $J$ = 7.8 Hz, 1 H), 7.21–7.16 (m, 2 H), 7.01 (t, $J$ = 8.0 Hz, 1 H), 2.97 (t, $J$ = 7.7 Hz, 2 H), 2.70 (t, $J$ = 7.8 Hz, 2 H), 2.48 (q, $J$ = 7.3 Hz, 2 H), 1.01 (t, $J$ = 7.2 Hz, 3 H); $^{13}$C NMR (175 MHz, CDCl$_3$) $\delta$ 210.0, 140.3, 132.7, 130.5, 127.8, 127.5, 124.1, 41.9, 35.9, 30.3, 7.7; IR (film) 2973, 2936, 1711 cm$^{-1}$.

1-(2-Bromophenyl)-4-methylpentan-3-one (S2c). General procedure A was used for the reaction of 3-(2-bromophenyl)-N-methoxy-N-methylpropanamide (2.72 g, 10 mmol), with isopropylmagnesium chloride (2 M THF, 7.50 mL). This procedure afforded 625 mg (25%) of the title compound as a clear oil. $^1$H NMR (700 MHz, CDCl$_3$) $\delta$ 7.50 (d, $J$ = 8.0 Hz, 1 H), 7.24–7.19 (m, 2 H), 7.05 (t, $J$ = 8.2 Hz, 1 H), 3.00 (t, $J$ = 7.5 Hz, 2 H), 2.77 (t, $J$ = 7.7 Hz, 2 H), 2.57 (sept, $J$ = 7.0 Hz, 1 H), 1.06 (d, $J$ = 7.0 Hz, 6 H); $^{13}$C NMR (175 MHz, CDCl$_3$) $\delta$ 213.4, 140.6, 132.8, 130.7, 127.9, 127.5, 124.2, 41.0, 40.0, 30.5, 18.1; IR (film) 2967, 2932, 1708 cm$^{-1}$.

**General procedure B: Wittig methylenation of ketones.** A flame dried round bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with methyltriphenylphosphonium bromide (1.4 equiv) and THF (0.15 M). The resulting solution was cooled to 0 °C then potassium tert-butoxide (1.4 equiv) added in one portion and the mixture was allowed to stir at 0 °C for 45 min. To this mixture was slowly
added a solution of the appropriate ketone (1.0 equiv) in THF (0.65 M). The reaction mixture was then allowed to slowly warm to rt and stir for 16 hours. The mixture was concentrated in vacuo, diluted with hexanes, and then filtered through celite. The filtrate was then concentrated in vacuo and the crude product was purified by flash chromatography on silica gel.

1-Bromo-2-(3-phenylbut-3-en-1-yl)benzene (S1d). General procedure B was used for the reaction of methyltriphenylphosphonium bromide (3.31 g, 9.28 mmol), with 3-(2-bromophenyl)-1-phenylpropan-1-one (1.92 g, 6.63 mmol). This procedure afforded 1.06 g (56%) of the title compound as a clear oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.53 (d, $J = 8.0$ Hz, 1 H), 7.46 (d, $J = 8.2$ Hz, 2 H), 7.34 (t, $J = 6.3$ Hz, 2 H), 7.30–7.13 (m, 3H), 7.04 (t, $J = 8.0$ Hz, 1 H), 5.31 (s, 1 H), 5.08 (s, 1 H), 2.91–2.83 (m, 2 H), 2.82–2.76 (m, 2 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 147.6, 141.2, 140.9, 132.8, 130.6, 128.4, 127.7, 127.5, 127.4, 126.2, 124.4, 112.9, 35.5, 35.4.

1-Bromo-2-(3-methylenepentyl)benzene (S1e). General procedure B was used for the reaction of methyltriphenylphosphonium bromide (620 mg, 1.73 mmol), with 1-(2-bromophenyl)pentan-3-one (300 mg, 1.24 mmol). This procedure afforded 163 mg (55%) of the title compound as a clear oil. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.54 (d, $J = 7.8$ Hz, 1 H), 7.23 (d, $J = 4.7$ Hz, 2 H), 7.08–7.04 (m, 1 H), 4.80 (s, 2 H), 2.89 (t, $J = 8.2$ Hz, 2 H), 2.35 (t, $J = 8.6$ Hz, 2 H), 2.13 (q, $J = 7.4$ Hz, 2 H), 1.08 (t, $J = 7.4$ Hz, 3 H); $^{13}$C NMR (175 MHz, CDCl$_3$) $\delta$ 150.7, 141.6, 132.8, 130.3, 127.6, 127.4, 124.4, 108.3, 36.4, 35.1, 29.0, 12.4; IR (film) 3024, 2920, 1494 cm$^{-1}$. 
1-Bromo-2-(4-methyl-3-methylenepentyl)benzene (S1f). General procedure B was used for the reaction of methyltriphenylphosphonium bromide (1.19 g, 3.34 mmol), with 1-(2-bromophenyl)-4-methylpentan-3-one (610 mg, 2.40 mmol). This procedure afforded 230 mg (38%) of the title compound as a clear oil. $^1$H NMR (700 MHz, CDCl$_3$) $\delta$ 7.55 (d, $J$ = 7.8 Hz, 1 H), 7.25 (d, $J$ = 4.4 Hz, 2 H), 7.09–7.05 (m, 1 H), 4.86 (s, 1 H), 4.81 (s, 1 H), 2.89 (t, $J$ = 8.2 Hz, 2 H), 2.37–2.31 (m, 3 H), 1.10 (d, $J$ = 7.0 Hz, 6 H); $^{13}$C NMR (175 MHz, CDCl$_3$) $\delta$ 155.2, 141.7, 132.8, 130.3, 127.6, 127.4, 124.4, 107.0, 35.4, 34.5, 34.0, 21.9; IR (film) 2959, 2927, 1470 cm$^{-1}$.

General procedure C: synthesis of 2-(3-methylbut-3-en-1-yl)aniline substrates. A flame dried Schlenk flask equipped with a stir bar was cooled under a stream of nitrogen and charged with Pd$_2$(dba)$_3$ (0.75 mol %), XPhos (2.25 mol %), 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (1.0 equiv), the appropriate aniline derivative (1.2 equiv), and NaO$^\text{Bu}$ (1.5 equiv). The flask was then purged with nitrogen, and toluene (0.5 M) was added. The resulting mixture was heated to 105 °C with stirring until the starting material had been consumed as judged by TLC, GC, or $^1$H NMR analysis of an aliquot removed from the reaction mixture (ca. 12 h). The reaction mixture was then cooled to rt, saturated aqueous ammonium chloride (6 mL/mmol substrate) was added, and the mixture was transferred to a separatory funnel. The mixture was extracted with ethyl acetate (3 x 20 mL) then the organic layers were combined, dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel using a hexanes/Et$_2$O mixture as the eluent.
2-(But-3-en-1-yl)-N-(4-methoxyphenyl)aniline (1a). General Procedure C was employed for the coupling of 1-bromo-2-(but-3-en-1-yl)benzene (211 mg, 1.0 mmol) and p-anisidine (147 mg, 1.2 mmol). This procedure afforded 190 mg (75%) of the title compound as a yellow oil. $^1$H NMR (700 MHz, CDCl$_3$) $\delta$ 7.18 (d, $J = 7.7$ Hz, 1 H), 7.09 (t, $J = 7.6$ Hz, 1 H), 7.03 (d, $J = 8.0$ Hz, 1 H), 6.98 (d, $J = 8.7$ Hz, 2 H), 6.89–6.83 (m, 3H), 5.95–5.89 (m, 1H), 5.18 (s, br, 1 H), 5.09 (app. d, $J = 17.0$ Hz, 1 H), 4.86 (app. d, $J = 10.2$ Hz, 1 H), 4.09 (s, 3 H), 2.69 (t, $J = 7.5$ Hz, 2 H), 2.42 (q, $J = 6.6$ Hz, 2 H); $^{13}$C NMR (175 MHz, CDCl$_3$) $\delta$ 155.0, 142.9, 138.2, 136.9, 129.8, 129.7, 127.0, 121.7, 120.6, 116.8, 115.3, 114.8, 55.7, 33.5, 30.9; IR (film) 3402, 2924, 1508 cm$^{-1}$; MS (ESI+) 254.1543 (254.1539 calcd for C$_{17}$H$_{19}$NO, M + H$^+$).

N-(4-Methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (1b). General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (1.125 g, 5.0 mmol) and p-anisidine (738 mg, 6.0 mmol). This procedure afforded 1.09 g (82%) of the title compound as a yellow oil. $^1$H NMR (700 MHz, CDCl$_3$) $\delta$ 7.28 (d, $J = 7.7$ Hz, 1 H), 7.18 (t, $J = 8.7$ Hz, 1 H), 7.14 (d, $J = 8.4$ Hz, 1 H), 7.06 (d, $J = 8.4$ Hz, 2 H), 6.98–6.92 (m, 3H), 5.40 (s, br, 1 H), 4.88 (s, 1 H), 4.86 (s, 1 H), 3.86 (s, 3 H), 2.82 (t, $J = 8.4$ Hz, 2 H), 2.45 (t, $J = 8.4$ Hz, 2 H), 1.88 (s, 3 H); $^{13}$C NMR (175 MHz, CDCl$_3$) $\delta$ 155.0, 145.6, 142.8, 137.0, 130.2, 129.7, 127.0, 121.5, 120.7, 117.0, 114.8, 110.5, 55.6, 37.4, 29.9, 22.8; IR (film) 3398, 2933, 1507 cm$^{-1}$; MS (ESI+) 268.1704 (268.1696 calcd for C$_{18}$H$_{21}$NO, M + H$^+$).
4-[(2-(3-Methylbut-3-en-1-yl)phenyl)amino]benzonitrile (1c). General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (225 mg, 1.0 mmol) and p-cyanoaniline (142 mg, 1.20 mmol) except using a catalyst loading of 1 mol % Pd$_2$(dba)$_3$. This procedure afforded 223 mg (28%) of the title compound as a yellow oil. $^1$H NMR (500 MHz, CDCl$_3$) δ 7.44 (d, $J = 11.9$ Hz, 2 H), 7.30 (d, $J = 11.2$ Hz, 1 H), 7.28–7.16 (m, 3 H), 6.75 (d, $J = 12.6$ Hz, 2 H), 5.89 (s, br, 1 H), 4.74 (s, 1 H), 4.65 (s, 1 H), 2.73 (t, $J = 10.5$ Hz, 2 H), 2.26 (t, $J = 11.9$ Hz, 2 H), 1.72 (s, 3 H); 13C NMR (175 MHz, CDCl$_3$) δ 149.9, 145.2, 137.8, 137.4, 133.8, 130.5, 127.3, 126.1, 125.3, 120.3, 114.2, 110.9, 100.5, 38.4, 30.1, 22.7; IR (film) 3338, 2927, 2213, 1513 cm$^{-1}$; MS (ESI+) 263.1546 (263.1543 calcd for C$_{18}$H$_{18}$N$_2$, M + H$^+$).

N-[4-(tert-Butyl)phenyl]-2-(3-methylbut-3-en-1-yl)aniline (1d). General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (225 mg, 1.0 mmol) and p-tert-butylaniline (0.19 mL, 1.2 mmol). This procedure afforded 210 mg (72%) of the title compound as a yellow oil. $^1$H NMR (700 MHz, CDCl$_3$) δ 7.41 (d, $J = 9.1$ Hz, 2 H), 7.37 (d, $J = 7.7$ Hz, 1 H), 7.32 (d, $J = 7.7$ Hz, 1 H), 7.25 (t, $J = 7.7$ Hz, 1 H), 7.09–7.03 (m, 3 H), 5.52 (s, br, 1 H), 4.91 (s, 1 H), 4.88 (s, 1 H), 2.87 (t, $J = 8.4$ Hz, 2 H), 2.46 (t, $J = 9.1$ Hz, 2 H), 1.90 (s, 3 H), 1.45 (s, 9 H); 13C NMR (175 MHz, CDCl$_3$) δ
145.6, 143.5, 141.7, 141.5, 132.1, 129.9, 127.0, 126.2, 121.9, 119.2, 117.6, 110.7, 37.8, 34.2, 31.7, 30.1, 22.8; IR (film) 3398, 2961, 1514 cm\(^{-1}\); MS (ESI+) 294.2228 (294.2216 calcd for C\(_{21}\)H\(_{27}\)N, M + H\(^+\)).

\(\text{N',N'-Dimethyl-N'[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (1e).}\)

General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)benzene (1.10 g, 4.88 mmol) and 4-(dimethylamino)aniline (798 mg, 5.86 mmol). This procedure afforded 1.16 g (85%) of the title compound as a yellow oil. \(^1\)H NMR (700 MHz, C\(_6\)D\(_6\)) \(\delta\) 7.15–7.13 (m, 1 H), 7.10 (d, \(J = 9.7\) Hz, 1 H), 7.07 (t, \(J = 7.8\) Hz, 1 H), 6.98 (d, \(J = 8.9\) Hz, 2 H), 6.86 (t, \(J = 7.3\) Hz, 1 H), 6.61 (d, \(J = 8.9\) Hz, 2 H), 5.06 (s, 1 H), 4.78 (s, 2 H), 2.61 (t, \(J = 7.8\) Hz, 2 H), 2.56 (s, 6 H), 2.29 (t, \(J = 8.5\) Hz, 2 H), 1.62 (s, 3 H); \(^{13}\)C NMR (175 MHz, C\(_6\)D\(_6\)) \(\delta\) 147.5, 145.6, 144.5, 134.0, 129.8, 129.4, 127.3, 123.4, 120.2, 116.4, 114.5, 110.8, 41.0, 37.5, 30.2, 22.7; IR (film) 3410, 2934, 1516 cm\(^{-1}\); MS (ESI+) 281.2018 (281.2012 calcd for C\(_{19}\)H\(_{24}\)N\(_2\), M + H\(^+\)).

\(4\text{-Methoxy-N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (1f).}\)

General Procedure C was employed for the coupling of 1-bromo-4-methoxy-2-(3-methylbut-3-en-1-yl)benzene (420 mg, 1.65 mmol) and \(p\)-anisidine (243 mg, 1.98 mmol) and a reaction time of 3 h. This procedure afforded 421 mg (86%) of the title compound as an orange
oil. \(^1\)H NMR (700 MHz, C\(_6\)D\(_6\)) \(\delta\) 7.05 (d, \(J = 8.7\) Hz, 1 H), 6.88 (d, \(J = 2.7\) Hz, 1 H), 6.77 (d, \(J = 8.9\) Hz, 2 H), 6.68–6.62 (m, 3 H), 4.77–4.74 (m, 2 H), 4.66 (s, br, 1 H), 3.40 (s, 3 H), 3.36 (s, 3 H), 2.64 (t, \(J = 8.9\) Hz, 2 H), 2.24 (t, \(J = 8.3\) Hz, 2 H), 1.59 (s, 3 H); \(^{13}\)C NMR (175 MHz, C\(_6\)D\(_6\)) \(\delta\) 156.3, 153.7, 145.1, 140.3, 137.0, 135.0, 124.4, 117.3, 115.6, 114.8, 111.9, 110.4, 54.8, 54.7, 38.0, 30.2, 22.2; IR (film) 3379, 2933, 1509 cm\(^{-1}\); MS (ESI\(^+\)) 297.1726 (297.1723 calcd for C\(_{19}\)H\(_{23}\)NO\(_2\), M + H\(^+\)).

\(N\)-(4-Methoxyphenyl)-2-(3-methylbut-3-en-1-yl)naphthalen-1-amine (1g). General Procedure C was employed for the coupling of 1-bromo-2-(3-methylbut-3-en-1-yl)naphthalene (380 mg, 1.38 mmol) and \(p\)-anisidine (204 mg, 1.65 mmol) except using a catalyst composed of Pd\(_2\)(dba)\(_3\) (25 mg, 0.0276 mmol, 2.0 mol %) and JohnPhos (16.5 mg, 0.0552 mmol, 4.0 mol %). This procedure afforded 308 mg (70%) of the title compound as a yellow solid, mp 80–84 °C. \(^1\)H NMR (700 MHz, C\(_6\)D\(_6\)) \(\delta\) 8.06 (d, \(J = 8.2\) Hz, 1 H), 7.71 (d, \(J = 7.2\) Hz, 1 H), 7.59 (d, \(J = 8.3\) Hz, 1 H), 7.28–7.23 (m, 3 H), 6.66 (d, \(J = 8.9\) Hz, 2 H), 6.36 (d, \(J = 8.9\) Hz, 2 H), 4.93 (s, 1H), 4.73 (d, \(J = 15.2\) Hz, 2 H), 3.30 (s, 3 H), 2.80 (t, \(J = 8.8\) Hz, 2 H), 2.24 (t, \(J = 8.2\) Hz, 2 H), 1.60 (s, 3 H); \(^{13}\)C NMR (175 MHz, C\(_6\)D\(_6\)) \(\delta\) 153.4, 145.2, 142.1, 136.7, 135.9, 134.2, 132.5, 128.6, 128.5, 128.4, 126.4, 125.7, 124.6, 115.4, 115.2, 111.0, 55.2, 39.1, 31.1, 22.5; IR (film) 3382, 2959, 1505 cm\(^{-1}\); MS (ESI\(^+\)) 317.1771 (317.1771 calcd for C\(_{22}\)H\(_{23}\)NO, M + H\(^+\)).

\(N\)-(4-Methoxyphenyl)-2-(3-methylenepentyl)aniline (1h). General Procedure C was
employed for the coupling of 1-bromo-2-(3-methylenepentyl)benzene (160 mg, 0.67 mmol) and \( p \)-anisidine (99 mg, 0.80 mmol) except using a catalyst composed of \( \text{Pd}_2(\text{dba})_3 \) (9.2 mg, 0.010 mmol, 1.5 mol \%) and XPhos (12.7 mg, 0.0268 mmol, 4.0 mol \%). This procedure afforded 170 mg (90\%) of the title compound as a pale yellow oil. \(^1\)H NMR (500 MHz, CDCl\(_3\)) \( \delta \) 7.17 (d, \( J = 7.3 \) Hz, 1 H), 7.09 (t, \( J = 7.8 \) Hz, 1 H), 7.04 (d, \( J = 7.8 \) Hz, 1 H), 6.98 (d, \( J = 9.2 \) Hz, 2 H), 6.90–6.83 (m, 3H), 5.30 (s, br, 1 H), 4.8 (s, 2 H), 3.80 (s, 3 H), 2.73 (t, \( J = 7.8 \) Hz, 2 H), 2.39 (t, \( J = 8.3 \) Hz, 2 H), 2.10 (q, \( J = 7.3 \) Hz, 2 H), 1.06 (t, \( J = 7.3 \) Hz, 3 H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \( \delta \) 155.0, 151.2, 142.8, 136.9, 130.2, 129.6, 126.9, 121.3, 120.6, 116.7, 114.8, 108.2, 55.7, 35.9, 30.2, 29.1, 12.4; IR (film) 3392, 2960, 1508 cm\(^{-1}\); MS (ESI+) 282.1849 (282.1852 calcd for C\(_{19}\)H\(_{23}\)NO, M + H\(^+\)).

![Chemical Structure](image)

**N-(4-Methoxyphenyl)-2-(3-phenylbut-3-en-1-yl)aniline (1i).** General Procedure C was employed for the coupling of 1-bromo-2-(3-phenylbut-3-en-1-yl)benzene (1.06 g, 3.70 mmol) and \( p \)-anisidine (547 mg, 4.44 mmol). This procedure afforded 1.18 g (95\%) of the title compound as a pale yellow oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.50 (d, \( J = 7.6 \) Hz, 2 H), 7.42–7.32 (m, 3 H), 7.24–7.08 (m, 3 H), 6.98–6.85 (m, 5 H), 5.38 (s, 1 H), 5.20 (s, br, 1 H), 5.14 (s, 1H), 3.83 (s, 3 H), 2.92 (t, \( J = 8.8 \) Hz, 2 H), 2.80 (t, \( J = 9.0 \) Hz, 2 H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \( \delta \) 154.8, 147.7, 147.6, 142.6, 140.7, 136.9, 130.1, 129.8, 128.5, 127.6, 127.0, 126.1, 121.1, 120.7, 117.2, 114.7, 113.0, 55.6, 35.3, 30.5; IR (film) 3406, 2938, 1508 cm\(^{-1}\); MS (ESI+) 330.1852 (330.1852 calcd for C\(_{23}\)H\(_{23}\)NO, M + H\(^+\)).
**N-(4-Methoxyphenyl)-2-(4-methyl-3-methylenepentyl)aniline (1j).** General Procedure C was employed for the coupling of 1-bromo-2-(4-methyl-3-methylenepentyl)benzene (150 mg, 0.59 mmol) and p-anisidine (88 mg, 0.71 mmol) except using a catalyst composed of Pd$_2$(dba)$_3$ (11 mg, 0.0118 mmol, 2.0 mol %) and XPhos (11 mg, 0.0236 mmol, 4.0 mol %). This procedure afforded 170 mg (97%) of the title compound as a pale yellow oil. $^1$H NMR (700 MHz, CDCl$_3$) $\delta$ 7.16 (d, $J = 7.3$ Hz, 1 H), 7.08 (t, $J = 8.0$ Hz, 1 H), 7.03 (d, $J = 7.8$ Hz, 1 H), 6.98 (d, $J = 8.7$ Hz, 2 H), 6.87–6.83 (m, 3H), 5.29 (s, br, 1 H), 4.82 (s, 1 H), 4.77 (s, 1 H), 3.79 (s, 3 H), 2.72 (t, $J = 8.0$ Hz, 2 H), 2.36 (t, $J = 8.5$ Hz, 2 H), 2.28 (sept, $J = 7.0$ Hz, 1 H), 1.04 (d, $J = 6.8$ Hz, 6 H); $^{13}$C NMR (175 MHz, CDCl$_3$) $\delta$ 155.7, 155.0, 142.8, 136.8, 130.2, 129.7, 126.9, 121.7, 120.5, 116.6, 114.7, 106.9, 55.7, 34.1, 33.9, 30.4, 21.9; IR (film) 3389, 2958, 1508 cm$^{-1}$; MS (ESI+) 296.2007 (296.2009 calcd for C$_{20}$H$_{25}$NO, M + H$^+$).

**N-Methyl-N-(2-methylallyl)-2-nitroaniline (S3a).** A flame dried flask equipped with a stir bar was cooled under nitrogen and charged with 1-Fluoro-2-nitrobenzene (2.10 mL, 20 mmol) and anhydrous DMF (20 mL). Methylamine (20 mL, 40 mmol, 2.0 M in THF) was added and the resulting mixture was heated to 50 °C for 12 hours. The reaction mixture was then cooled to rt, and excess methylamine and THF were evaporated in vacuo to afford crude N-methyl-2-nitroaniline, which was dissolved in DMF (15 mL) and added slowly to a flame dried flask containing a suspension of NaH (880 mg, 22 mmol, 60% in mineral oil) in DMF (10 mL) that had been cooled to 0 °C. The resulting mixture was stirred for 30 minutes at 0 °C then 3-bromo-2-methylpropene (3.05 mL, 30 mmol),
was added slowly. The ice bath was removed, the reaction flask was placed in an oil bath (rt) and then was heated to 100 °C for 6 h. The mixture was then allowed to cool to room temperature and saturated aqueous NH₄Cl (40 mL) and EtOAc (80 mL) were added. The mixture was transferred to a separatory funnel, the layers were separated, and the organic layer was separated and washed with brine (2 x 10 mL). The organic layer was then dried over anhydrous sodium sulfate, filtered and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel using hexanes/Et₂O as the eluant to afford 2.25 g (51%) of the title compound as a yellow oil. 

\[ ^1H \text{NMR (400 MHz, CDCl}_3 \text{)} \delta 7.70 (d, J = 8.2 \text{ Hz}, 1 \text{ H}), 7.34 (t, J = 7.8 \text{ Hz}, 1 \text{ H}), 7.01 (d, J = 8.4 \text{ Hz}, 1 \text{ H}), 6.81 (t, J = 7.6 \text{ Hz}, 1 \text{ H}), 4.91 (s, 1 \text{ H}), 4.84 (s, 1 \text{ H}), 3.71 (s, 2 \text{ H}), 2.76 (s, 3 \text{ H}), 1.69 (s, 3 \text{ H}); ^{13}C \text{NMR (100 MHz, CDCl}_3 \text{)} \delta 145.8, 140.6, 140.1, 133.1, 126.6, 119.8, 118.5, 113.1, 60.6, 40.2, 20.2; \text{IR (film) 2914, 1604, 1512 cm}^{-1}. \]

\[
\text{N-Methyl-2-nitro-N-(2-phenylallyl)aniline (S3b). Following the above procedure, 1-Fluoro-2-nitrobenzene (0.97 mL, 9.25 mmol) and methylamine (9.25 mL, 18.5 mmol, 2.0 M in THF) were reacted to form the crude N-methyl-2-nitroaniline. This crude product was added dropwise to a flame dried round bottom flask containing a suspension of NaH (60% in mineral oil 406 mg, 10.15 mmol) in DMF at 0 °C. Neat (3-bromoprop-1-en-2-yl)benzene}^5 \text{ (2.73 g, 13.85 mmol) was added to this mixture. This procedure afforded 1.64 g (66%) of the title compound as a yellow oil. }^1H \text{NMR (400 MHz, CDCl}_3 \text{)} \delta 7.74 (d, J = 8.4 \text{ Hz}, 1 \text{ H}), 7.38–7.26 (m, 6 \text{ H}), 6.95 (d, J = 8.6 \text{ Hz}, 1 \text{ H}), 6.82 (t, J = 7.4 \text{ Hz}, 1 \text{ H}), 5.51 (s, 1 \text{ H}), 5.22 (s, 1 \text{ H}), 4.22 (s, 2 \text{ H}), 2.84 (s, 3 \text{ H}); ^{13}C \text{NMR (100 MHz, CDCl}_3 \text{)} \delta 145.4, 142.5, 139.6, 138.9, 132.9, 128.3, 127.9, 126.3, 126.0, 119.5, 118.2, 114.3, 58.0, 39.9; \text{IR (film) 2931, 1604, 1512 cm}^{-1}. \]
**N-{2-[(Benzyloxy)methyl]allyl}-N-methyl-2-nitroaniline (S3c).** Following the above procedure, 1-Fluoro-2-nitrobenzene (0.84 mL, 8.00 mmol) and methylamine (8 mL, 16 mmol, 2.0 M in THF) were reacted to form the crude N-methyl-2-nitroaniline. This crude product was added dropwise to a flame dried round bottom flask containing a suspension of NaH (60% in mineral oil, 352 mg, 8.80 mmol) in DMF at 0 °C. Neat ([(2-(chloromethyl)allyl)oxy]methyl)benzene6 (2.35 g, 12.0 mmol) was added to this mixture. This procedure afforded 810 mg (33%) of the title compound as a yellow oil containing a slight unknown impurity. This material was used without further purification. ¹H NMR (400 MHz, CDCl₃) δ 7.72 (d, J = 7.6 Hz, 1 H), 7.40–7.23 (m, 6 H), 7.05 (d, J = 8.4 Hz, 1 H), 6.82 (t, J = 7.2 Hz, 1 H), 5.24 (s, 1 H), 5.12 (s, 1 H), 4.46 (s, 2 H), 3.98 (s, 2 H), 3.89 (s, 2 H), 2.76 (s, 3 H); ¹³C NMR (100 MHz, CDCl₃) δ 160.6, 145.6, 141.1, 138.1, 133.0, 128.4, 127.7, 127.6, 126.4, 119.8, 118.5, 115.1, 72.4, 71.3, 56.7, 40.4; IR (film) 2916, 2848, 1511 cm⁻¹.

**N'-Methyl-N'-(2-methylallyl)benzene-1,2-diamine (S4a).** A flame dried flask equipped with a stir bar was cooled under nitrogen and charged with zinc dust (8.56 g, 130.9 mmol) and anhydrous ethanol (75 mL). The resulting suspension was vigorously stirred, glacial acetic acid (7.50 mL, 131 mmol) was added, and the mixture was cooled to 0 °C. A solution of N-methyl-N-(2-methylallyl)-2-nitroaniline (1.80 g, 8.7 mmol) in anhydrous ethanol (15 mL) was added, the ice bath was removed, and the mixture was stirred vigorously at room temperature for 2 h. The mixture was then filtered through celite and the filtrate was evaporated in vacuo. The resulting material was dissolved in EtOAc (50 mL), transferred to a separatory funnel, and washed with saturated aqueous NaHCO₃.
The layers were separated and the aqueous layer was washed with EtOAc (2 x 50 mL). The combined organic layers were then washed with brine (30 mL), dried over anhydrous sodium sulfate, and concentrated in vacuo. The crude product was purified by flash chromatography on silica gel using hexanes/Et2O as the eluant to afford 1.12 g (73%) of the title compound as a red oil. 1H NMR (700 MHz, CDCl3) δ 7.02 (d, J = 7.8 Hz, 1 H), 6.91 (t, J = 7.8 Hz, 1 H), 6.76–6.71 (m, 2 H), 5.03 (s, 1 H), 4.90 (s, 1 H), 4.00 (s, br, 2 H), 3.35 (s, 2 H), 2.58 (s, 3 H), 1.78 (s, 3 H); 13C NMR (175 MHz, CDCl3) δ 143.2, 141.8, 140.4, 124.4, 120.5, 118.6, 115.3, 112.5, 62.6, 40.5, 20.6; IR (film) 3441, 2970, 1607, 1499 cm⁻¹.

**N'1-methyl-N'1-(2-phenylallyl)benzene-1,2-diamine (S4b).** Following the above procedure, zinc dust (5.99 g, 91.7 mmol), acetic acid (5.30 mL, 91.7 mmol), and N'-methyl-2-nitro-N'(2-phenylallyl)aniline (1.64 g, 6.11 mmol) were reacted to afford 802 mg (55%) of the title compound as a red oil. 1H NMR (500 MHz, CDCl3) δ 7.44 (d, J = 7.6 Hz, 2 H), 7.35–7.25 (m, 3 H), 7.08 (d, J = 7.8 Hz, 1 H), 6.93 (t, J = 7.6 Hz, 1 H), 6.75 (t, J = 7.8 Hz, 1 H), 6.70 (d, J = 7.8 Hz, 1 H), 5.46 (s, 1 H), 5.39 (s, 1 H), 3.88 (s, 2 H), 3.78 (s, br, 2 H), 2.60 (s, 3 H); 13C NMR (100 MHz, CDCl3) δ 145.6, 142.1, 140.1, 139.9, 128.2, 127.6, 126.5, 124.6, 120.9, 118.3, 115.1, 114.6, 60.4, 40.6; IR (film) 3439, 3347, 1606, 1499 cm⁻¹.

**N'1-{2-[(Benzyloxy)methyl]allyl}-N'1-methylbenzene-1,2-diamine (S4c).** Following the above procedure, zinc dust (2.55 g, 39 mmol), acetic acid (2.25 mL, 39 mmol), and N-
{2-[(benzyloxy)methyl]allyl}-N-methyl-2-nitroaniline (810 mg, 2.60 mmol) were reacted to afford 372 mg (50%) of the title compound as a red oil. \( ^1 \)H NMR (700 MHz, CDCl\(_3\)) \( \delta \) 7.36–7.32 (m, 4 H), 7.30–7.27 (m, 1 H), 7.01 (d, \( J = 7.7 \) Hz, 1 H), 6.89 (t, \( J = 7.5 \) Hz, 1 H), 6.70 (t, \( J = 7.7 \) Hz, 1 H), 6.67 (d, \( J = 7.8 \) Hz, 1 H), 5.26 (s, 1 H), 5.22 (s, 1 H), 4.50 (s, 2 H), 4.09 (s, 2 H), 4.03 (s, br, 2 H), 3.49 (s, 2 H), 2.59 (s, 3 H); \( ^{13} \)C NMR (175 MHz, CDCl\(_3\)) \( \delta \) 143.4, 141.8, 139.6, 138.2, 128.4, 127.8, 127.7, 124.4, 120.3, 118.1, 115.2, 115.1, 72.0, 71.5, 58.8, 40.4; IR (film) 2920, 1604, 1494 cm\(^{-1}\).

\[ N^1-(4\text{-methoxyphenyl})-N^2\text{-methyl-}N^2\text{-}(2\text{-methylallyl})\text{benzene-1,2-diamine} \quad (3a). \]

General Procedure C was employed for the coupling of 4-bromoanisole (0.80 mL, 6.35 mmol) and \( N^1 \)-methyl-\( N^1 \)-(2-methylallyl)benzene-1,2-diamine (1.12 g, 6.35 mmol), except using a catalyst composed of Pd\(_2\)(dba)\(_3\) (116 mg, 0.127 mmol, 2.0 mol %) and JohnPhos (75 mg, 0.250 mmol, 4.0 mol %) and a reaction temperature of 85 \(^\circ\)C. This procedure afforded 1.49 g (79%) of the title compound as a red oil. \( ^1 \)H NMR (700 MHz, CDCl\(_3\)) \( \delta \) 7.16–7.10 (m, 4 H), 6.98 (t, \( J = 7.3 \) Hz, 1 H), 6.89 (d, \( J = 7.5 \) Hz, 2 H), 6.81 (t, \( J = 7.3 \) Hz, 1 H), 6.57 (s, br, 1 H), 5.07 (s, 1 H), 4.95 (s, 1 H), 3.83 (s, 3 H), 3.39 (s, 2 H), 2.65 (s, 3 H), 1.81 (s, 3 H); \( ^{13} \)C NMR (175 MHz CDCl\(_3\)) \( \delta \) 155.1, 143.1, 141.2, 140.2, 136.2, 124.5, 122.0, 120.7, 118.9, 114.8, 113.0, 112.7, 63.2, 55.7, 40.8, 20.7; IR (film) 3355, 2933, 1510 cm\(^{-1}\); MS (ESI+) 283.1807 (283.1805 calcd for C\(_{18}\)H\(_{20}\)N\(_2\)O, M + H\(^+\)).
**N^1-[(Benzyloxy)methyl]allyl]-N^2-(4-methoxyphenyl)-N^1'-methylbenzene-1,2-diamine (3b):** General Procedure C was employed for the coupling of 4-bromoanisole (0.165 mL, 1.32 mmol) and N^1-[(benzyloxy)methyl]allyl]-N^1'-methylbenzene-1,2-diamine (372 mg, 1.32 mmol), except using a catalyst composed of Pd_2(dba)_3 (26 mg, 0.0264 mmol, 2.0 mol %) and JohnPhos (16 mg, 0.528 mmol, 4.0 mol %) and a reaction temperature of 85 °C. This procedure afforded 380 mg (75%) of the title compound as a red oil. \(^1\)H NMR (700 MHz, CDCl_3) δ 7.32–7.26 (m, 4 H), 7.13 (d, \(J = 7.7\) Hz, 1 H), 7.09 (d, \(J = 8.0\) Hz, 1 H), 7.06 (d, \(J = 8.7\) Hz, 2 H), 6.96 (t, \(J = 7.8\) Hz, 1 H), 6.84 (d, \(J = 8.9\) Hz, 2 H), 6.78 (t, \(J = 7.7\) Hz, 1 H), 6.73 (s, br, 1 H), 5.29 (s, 1 H), 4.44 (s, 2 H), 4.09 (s, 2 H), 3.80 (s, 3 H), 3.53 (s, 2 H), 2.65 (s, 3 H); \(^13\)C NMR (175 MHz CDCl_3) δ 155.0, 143.2, 140.4, 140.2, 138.1, 135.9, 129.4, 127.8, 127.6, 124.4, 122.5, 120.5, 118.4, 115.5, 114.5, 112.7, 71.7, 71.4, 59.4, 55.6, 40.7; IR (film) 3338, 2946, 1509 cm \(^{-1}\); MS (ESI+) 389.2226 (389.2224 calcd for C_{25}H_{28}N_{2}O_{2}, M + H\(^+\)).

**N^1-(4-Methoxyphenyl)-N^2-methyl-N^2-(2-phenylallyl)benzene-1,2-diamine (3c).** General Procedure C was employed for the coupling of 4-bromoanisole (0.41 mL, 3.3 mmol) and N^1-methyl-N^1-(2-phenylallyl)benzene-1,2-diamine (790 mg, 3.3 mmol), except using a catalyst composed of Pd_2(dba)_3 (60 mg, 0.066 mmol, 2.0 mol %) and JohnPhos (40 mg, 0.132 mmol, 4.0 mol %) and a reaction temperature of 85 °C. This procedure afforded 750 mg (66%) of the title compound as a red oil. \(^1\)H NMR (400 MHz, CDCl_3) δ 7.57–7.52 (m, 2 H), 7.46–7.40 (m, 3 H), 7.30 (d, \(J = 7.8\) Hz, 1 H), 7.24 (d, \(J =
8.0 Hz, 1 H), 7.12 (t, J = 7.4 Hz, 1 H), 7.03 (d, J = 8.8 Hz, 2 H), 6.98–6.89 (m, 3 H), 6.41 (s, br, 1 H) 5.56 (s, 1 H), 5.48 (s, 1 H), 4.03 (s, 2 H), 3.89 (s, 3 H), 2.73 (s, 3 H); <sup>13</sup>C NMR (100 MHz CDCl<sub>3</sub>) δ 154.8, 145.8, 140.5, 140.4, 139.9, 135.6, 128.3, 127.5, 126.5, 124.8, 121.8, 121.3, 118.4, 115.2, 114.4, 112.2, 61.0, 55.4, 41.0; IR (film) 3347, 2947, 1510 cm<sup>-1</sup>; MS (ESI+) 239.1537 (239.1543 calcd for C<sub>23</sub>H<sub>24</sub>N<sub>2</sub>O, M + H<sup>+</sup>).

**Methyl (2-allylbenzyl)carbamate (5).** A flame dried round bottom flask equipped with a stir bar was cooled under a stream of nitrogen and charged with ether (20 mL) and LiAlH<sub>4</sub> (39 mL, 39 mmol, 1.0 M in ether). The mixture was cooled to 0 °C, stirred for five min, then a solution of 2-allylbenzonitrile<sup>1</sup> (2.80 g, 19.5 mmol) in ether (15 mL) was added slowly dropwise. The reaction mixture was stirred for 1.5 h at 0 °C and then was slowly quenched with 1.5 mL H<sub>2</sub>O, 1.5 mL 15% NaOH and 3.0 mL H<sub>2</sub>O. The resulting mixture was stirred at rt for 20 min, then the salts were filtered off through a fritted funnel. The filtrate was dried over anhydrous MgSO<sub>4</sub> and concentrated in vacuo to afford 2-allylbenzylamine, which was used without further purification.

The crude 2-allylbenzylamine product from above was dissolved in dichloromethane (60 mL) and added to a flame dried round bottom flask equipped with a stir bar. Solid K<sub>2</sub>CO<sub>3</sub> (2.95 g, 21.3 mmol) was added to the flask and the resulting mixture was cooled to 0 °C. Methyl chloroformate (1.0 equiv., 1.5 mL) was then slowly added, and the resulting mixture was warmed to room temperature and stirred for 16 h. The reaction mixture was filtered, and the filtrate was concentrated in vacuo. The crude product was purified by flash chromatography on silica gel using hexanes/ Et<sub>2</sub>O as the eluent to afford 3.18 g (79%) as a clear oil. <sup>1</sup>H NMR (500 MHz, d<sub>8</sub>-toluene, 90 °C) δ 7.07 (d, J = 6.6 Hz, 1 H), 7.02–6.93 (m, 3 H), 5.82–7.72 (m, 1 H), 4.91 (d, J = 10.0 Hz, 1 H); 4.85 (d, J = 16.9 Hz, 1 H), 4.58 (s, br, 1 H), 4.16 (d, J = 5.9 Hz, 2 H), 3.42 (s, 3 H), 3.18 (d, J = 6.1 Hz, 2 H); <sup>13</sup>C NMR (125 MHz, d<sub>8</sub>-toluene, 90 °C) δ 157.7, 139.2, 138.5, 138.2, 131.2, 130.0, 519.
General Procedure D: Asymmetric Pd-catalyzed carboamination reactions. A flame dried Schlenk flask equipped with a stir bar was cooled under a stream of nitrogen and charged with Pd$_2$(dba)$_3$ (2 mol %), (S)-Siphos-PE (6 mol %), the aryl or alkenyl halide (1.0–2.0 equiv.), NaO$\text{t}$Bu (1.3–2.0 equiv.), and the amino alkene substrate. The flask was purged with nitrogen, and toluene (0.1 M) was added (xylenes was used as solvent in cases where reactions were heated over 110 °C). The resulting mixture was heated to 80–125 °C with stirring for 2–15 hrs. The reaction mixture was then cooled to rt, saturated aqueous ammonium chloride (6 mL/mmol) was added, and the mixture was transferred to a separatory funnel. The mixture was extracted with ethyl acetate and the combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated in vacuo. The crude product was then purified by flash chromatography on silica gel using hexanes/Et$_2$O as the eluant.

(R)-(+)−2-[4-(tert-Butyl)benzyl]-1-(4-methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinoline (2b). General Procedure D was employed for the coupling of N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (27 mg, 0.10 mmol) and 1-bromo-4-tert-butylbenzene (43 mg, 0.20 mmol) using NaO$\text{t}$Bu (19 mg, 0.20 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 35 mg (86%) of the title compound as a white solid, mp 47–50 °C. This material was judged to be 92:8 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 0.5% IPA/Hexanes, 1 mL/min, $\lambda$ 254 nm, RT= 6.1 and 7.3 min). $[\alpha]^{23}_D$ $+50.6$ (c 3.33, CH$_2$Cl$_2$); $^1$H NMR (500
(R)-(+)\-4-\{2-[4-(tert-Butyl)benzyl]-2-methyl-3,4-dihydroquinolin-1(2H)-yl\}benzonitrile (2c). General Procedure D was employed for the coupling of 4-\{2-(3-methylbut-3-en-1-yl)phenyl\}amino)benzonitrile (26 mg, 0.10 mmol,) and 1-bromo-4-tert-butylbenzene (43 mg, 0.20 mmol) using NaO\textsubscript{t}Bu (19 mg, 0.20 mmol) as the base and a reaction temperature 110 °C for 14 h. This procedure afforded 20 mg (51%) of the title compound as an orange oil. This material was judged to be 62:38 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 254 nm, RT= 5.6 and 8.0 min). \([\alpha]^{23}_D+50.3 (c 0.68, \text{CH}_2\text{Cl}_2)\); \(^1\)H NMR (700 MHz, CDCl\textsubscript{3}) \(\delta\) 7.52 (d, \(J = 8.7\) Hz, 2 H), 7.28 (d, \(J = 8.2\) Hz, 2 H), 7.11 (d, \(J = 7.5\) Hz, 1 H), 7.02 (d, \(J = 8.2\) Hz, 2 H), 6.91 (t, \(J = 8.0\) Hz, 1 H), 6.88–6.78 (m, 2 H), 6.73 (t, \(J = 7.3\) Hz, 1 H), 6.15 (d, \(J = 8.2\) Hz, 1 H), 3.09 (ddd, \(J = 6.3, 11.2, 17.3\) Hz, 1 H), 2.93–2.86 (m, 2 H), 2.66 (d, \(J = 13.1\) Hz, 1 H), 1.95 (ddd, \(J = 4.2, 6.3, 13.3\) Hz, 1 H), 1.84 (ddd, \(J = 6.0, 11.2, 13.2\) Hz, 1 H), 1.31 (s, 9 H), 1.05 (s, 3 H); \(^{13}\)C NMR (175 MHz, CDCl\textsubscript{3}) \(\delta\) 150.0, 149.7, 145.0, 134.8, 133.1, 132.3, 130.7, 129.7, 126.8, 125.1, 123.1, 119.1, 118.6, 118.5, 109.0, 58.2,
43.4, 34.6, 33.7, 31.6, 26.7, 24.2; IR (film) 2962, 1596, 1500 cm⁻¹; MS (ESI+) 395.2472 (395.2482 calcd for C₂₈H₃₀N₂, M + H⁺).

(R)-(+)/2-[4-(tert-Butyl)benzyl]-1-[4-(tert-butyl)phenyl]-2-methyl-1,2,3,4-tetrahydroquinoline (2d). General Procedure D was employed for the coupling of N-[4-(tert-butyl)phenyl]-2-(3-methylbut-3-en-1-yl)aniline (29 mg, 0.10 mmol) and 1-bromo-4-tert-butylbenzene (43 mg, 0.20 mmol) using NaO'Bu (19 mg, 0.20 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 40 mg (93%) of the title compound as a pale yellow solid, mp 59–63 °C. This material was judged to be 87:13 er by chiral HPLC analysis (LuxAmylose, 25 cm x 4.6 mm, 1% IPA/Hexanes, 0.3 mL/min, λ 254nm, RT= 12.0 and 12.5 min). [α]²³D +40.3 (c 2.30, CH₂Cl₂); ¹H NMR (500 MHz, CDCl₃) δ 7.40 (d, J = 8.1 Hz, 2 H), 7.29 (d, J = 8.3 Hz, 2 H), 7.12–6.98 (m, 5 H), 6.87 (t, J = 7.3 Hz, 1 H), 6.62 (t, J = 7.1 Hz, 1 H), 6.04 (d, J = 8.3 Hz, 1 H), 3.12 (ddt, J = 5.6, 11.1, 15.5 Hz, 1 H), 2.94–2.81 (m, 3 H), 1.99–1.92 (m, 1 H), 1.80 (dd, J = 5.7, 9.6, 13.2 Hz, 1 H), 1.39 (s, 9 H), 1.33 (s, 9 H), 1.11 (s, 3 H); ¹³C NMR (125 MHz, CDCl₃) δ 149.7, 149.1, 146.4, 140.9, 135.4, 132.0, 130.7, 129.3, 126.6, 126.4, 124.9, 121.0, 116.2, 115.5, 57.5, 44.8, 34.8, 34.6, 32.5, 31.7, 31.6, 26.1, 24.7; IR (film) 2962, 1600, and 1507 cm⁻¹; MS (ESI+) 426.3174 (426.3155 calcd for C₃₁H₃₉N, M + H⁺).
(R)-(+)-4-{2-[4-(tert-Butyl)benzyl]-2-methyl-3,4-dihydroquinolin-1(2H)-yl}-N,N-dimethylaniline (2e). General Procedure D was employed for the coupling of N1,N1-dimethyl-N4-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 1-bromo-4-tert-butylbenzene (85 mg, 0.40 mmol), using NaO\textsuperscript{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 80 mg (95%) of the title compound as a white solid, mp 144–147 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1.2% IPA/Hexanes, 2 mL/min, λ 254 nm, RT = 3.6 and 5.0 min). [α]\textsuperscript{23}\text{D} +31.8 (c 1.75, CH\textsubscript{2}Cl\textsubscript{2}); \textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) δ 7.33 (d, J = 8.1 Hz, 2 H), 7.15–7.01 (m, 5 H), 6.90 (t, J = 7.1 Hz, 1 H), 6.85–6.78 (m, 2 H), 6.63 (t, J = 7.1 Hz, 1 H), 6.12 (d, J = 8.3 Hz, 1 H), 3.20–3.09 (m, 1 H), 3.08–2.94 (m, 7 H), 2.92–2.83 (m, 2 H), 1.98 (m, 1 H), 1.82 (ddd, J = 5.3, 9.6, 13.0 Hz, 1 H), 1.36 (s, 9 H), 1.15 (s, 3 H); \textsuperscript{13}C NMR (125 MHz CDCl\textsubscript{3}) δ 149.3, 149.0, 147.0, 135.5, 133.2, 132.3, 130.6, 129.1, 126.6, 124.9, 120.8, 115.7, 115.0, 113.4, 113.1, 57.6, 44.9, 40.8, 34.5, 32.4, 31.6, 26.0, 24.7 (an extra peak at 113.1 is present due to apparent slow bond rotation); IR (film) 2961, 1609, 1516 cm\textsuperscript{-1}; MS (ESI+) 413.2955 (413.2951 calcd for C\textsubscript{29}H\textsubscript{36}N\textsubscript{2}, M + H\textsuperscript{+}).
(R)-(+)-N,N-Dimethyl-4-[2-methyl-2-(4-morpholinobenzyl)-3,4-dihydroquinolin-1(2H)-yl]aniline (2f). General Procedure D was employed for the coupling of \(N^1,N^1\)-dimethyl-\(N^4\)-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-(4-bromophenyl)morpholine (97 mg, 0.40 mmol) using NaO\(\text{tBu}\) (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 77 mg (87%) of the title compound as a yellow solid, mp 158–161 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 2 mL/min, \(\lambda\) 254 nm, RT = 14.6 and 20.1 min). \([\alpha]_{D}^{23}\) +24.7 (c 1.10, CH\(_2\)Cl\(_2\)); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.13–7.03 (m, 5 H), 6.92–6.77 (m, 5 H), 6.61 (t, \(J = 7.2\) Hz, 1 H), 6.10 (d, \(J = 8.3\) Hz, 1 H), 3.92–3.86 (m, 4 H), 3.20–3.14 (m, 4 H), 3.10–3.04 (m, 1 H), 3.02 (s, 6 H), 2.92–2.79 (m, 3 H), 1.95 (m, 1 H), 1.78 (ddd, \(J = 5.5, 9.5, 13.2\) Hz, 1 H), 1.12 (s, 3 H); \(^1^3\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 149.7, 149.1, 147.0, 133.2, 133.1, 132.2, 131.6, 130.1, 129.1, 126.6, 120.8, 115.7, 115.3, 115.0, 113.3, 113.0, 67.1, 57.6, 49.6, 44.5, 40.8, 32.3, 25.9, 24.7 (extra peaks at 133.1 and 113.0 are present due to apparent slow bond rotation); IR (film) 2963, 1609, 1514 cm\(^{-1}\); MS (ESI+) 442.285 (442.28 calcd for C\(_{29}\)H\(_{35}\)N\(_3\)O, M + H\(^{+}\)).
(R)-(+)-4-[2-(4-Methoxybenzyl)-2-methyl-3,4-dihydroquinolin-1(2H)-yl]-N,N-dimethylaniline (2g). General Procedure D was employed for the coupling of \(N_1,N_1\)-dimethyl-\(N_4\)-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-bromoanisole (75 mg, 0.40 mmol), using NaO\(\text{tBu}\) (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 68 mg (88%) of the title compound as a yellow solid, mp 101–105 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, \(\lambda\) 254 nm, RT= 10.2 and 15.1 min). \([\alpha]^{23}_D\) +30.6 (c 2.10, CH\(_2\)Cl\(_2\)); \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.13–7.01 (m, 5 H), 6.90 (t, \(J = 7.3\) Hz, 1 H), 6.85–6.77 (m, 4 H), 6.62 (t, \(J = 7.4\) Hz, 1 H), 6.10 (d, \(J = 8.1\) Hz, 1 H), 3.82 (s, 3 H), 3.14–3.05 (m, 1 H), 3.02 (s, 6 H), 2.92–2.80 (m, 3 H), 1.94 (dt, \(J = 6.0, 12.5\) Hz, 1 H), 1.79 (ddd, \(J = 5.5, 9.4, 13.3\) Hz, 1 H), 1.11 (s, 3 H); \(^{13}\)C NMR (125 MHz CDCl\(_3\)) \(\delta\) 158.1, 149.2, 146.9, 133.2, 133.0, 132.3, 131.8, 130.7, 129.1, 126.6, 120.8, 115.8, 115.1, 113.4, 113.3, 113.0, 57.5, 55.3, 44.5, 40.8, 32.3, 25.8, 24.6 (extra peaks at 133.0 and 113.0 are present due to apparent slow bond rotation); IR (film) 2962, 1609, 1512 cm\(^{-1}\); MS (ESI+) 387.2432 (387.2431 calcd for C\(_{26}\)H\(_{30}\)N\(_2\)O, M + H\(^+\)).
(R)-(+)\text{-}N,N\text{-}Dimethyl-4\text{-}[2\text{-}methyl\text{-}2\text{-}[4\text{-}(trifluoromethyl)benzyl\text{-}3,4\text{-}dihydroquinolin-1(2H)\text{-}yl\text{]}aniline (2h). General Procedure D was employed for the coupling of \(N^1,N^1\)-dimethyl-\(N^4\)-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-bromobenzotrifluoride (90.0 mg, 0.40 mmol), using NaO\text{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 75.6 mg (89\%) of the title compound as a white solid, mp 127-130 °C. This material was judged to be 94:6 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1\% IPA/Hexanes, 1 mL/min, \(\lambda\) 254 nm, RT= 6.1 and 8.9 min). \([\alpha]_{23}^{23}\) +31.5 (c 1.43, CH\(_2\)Cl\(_2\)); \(^1\text{H} NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.56 (d, \(J = 8.0 \text{ Hz, 2 H}\)), 7.30 (d, \(J = 8.0 \text{ Hz, 2 H}\)), 7.14–6.97 (m, 3 H), 6.91 (t, \(J = 7.7 \text{ Hz, 1 H}\)), 6.86–6.76 (m, 2 H), 6.65 (t, \(J = 7.1 \text{ Hz, 1 H}\)), 6.14 (d, \(J = 8.2 \text{ Hz, 1 H}\)), 3.15–2.85 (m, 10 H), 1.95 (dt, \(J = 6.0, 12.4 \text{ Hz, 1 H}\)), 1.79 (ddd, \(J = 5.6, 9.5, 13.1 \text{ Hz, 1 H}\)), 1.13 (s, 3 H); \(^{13}\text{C} NMR (125 MHz CDCl\(_3\)) \(\delta\) 149.4, 146.8, 143.0, 133.0, 132.2, 131.2, 129.2, 128.6 (q, \(J = 32 \text{ Hz}\)), 126.8, 124.6 (q, \(J = 270 \text{ Hz}\)), 124.9 (q, \(J = 3 \text{ Hz}\)), 120.8, 116.2, 115.6, 113.4, 113.0, 57.3, 45.2, 40.6, 32.4, 25.7, 24.4 (an extra peak at 113.0 is present due to apparent slow bond rotation); IR (film) 2971, 1610, 1517 cm\(^{-1}\); MS (ESI+) 425.2202 (425.2199 calcd for C\(_{26}\)H\(_{27}\)F\(_3\)N\(_2\), M + H\(^+\)).
(R)-(+)\,-N,N-Dimethyl-4-{2-methyl-2-[3-(trifluoromethyl)benzyl]-3,4-dihydroquinolin-1(2H)-yl}aniline (2i). General Procedure D was employed for the coupling of \(N^1,N^1\)-dimethyl-\(N^4\)-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 3-bromobenzotrifluoride (90 mg, 0.40 mmol) using NaO\(\text{tBu}\) (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 80 mg (94%) of the title compound as a brown solid, mp 91–94 °C. This material was judged to be 92:8 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 1 % IPA/Hexanes, 1 mL/min, \(\lambda\) 254 nm, RT= 4.2 and 5.4 min). [\(\alpha\)]\textsuperscript{23}D \, +22.5 (c 1.35, CH\textsubscript{2}Cl\textsubscript{2}); \(^1\)H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 7.52 (d, \(J\) = 7.3 Hz, 1 H), 7.46–7.35 (m, 3 H), 7.12–6.93 (m, 3 H), 6.91 (t, \(J\) = 7.5 Hz, 1 H), 6.85–6.75 (m, 2 H), 6.65 (t, \(J\) = 7.3 Hz, 1 H), 6.15 (d, \(J\) = 8.3 Hz, 1 H), 3.15–3.02 (m, 7 H), 3.00–2.97 (m, 2 H), 3.93–3.85 (m, 1 H), 1.95 (dt, \(J\) = 5.9, 13.9 Hz, 1 H), 1.82 (ddd, \(J\) = 5.50, 9.40, 13.30 Hz, 1 H), 1.12 (s, 3 H); \(^{13}\)C NMR (125 MHz, CDCl\textsubscript{3}) \(\delta\) 149.4, 146.8, 139.7, 134.3, 133.4, 133.0, 132.9, 132.3, 130.4 (q, \(J\) = 31 Hz), 129.2, 128.5, 127.5 (q, \(J\) = 4 Hz), 126.8, 124.4 (q, \(J\) = 270 Hz), 123.2 (q, \(J\) = 4 Hz), 120.8, 116.3, 115.7, 113.4, 112.9, 57.3, 45.3, 40.8, 32.6, 25.8, 24.6 (extra peaks at 132.9 and 112.9 are present due to apparent slow bond rotation); IR (film) 2971, 1609, 1518 cm\textsuperscript{-1}; MS (ESI+) 425.22 (425.2199 calcd for C\textsubscript{26}H\textsubscript{27}F\textsubscript{3}N\textsubscript{2}, M + H\textsuperscript{+}).
(R)-(+)-[4-([1-[4-((Dimethylamino)phenyl]-2-methyl-1,2,3,4-tetrahydroquinolin-2-yl]methyl)phenyl)(phenyl)methanone (2j). General Procedure D was employed for the coupling of \(N^1,N^1\)-dimethyl-\(N^4\)-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 4-bromobenzophenone (104 mg, 0.40 mmol) using NaO\textsubscript{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 76 mg (82%, 93% pure) of the title compound as a yellow solid, mp 156–159 °C. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 25 cm x 4.6 mm, 5 % IPA/Hexanes, 1 mL/min, \(\lambda\) 254 nm, RT= 11.6 and 25.8 min). \([\alpha]^{23}_{25} +32.7 (c 1.10, CH\textsubscript{2}Cl\textsubscript{2}); \) \(^1\)H NMR (700 MHz, CDCl\textsubscript{3}) \(\delta\) 7.83 (d, \(J\) = 7.5 Hz, 2 H), 7.76 (d, \(J\) = 8.0 Hz, 2 H), 7.61 (t, \(J\) = 7.4 Hz, 1 H), 7.51 (t, \(J\) = 7.6 Hz, 2 H), 7.30 (d, \(J\) = 8.0 Hz, 2 H), 7.10–6.98 (m, 3 H), 6.91 (t, \(J\) = 7.7 Hz, 1 H), 6.79 (dd, \(J\) = 8.5, 19.0 Hz, 2 H), 6.63 (t, \(J\) = 7.1 Hz, 1 H), 6.13 (d, \(J\) = 8.3 Hz, 1 H), 3.15–3.08 (m, 1 H), 3.06–3.00 (m, 7 H), 2.99 (d, \(J\) = 12.8 Hz, 1 H), 2.89 (dt, \(J\) = 5.9, 16.6 Hz, 1 H), 1.98 (dt, \(J\) = 5.9, 12.4 Hz, 1 H), 1.83 (ddd, \(J\) = 5.5, 9.6, 13.2 Hz, 1 H), 1.15 (s, 3 H); \(^{13}\)C NMR (175 MHz, CDCl\textsubscript{3}) \(\delta\) 196.4, 149.2, 146.6, 143.8, 137.8, 135.4, 132.8, 132.2, 132.0, 130.7, 130.0, 129.8, 129.0, 128.2, 126.6, 120.6, 116.0, 115.3, 113.2, 112.8, 57.4, 45.3, 40.6, 32.5, 25.9, 24.4 (2 extra peaks in the arene region are present due to apparent slow bond rotation); IR (film) 2927, 1656, 1517 cm\(^{-1}\); MS (ESI+) 461.2583 (461.2587 calcd for C\textsubscript{32}H\textsubscript{32}N\textsubscript{2}O, M + H\textsuperscript{+}).
(R)-(+)-N,N-Dimethyl-4-[2-methyl-2-(3-methylbut-2-en-1-yl)-3,4-dihydroquinolin-1(2H)-yl]aniline (2k). General Procedure D was employed for the coupling of $N^1,N^1$-dimethyl-$N^4$-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 2-bromopropene (48 mg 0.40 mmol) using NaO$\text{t}$$\text{Bu}$ (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylenes) for 14 h. This procedure afforded 55 mg (86%) of the title compound as an off-white solid, mp 76–79 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ODH, 25 cm x 4.6 mm, 1% IPA/Hexanes, 0.2 mL/min, λ 254nm, RT = 13.4 and 15.2 min). $[\alpha]^{23}_D$ +37.7 (c 1.81, CH$_2$Cl$_2$); $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.12–7.00 (m, 3 H), 6.85 (t, $J$ = 7.1 Hz, 1 H), 6.79 (d, $J$ = 8.8 Hz, 2 H), 6.58 (t, $J$ = 7.3 Hz, 1 H), 6.04 (d, $J$ = 8.3 Hz, 1 H), 4.92 (s, 1 H), 4.78 (s, 1 H), 3.06–2.98 (m, 7H), 2.85 (dt, $J$ = 5.9, 16.5 Hz, 1 H), 2.50 (d, $J$ = 13.1 Hz, 1 H), 2.29 (d, $J$ = 13.0 Hz, 1 H), 2.07 (dt, $J$ = 5.9, 13.1 Hz, 1 H), 1.93 (ddd, $J$ = 5.5, 9.6, 13.2 Hz, 1 H), 1.80 (s, 3 H), 1.21 (s, 3 H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 149.3, 146.8, 142.7, 133.1, 132.3, 129.1, 126.6, 120.8, 115.7, 115.5, 114.9, 113.3, 113.0, 57.2, 46.9, 40.8, 33.0, 27.1, 25.5, 24.7 (an extra peak at 113.0 is present due to apparent slow bond rotation); IR (film) 2969, 1609, 1517 cm$^{-1}$; MS (ESI+) 321.2329 (321.2325 calcd for C$_{22}$H$_{28}$N$_2$, M + H$^+$).

(R)-(+)-N,N-Dimethyl-4-[2-methyl-2-(3-methylbut-2-en-1-yl)-3,4-dihydroquinolin-1(2H)-yl]aniline (2l). General Procedure D was employed for the coupling of $N^1,N^1$-
dimethyl-$N^d$-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and 1-bromo-2-methyl-1-propene (54 mg, 0.40 mmol) using NaO'Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylanes) for 14 h. This procedure afforded 66 mg (98%) of the title compound as an orange oil. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 0.9% IPA/Hexanes, 0.1 mL/min, λ 254 nm, RT = 26.1 and 28.2 min). \([\alpha]^{23}_{D} +53.4\) (c 2.35 CH$_2$Cl$_2$); $^1$H NMR (500 MHz, CDCl$_3$) δ 7.12–6.96 (m, 3 H), 6.83 (t, $J$ = 7.1 Hz, 1 H), 6.77 (d, $J$ = 8.3 Hz, 2 H), 6.55 (t, $J$ = 7.1 Hz, 1 H), 6.02 (d, $J$ = 8.3 Hz, 1 H), 5.19 (t, $J$ = 7.3 Hz, 1 H), 3.01 (s, 3 H), 2.88–2.82 (m, 1 H), 2.32 (dd, $J$ = 7.2, 14.4 Hz, 1 H), 2.20 (dd, $J$ = 7.5, 14.4 Hz, 1 H), 2.00 (dt, $J$ = 6.2, 12.9 Hz, 1 H), 1.79 (ddd, $J$ = 5.9, 8.1, 13.5 Hz, 1 H), 1.72 (s, 3 H), 1.56 (s, 3 H), 1.22 (s, 3 H); $^{13}$C NMR (125 MHz, CDCl$_3$) δ 149.3, 147.2, 133.6, 133.0, 132.3, 129.1, 126.6, 121.0, 120.3, 115.5, 114.8, 113.3, 57.6, 40.8, 37.9, 32.8, 26.3, 25.9, 24.7, 18.3; IR (film) 2967, 1608, 1516 cm$^{-1}$; MS (ESI+) 335.2136 (335.2482 calcd for C$_{23}$H$_{30}$N$_2$, M + H$^+$).

(Z,R)-(+-)$N,N$-Dimethyl-$4$-[2-methyl-2-(pent-2-en-1-yl)-3,4-dihydroquinolin-1(2H)-yl]aniline (2m). General Procedure D was employed for the coupling of $N^i,N^i$-dimethyl-$N^d$-[2-(3-methylbut-3-en-1-yl)phenyl]benzene-1,4-diamine (56 mg, 0.20 mmol) and (Z)-1-bromo-1-butene (54 mg, 0.40 mmol) using NaO'Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 125 °C (xylanes) for 14 h. This procedure afforded 61 mg (91%) of the title compound as an off-white solid, mp 122–125 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 0.8% IPA/Hexanes, 0.1 mL/min, λ 254 nm, RT = 30.0 and 33.3 min). \([\alpha]^{23}_{D} +59.3\) (c 1.50, CH$_2$Cl$_2$); $^1$H NMR (500 MHz, CDCl$_3$) δ 7.16–6.93 (m, 3 H), 6.88 (t, $J$ = 7.3 Hz, 1 H), 6.81 (d, $J$ = 8.8 Hz, 2 H), 6.59 (t, $J$ = 7.1 Hz, 1 H), 6.09 (d, $J$ = 8.3 Hz, 1 H), 5.55–5.38 (m, 2
(R)-(+)-4-(4-{[1-(4-Methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinolin-2-yl]methyl}phenyl)morpholine (2n). General Procedure D was employed for the coupling of N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 4-(4-bromophenyl)morpholine (97 mg, 0.40 mmol) using NaO\textsubscript{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 70 mg (82%) of the title compound as a white solid, mp 62–65 °C. This material was judged to be 94:6 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 8.8 and 11.1 min). \([\alpha]^{23}_D +56.1 (c 1.11, \text{CH}_2\text{Cl}_2); ^1\text{H NMR} (700 \text{ MHz, CDCl}_3) \delta 7.14–7.05 (m, 5 H), 7.02–6.95 (m, 2 H), 6.88 (t, \(J = 7.2\) Hz, 1 H), 6.84 (d, \(J = 8.3\) Hz, 2 H), 6.63 (t, \(J = 7.2\) Hz, 1 H), 6.05 (d, \(J = 8.4\) Hz, 1 H), 3.92–3.84 (m, 7 H), 3.18–3.12 (m, 4H), 3.09 (ddd, \(J = 5.7, 9.6, 15.9\) Hz, 1 H), 2.89–2.81 (m, 2 H), 2.78 (d, \(J = 13.2\) Hz, 1 H), 1.94 (dt, \(J = 6.0, 12.5\) Hz, 1 H), 1.78 (ddd, \(J = 5.5, 9.6, 13.2\) Hz, 1 H), 1.09 (s, 3 H); \(^{13}\text{C NMR} (175 \text{ MHz, CDCl}_3) \delta 158.2, 149.7, 146.6, 136.2, 133.6, 133.5, 131.6, 129.8, 129.2, 126.6, 121.0, 116.1, 115.3, 115.2, 114.9, 114.5, 67.1, 57.5, 55.5, 49.5, 44.4, 32.3, 25.8, 24.6 (extra peaks at 133.5 and 114.5 are present due to apparent slow bond rotation); IR (film) 2928, 1606, 1505 cm\(^{-1}\); MS (ESI+) 429.2521 (429.2537 calcd for C\(_{28}\)H\(_{32}\)N\(_2\)O\(_2\), M + H\(^+\)).
(R)-(+)2-(4-Chlorobenzyl)-1-(4-methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinoline (2o). General Procedure D was employed for the coupling of N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 4-bromochlorobenzene (77 mg, 0.40 mmol) using NaO\textsuperscript{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 62 mg (82%) of the title compound as a viscous oil. This material was judged to be 89:11 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 0.4% IPA/Hexanes, 1.1 mL/min, λ 254 nm, RT = 8.9 and 10.5 min). [\alpha]\textsuperscript{23}D +56.2 (c 0.97, CH\textsubscript{2}Cl\textsubscript{2}); \textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 7.25 (d, \(J\) = 8.6 Hz, 2 H), 7.15–6.91 (m, 7 H), 6.89 (t, \(J\) = 7.3 Hz, 1 H), 6.65 (t, \(J\) = 7.3 Hz, 1 H), 6.08 (d, \(J\) = 8.3 Hz, 1 H), 3.87 (s, 3 H), 3.04 (ddd, \(J\) = 5.8, 9.6, 15.9 Hz, 1 H), 2.87–2.79 (m, 3 H), 1.90 (dt, \(J\) = 5.9, 13.4 Hz, 1 H), 1.77 (ddddd, \(J\) = 5.6, 9.6, 13.3 Hz, 1 H), 1.08 (s, 3 H); \textsuperscript{13}C NMR (125 MHz, CDCl\textsubscript{3}) \(\delta\) 158.3, 146.4, 136.9, 136.3, 133.4, 132.3, 132.2, 129.3, 128.2, 126.7, 121.0, 116.5, 115.7, 115.1, 114.5, 57.3, 55.6, 44.6, 32.5, 25.8, 24.5 (an extra peak at 114.5 is present due to apparent slow bond rotation); IR (film) 2928, 1604, 1505 cm\textsuperscript{-1}; MS (ESI+) 378.1620 (378.1619 calcd for C\textsubscript{24}H\textsubscript{24}ClNO, M + H\textsuperscript{+}).

(R)-(+)2-(4-[[1-(4-Methoxyphenyl)-2-methyl-1,2,3,4-tetrahydroquinolin-2-yl]methyl]phenyl)(phenyl)methanone (2p). General Procedure D was employed for the coupling of N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20
mmol) and 4-bromobenzophenone (104 mg, 0.40 mmol) using NaO\text{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 74 mg (83%) of the title compound as a white solid, mp 58–61 °C. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 8% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 10.4 and 16.5 min). [\alpha]_{23}^{20} +44.7 (c 0.90, CH₂Cl₂); ¹H NMR (500 MHz, CDCl₃) δ 7.83 (d, J = 7.1 Hz, 2 H), 7.76 (d, J = 8.3 Hz, 2 H), 7.60 (t, J = 7.3 Hz, 1 H), 7.49 (t, J = 7.8 Hz, 2 H), 7.28 (d, J = 8.1 Hz, 2 H), 7.15–6.91 (m, 5 H), 6.89 (t, J = 7.1 Hz, 1 H), 6.65 (t, J = 6.4 Hz, 1 H), 6.08 (d, J = 8.1 Hz, 1 H), 3.87 (s, 3 H), 3.10 (ddd, J = 5.8, 9.7, 16.1 Hz, 1 H), 3.02–2.93 (m, 2 H), 2.87 (dt, J = 5.8, 16.7 Hz, 1 H), 1.95 (dt, J = 5.9, 13.2 Hz, 1 H), 1.82 (ddd, J = 5.6, 9.7, 13.2 Hz, 1 H), 1.13 (s, 3 H); ¹³C NMR (125 MHz, CDCl₃) δ 196.5, 158.3, 146.4, 143.7, 137.9, 136.3, 135.7, 133.4, 132.4, 130.8, 130.1, 130.0, 129.3, 128.4, 126.8, 121.0, 116.6, 115.8, 115.1, 114.5, 57.5, 55.5, 45.3, 32.6, 26.1, 24.5 (an extra peak at 114.5 is present due to apparent slow bond rotation); IR (film) 2928, 1656, 1603, 1505 cm⁻¹; MS (ESI+) 448.2269 (448.2271 calcd for C₃₁H₂₉NO₂, M + H⁺).

(R)-(+)1-(4-Methoxyphenyl)-2-methyl-2-(3-methylbut-2-en-1-yl)-1,2,3,4-tetrahydroquinoline (2q). General Procedure D was employed for the coupling of N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 1-Bromo-2-methyl-1-propene (41 µL, 0.40 mmol) using NaO\text{t}Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 63 mg (98%) of the title compound as an orange oil. This material was judged to be 95:5 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 8% IPA/Hexanes, 0.2 mL/min, λ 254 nm, RT = 22.3 and 24.4 min). [\alpha]_{23}^{20} +58.0 (c 1.11, CH₂Cl₂); ¹H NMR (500 MHz, CDCl₃) δ 7.23–7.08 (m, 2 H), 7.07 (d, J = 7.3 Hz, 1 H), 6.99 (d, J = 9.1 Hz, 2 H), 6.87 (t, J = 7.3 Hz, 1 H), 6.61 (t, J = 7.4 Hz, 1 H), 6.02 (d, J = 8.3 Hz, 1 H), 5.23 (app. t, J = 7.1 Hz, 1 H), 3.88 (s, 3 H), 2.95–2.85 (m, 2 H), 2.34 (dd, J = 7.2, 14.3 Hz, 1 H), 2.22 (dd, J = 7.5,
14.3 Hz, 1 H), 2.05 (dt, $J = 6.4, 13.0$ Hz, 1 H), 1.89–1.81 (m, 1 H), 1.77 (s, 3 H), 1.60 (s, 3H), 1.13 (s, 3H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 158.3, 146.8, 136.2, 133.8, 133.4, 129.2, 126.6, 121.1, 120.0, 115.9, 114.9, 114.7, 57.5, 55.5, 37.9, 32.8, 26.3, 25.9, 24.6, 18.3; IR (film) 2926, 1599, 1507 cm$^{-1}$; MS (ESI+) 322.2170 (322.2165 calcd for C$_{22}$H$_{27}$NO, M$^+$).

**$^{(*)}$-(+)-1-(4-Methoxyphenyl)-2-methyl-2-(2-methylallyl)-1,2,3,4-tetrahydroquinoline (2r).** General Procedure D was employed for the coupling of N-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (54 mg, 0.20 mmol) and 2-Bromopropene (35 $\mu$L, 0.40 mmol) using NaO$^\text{t}$Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 52 mg (85%) of the title compound as an orange oil. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 0.8% IPA/Hexanes, 0.2 mL/min, $\lambda$ 254 nm, RT = 22.9 and 23.9 min). $[\alpha]^\text{23}_D$ +49.8 (c 1.04, CH$_2$Cl$_2$); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.22–7.00 (m, 3 H), 6.98 (d, $J = 8.2$ Hz, 2 H), 6.85 (t, $J = 7.4$ Hz, 1 H), 6.61 (t, $J = 7.0$ Hz, 1 H), 6.00 (d, $J = 8.2$ Hz, 1 H), 4.95 (s, 1 H), 4.77 (s, 1 H), 3.87 (s, 3 H), 3.02 (ddd, $J = 5.5, 9.2, 15.7$ Hz, 1 H), 2.85 (dt, $J = 6.0, 16.8$ Hz, 1 H), 2.48 (d, $J = 13.1$ Hz, 1 H), 2.27 (d, $J = 13.2$ Hz, 1 H), 2.07 (dt, $J = 5.8, 12.3$ Hz, 1 H), 1.93 (ddd, $J = 5.7, 9.6, 14.0$ Hz, 1 H), 1.81 (s, 3 H), 1.19 (s, 3 H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 158.3, 146.4, 142.4, 136.2, 133.5, 129.2, 126.6, 121.0, 116.1, 115.7, 115.1, 114.8, 57.1, 55.5, 46.8, 32.9, 27.0, 25.4, 24.6; IR (film) 2928, 1599, and 1506 cm$^{-1}$; MS (ESI+) 308.2008 (308.2009 calcd for C$_{21}$H$_{25}$NO, M + H$^+$).
\((R)-(\pm)-2\text{-Benzyl-2-ethyl-1-(4-methoxyphenyl)-1,2,3,4-tetrahydroquinoline} \quad (2\text{s})\). General Procedure D was employed for the coupling of \(N\)-(4-methoxyphenyl)-2-(3-methylenepentyl)aniline (28 mg, 0.10 mmol) and bromobenzene (21 \(\mu\)L, 0.20 mmol) using NaO\(\text{tBu}\) (19 mg, 0.20 mmol) as the base and a reaction temperature of 110 °C for 12 h. This procedure afforded 31 mg (86%) of the title compound as a viscous white oil. This material was judged to be 75:25 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 1.00% IPA/Hexanes, 0.75 mL/min, \(\lambda\) 254 nm, RT = 6.1 and 7.5 min) \([\alpha]^{23}_D +13.03 \ (c 1.35, \text{CH}_2\text{Cl}_2)\); \(^1\)H NMR (700 MHz, \(\text{C}_6\text{D}_6\)) \(\delta\) 7.26 (t, \(J = 7.2\) Hz, 2 H), 7.22 (t, \(J = 7.2\) Hz, 1 H), 7.18–7.09 (m, 3 H), 7.06 (d, \(J = 7.3\) Hz, 1 H), 6.93–6.71 (m, 4 H), 6.62 (t, \(J = 7.3\) Hz, 1 H), 6.11 (d, \(J = 8.3\) Hz, 1 H), 3.82 (s, 3 H), 3.16 (ddd, \(J = 5.9, 11.5, 17.0\) Hz, 1 H), 2.95 (d, \(J = 13.5\) Hz, 1 H), 2.90–2.81 (m, 2 H), 1.95–1.83 (m, 2 H), 1.54–1.47 (m, 1 H), 1.37 dq, \(J = 7.2, 14.5\) Hz, 1 H) 0.88 (t, \(J = 7.3\) Hz, 3 H); \(^1\)C NMR (175 MHz, CDCl\(_3\)) \(\delta\) 157.8, 147.3, 138.5, 136.9, 133.1, 130.9, 128.9, 127.9, 126.4, 126.1, 122.1, 117.0, 116.4, 114.4, 60.7, 55.4, 42.2, 29.2, 27.8, 24.2, 8.5; IR (film) 2927, 1599, and 1507 cm\(^{-1}\); MS (ESI+) 358.2163 (358.2165 calcd for \(\text{C}_{25}\text{H}_{27}\text{NO}\), M + H\(^+\)).

\((R)-(\pm)-6\text{-Methoxy-1-(4-methoxyphenyl)-2-methyl-2-(2-methylallyl)-1,2,3,4-tetrahydroquinoline} \quad (2\text{t})\). General Procedure D was employed for the coupling of 4-methoxy-\(N\)-(4-methoxyphenyl)-2-(3-methylbut-3-en-1-yl)aniline (60 mg, 0.20 mmol) and 2-bromopropene (35 \(\mu\)L, 0.40 mmol) using NaO\(\text{tBu}\) (38 mg, 0.40 mmol) as the base and a reaction temperature of 90 °C for 14 h. This procedure afforded 55 mg (82%) of the title compound as an orange oil. This material was judged to be 96:4 er by chiral HPLC
analysis (lux-amylose, 15 cm x 4.6 mm, 2.5% IPA/Hexanes, 0.2 mL/min, λ 254 nm, RT = 29.7 and 32.9). [α]$_{23}^D$ +68.3 (c 1.13, CH$_2$Cl$_2$); $^1$H NMR (700 MHz, CDCl$_3$) δ 7.16–7.03 (m, 2 H), 6.93 (d, $J$ = 7.7 Hz, 2 H), 6.65 (d, $J$ = 2.9 Hz, 1 H), 6.47 (dd, $J$ = 3.0, 8.9 Hz, 1 H), 5.98 (d, $J$ = 9.1 Hz, 1 H), 4.92 (s, 1 H), 4.76 (s, 1 H), 3.84 (s, 3 H), 3.72 (s, 3 H), 2.98 (ddd, $J$ = 5.8, 9.6, 16.0 Hz, 1 H), 2.83 (dt, $J$ = 5.9, 16.8 Hz, 1 H), 2.40 (d, $J$ = 13.1 Hz, 1 H), 2.25 (d, $J$ = 13.1 Hz, 1 H), 2.01 (dt, $J$ = 6.0, 13.2 Hz, 1 H), 1.90 (ddd, $J$ = 5.6, 9.6, 13.1 Hz, 1 H), 1.80 (s, 3 H), 1.14 (s, 3 H); $^{13}$C NMR (175 MHz, CDCl$_3$) δ 157.9, 150.9, 142.5, 140.8, 137.2, 133.3, 122.2, 116.7, 115.3, 114.4, 114.2, 112.5, 56.7, 55.7, 55.4, 46.4, 32.9, 26.6, 25.3, 24.7; IR (film) 2933, 1493 cm$^{-1}$; MS (ESI+) 337.2032 (337.2036 calcd for C$_{22}$H$_{27}$NO$_2$, M$^+$).

(R)-(+)-1-(4-Methoxyphenyl)-2-methyl-2-(3-methylbut-2-en-1-yl)-1,2,3,4-tetrahydrobenzo[h]quinoline (2u). General Procedure D was employed for the coupling of N-(4-Methoxyphenyl)-2-(3-methylbut-3-en-1-yl)naphthalen-1-amine (63 mg, 0.20 mmol) and 1-Bromo-2-methyl-1-propene (41 µL, 0.40 mmol) using NaO'Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 95 °C for 18 h. This procedure afforded 47 mg (63%) of the title compound as a clear oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 0.8% IPA/Hexanes, 0.150 mL/min, λ 254 nm, RT = 38.1 and 40.9 min). [α]$_{23}^D$ +281.2 (c 1.67, CH$_2$Cl$_2$); $^1$H NMR (500 MHz, C$_6$D$_6$) δ 8.34 (d, $J$ = 8.5 Hz, 1 H), 7.56 (d, $J$ = 8.0 Hz, 1 H), 7.41 (d, $J$ = 8.3 Hz, 1 H), 7.18-7.00 (m, 3 H), 6.95 (d, $J$ = 8.2 Hz, 2 H), 6.51 (d, $J$ = 8.3 Hz, 2 H), 5.48-5.43 (m, 1 H), 3.14 (s, 3 H), 2.85-2.76 (m, 2H), 2.44 (dd, $J$ = 7.0, 14.6 Hz, 1 H), 2.12 (dd, $J$ = 7.6, 14.6 Hz, 1 H), 1.78 (dt, $J$ = 9.0, 13.5 Hz, 1 H), 1.66 (s, 3 H), 1.52-1.44 (m, 1H), 1.35 (s, 3 H), 1.07 (s, 3 H); $^{13}$C NMR (125 MHz, C$_6$D$_6$) δ 157.2, 143.2, 142.4, 134.9, 133.3, 131.6, 131.3, 128.0, 126.4, 125.8, 125.7, 125.4, 123.8, 121.9, 114.1, 58.9, 55.0, 37.7, 28.7, 27.4, 26.6, 25.9, 18.4 (one peak missing from arene region due to
apparent overlap); IR (film) 2926, 1502, and 1390 cm⁻¹; MS (ESI+) 372.2326 (372.2322 calcd for C₂₆H₂₉NO, M + H⁺).

(S)-(–)-2-Benzyl-1-(4-methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxaline (4a). General Procedure D was employed for the coupling of N¹-(4-methoxyphenyl)-N²-methyl-N²-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.20 mmol) and bromobenzene (42 µL, 0.40 mmol) using NaO'Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 57 mg (79%) of the title compound as an orange oil. This material was judged to be 97:3 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1mL/min, λ 254 nm, RT = 5.1 and 7.9 min). [α]²³D –28.9 (c 1.19, CH₂Cl₂); ¹H NMR (700 MHz, C₆D₆) δ 7.15 (d, J = 7.5 Hz, 2 H), 7.11 (d, J = 6.6 Hz, 1 H), 7.08 (d, J = 7.5 Hz, 2 H), 6.96 (d, J = 8.5 Hz, 2 H), 6.86 (t, J = 7.5 Hz, 1 H), 6.81–6.75 (m, 2 H), 6.74 (t, J = 7.8 Hz, 1 H), 6.70 (d, J = 8.0 Hz, 1 H), 6.40 (d, J = 8.0 Hz, 1 H), 3.32 (s, 3 H), 3.22 (d, J = 12.6 Hz, 1 H), 2.83 (d, J = 12.6 Hz, 1 H), 2.70 (d, J = 10.9 Hz, 1 H), 2.65 (d, J = 10.9 Hz, 1 H), 2.62 (s, 3 H), 0.90 (s, 3 H); ¹³C NMR (175 MHz, CDCl₃) δ 158.7, 139.1, 137.0, 136.5, 136.5, 133.5, 131.2, 128.4, 128.3, 126.5, 119.2, 118.4, 115.5, 115.0, 111.9, 58.0, 57.5, 54.9, 44.1, 38.9, 23.8 (an extra peak at 136.5 is present due to apparent slow bond rotation); IR (film) 2928, 1503 cm⁻¹; MS (ESI+) 359.2118 (359.2118 calcd for C₂₄H₂₆N₂O, M + H⁺).
(S)-(+)-(4-[[1-(4-Methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxalin-2-yl]methyl]phenyl)(phenyl)methanone (4b). General Procedure D was employed for the coupling of \(N^1\)-(4-methoxyphenyl)-\(N^2\)-methyl-\(N^2\)-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and 4-bromobenzophenone (104 mg, 0.40 mmol) using NaO\(\text{Bu}\) (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 72 mg (78%) of the title compound as a light yellow solid, mp 63–66 °C. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 3% IPA/Hexanes, 1 mL/min, \(\lambda\) 254 nm, RT = 18.2 and 25.9 min), \([\alpha]^{23}_D\) +18.7 (c 0.90, CH\(_2\)Cl\(_2\)); \(^1\)H NMR (700 MHz, CDCl\(_3\)) \(\delta\) 7.79 (d, \(J = 7.0\) Hz, 2 H), 7.75 (d, \(J = 8.0\) Hz, 2 H), 7.15 (d, \(J = 8.5\) Hz, 1 H), 7.08 (t, \(J = 7.6\) Hz, 2 H), 7.02 (d, \(J = 8.0\) Hz, 2 H), 6.96 (d, \(J = 8.9\) Hz, 2 H), 6.87 (t, \(J = 7.5\) Hz, 1 H), 6.80 (d, \(J = 8.2\) Hz, 2 H), 6.75 (t, \(J = 8.0\) Hz, 1 H), 6.71 (d, \(J = 8.0\) Hz, 1 H), 6.40 (d, \(J = 8.0\) Hz, 1 H), 3.34 (s, 3 H), 3.22 (d, \(J = 12.4\) Hz, 1 H), 2.81 (d, \(J = 12.3\) Hz, 1 H), 2.65 (d, \(J = 11.1\) Hz, 1 H), 2.61–2.56 (m, 4 H), 0.85 (s, 3 H); \(^1\)C NMR (175 MHz, CDCl\(_3\)) \(\delta\) 195.5, 158.8, 138.6, 136.8, 136.4, 136.3, 136.2, 133.4, 132.0, 131.0, 130.2, 130.2, 128.4, 119.3, 118.6, 115.6, 115.0, 112.0, 57.9, 57.6, 55.0, 43.9, 38.9, 23.8 (one aromatic carbon signal is missing due to incidental equivalence); IR (film) 2972, 1656 1504 cm\(^{-1}\); MS (ESI+) 463.2371 (463.2380 calcd for C\(_{31}\)H\(_{30}\)N\(_2\)O\(_2\), M + H\(^+\)).
**(S)-(+)-1-(4-Methoxyphenyl)-2,4-dimethyl-2-(naphthalen-2-ylmethyl)-1,2,3,4-tetrahydroquinoxaline (4c).** General Procedure D was employed for the coupling of \(N^1-(4\text{-methoxyphenyl})-N^2\text{-methyl}-N^3\text{-}(2\text{-methylallyl})benzene-1,2-diamine\) (59 mg, 0.2 mmol) and 2-bromonaphthalene (83 mg, 0.40 mmol) using \(\text{NaO}^\text{tBu}\) (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 67 mg (82%) of the title compound as a light yellow solid, mp 62–65 °C. This material was judged to be 93:7 er by chiral HPLC analysis (lux-amylose, 15 cm x 4.6 mm, 3% IPA/Hexanes, 0.25 mL/min, \(\lambda\) 254 nm, RT = 27.9 and 30.2 min), \([\alpha]^{23}_D\) +8.56 (c 1.39, \(\text{CH}_2\text{Cl}_2\)); \(^1\text{H NMR (700 MHz, CDCl}_3\)) \(\delta\) 7.70–7.64 (m, 2 H), 7.59 (t, \(J = 8.2\) Hz, 1 H), 7.52 (s, 1 H), 7.33–7.26 (m, 2 H), 7.22 (t, \(J = 8.4\) Hz, 1 H), 7.08–6.98 (m, 2 H), 6.90 (t, \(J = 7.7\) Hz, 1 H), 6.84–6.74 (m, 4 H), 6.44 (d, \(J = 8.0\) Hz, 1 H), 3.40 (d, \(J = 12.8\) Hz, 1 H), 3.35 (s, 3 H), 2.96 (d, \(J = 12.8\) Hz, 1 H), 2.71–2.62 (m, 5 H), 0.93 (s, 3 H); \(^{13}\text{C NMR (175 MHz, CDCl}_3\)) \(\delta\) 158.7, 137.0, 136.6, 136.5, 136.4, 134.1, 133.7, 133.5, 132.8, 129.8, 129.7, 128.0, 127.7, 126.2, 125.7, 119.3, 118.4, 115.4, 115.0, 112.0, 57.9, 57.8, 55.0, 44.1, 38.9, 23.9; IR (film) 2969, 1504 cm\(^{-1}\); MS (ESI+) 409.2268 (409.2274 calcd for \(\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}\,\text{M} + \text{H}^+\)).

**(S)-(+)-4-(4-[[1-(4-Methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxalin-2-yl]methyl]phenyl)morpholine (4d).** General Procedure D was employed for the
coupling of $N'$- (4-methoxyphenyl) - $N^2$-methyl- $N^2$-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and 4-(4-bromophenyl)morpholine (97 mg, 0.40 mmol) using NaO'Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 73 mg (82%) of the title compound as a white solid, mp 72–75 °C. This material was judged to be 96:4 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 2% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 16.3 and 17.8 min), $\left[\alpha\right]_{23}^{D} +21.6$ (c 0.99, CH₂Cl₂); $^1$H NMR (700 MHz, CDCl₃) δ 7.08 (d, $J$ = 8.4 Hz, 2 H), 7.02 (d, $J$ = 7.8 Hz, 2 H), 6.88 (t, $J$ =7.2 Hz, 1 H), 6.83–6.72 (m, 4 H), 6.67 (d, $J$ = 8.3 Hz, 2 H), 6.43 (d, $J$ = 7.0 Hz, 1 H), 3.58 (t, $J$ = 4.6 Hz, 4 H), 3.33 (s, 3 H), 3.25 (d, $J$ = 12.9 Hz, 1 H), 2.87 (d, $J$ = 12.9 Hz, 1 H), 2.83 (d, $J$ = 10.8 Hz, 1 H), 2.79 (d, $J$ = 4.7 Hz, 4 H), 2.72 (d, $J$ = 10.9 Hz, 1 H), 2.70 (s, 3 H), 0.99 (s, 3 H); $^{13}$C NMR (175 MHz, CDCl₃) δ 158.7, 150.3, 137.1, 136.6, 136.5, 133.5, 131.8, 130.0, 128.2, 119.2, 118.3, 115.7, 115.4, 114.9, 111.9, 67.0, 58.1, 57.7, 54.9, 49.6, 43.3, 39.0, 23.8 (an extra peak appears at 136.5 is present due to apparent slow bond rotation); IR (film) 2957, 150 4 cm⁻¹; MS (ESI+) 444.2645 (444.2646 calcd for C₂₈H₃₃N₃O₂, M + H⁺).

(S)-(+)-2-[4-(1H-pyrrol-1-yl)benzyl]-1-(4-methoxyphenyl)-2,4-dimethyl-1,2,3,4-tetrahydroquinoxaline (4e). General Procedure BD was employed for the coupling of $N'$- (4-methoxyphenyl) - $N^2$-methyl- $N^2$-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and 1-(4-iodophenyl)pyrrole (108 mg, 0.40 mmol) using NaO'Bu (38 mg, 0.40 mmol) as the base and a reaction temperature of 120 °C in xylenes for 14 h. This procedure afforded 60 mg (70%) of the title compound as an white solid, mp 65–68 °C. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ADH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 16.2 and 22.5 min), $\left[\alpha\right]_{23}^{D} +22.7$
(S,Z)-(+) -1-(4-methoxyphenyl)-2,4-dimethyl-2-(pent-2-en-1-yl)-1,2,3,4-tetrahydroquinoxaline (4f). General Procedure D was employed for the coupling of N1-(4-methoxyphenyl)-N2-methyl-N2-(2-methylallyl)benzene-1,2-diamine (59 mg, 0.2 mmol) and Z-1-bromobutene (54 mg, 0.40 mmol) using NaOTBu (38 mg, 0.40 mmol) as the base and a reaction temperature of 110 °C for 14 h. This procedure afforded 50 mg (75%) of the title compound as a clear oil. This material was judged to be 98:2 er by chiral HPLC analysis (Chriacel ADH, 15 cm x 4.6 mm, 0.50% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 6.6 and 9.3 min), [α]23D +21.7 (c 2.4, CH2Cl2); 1H NMR (500 MHz, C6D6) δ 7.04 (d, J = 7.3 Hz, 2 H), 6.84 (t, J = 7.3 Hz, 1 H), 6.78 (d, J = 8.6 Hz, 2 H), 6.72 (t, J = 7.7 Hz, 1 H), 6.65 (d, J = 7.8 Hz, 1 H), 6.35 (d, J = 8.0 Hz, 2 H), 5.50–5.41 (m, 1 H), 5.40–5.32 (m, 1 H), 3.32 (s, 3 H), 2.95 (d, J = 10.8 Hz, 1 H), 2.79 (d, J = 10.8 Hz, 1 H), 2.70–2.61 (m, 4 H), 2.48 (dd, J= 13.7, 7.1 Hz, 1H), 2.05–1.90 (m, 2H), 1.04 (s, 3 H), 0.89 (t, J= 7.3 Hz, 3H); 13C NMR (125 MHz, CDCl3) δ 158.7, 137.4, 136.4, 136.3, 134.5, 133.6, 124.8, 119.2, 118.2, 115.0, 114.9 111.8, 59.2, 57.0, 54.9, 39.4, 36.0, 23.9, 21.0, 14.5; IR (film) 2957, 1671, 1504 cm⁻¹; MS (ESI+) 337.2276 (337.2274 calcd for C22H28N2O, M + H⁺).
(S)-(−)-2-Benzyl-2-[[benzyloxy)methyl]-1-(4-methoxyphenyl)-4-methyl-1,2,3,4-tetrahydroquinoxaline (4g): General Procedure D was employed for the coupling of \( N^1 \)-[2-[(benzyloxy)methyl]allyl]-\( N^2 \)-(4-methoxyphenyl)-\( N^1 \)-methylbenzene-1,2-diamine (39 mg, 0.1 mmol) and bromobenzene (21 µL, 0.20 mmol) using NaO\textsubscript{t}Bu (19 mg, 0.20 mmol) as the base and a reaction temperature of 125 °C for 12 h. This procedure afforded 37 mg (79%) of the title compound as a viscous white oil. This material was judged to be 96:4 er by chiral HPLC analysis (Chriacel ADH, 15 cm x 4.6 mm, 1.00% IPA/Hexanes, 1 mL/min, λ 254 nm, RT = 7.0 and 10.6 min), \([\alpha]^{23}\textsubscript{D} = -19.51 (c 1.23, CH\textsubscript{2}Cl\textsubscript{2})\); \(^1\text{H} NMR (700 MHz, C\textsubscript{6}D\textsubscript{6}) \delta 7.31 (t, \( J = 7.5 \) Hz, 2 H), 7.28–7.18 (m, 7 H), 7.16 (d, \( J = 7.3 \) Hz, 2 H), 6.98–6.90 (m, 1 H), 6.86 (d, \( J = 8.9 \) Hz, 2 H), 6.69–6.64 (m, 2H), 6.51 (t, \( J = 7.2 \) Hz, 1 H), 6.08 (d, \( J = 8.0 \) Hz, 1 H), 4.29 (d, \( J = 11.8 \) Hz, 1 H), 4.24 (d, \( J = 11.8 \) Hz, 1 H), 3.82 (s, 3 H), 3.39 (d, \( J = 9.9 \) Hz, 1 H), 3.23 (d, \( J = 11.1 \) Hz, 1 H), 3.21 (d, \( J = 9.7 \) Hz, 1 H) 3.09 (d, \( J = 13.3 \) Hz, 1 H), 3.04 (d, \( J = 10.9 \) Hz, 1 H), 3.00 (d, \( J = 13.1 \) Hz, 1 H), 3.02 (s, 3 H); \(^{13}\text{C} NMR (175 MHz, CDCl\textsubscript{3}) \delta 158.1, 138.2, 138.1, 136.4, 136.0, 135.8, 133.4, 132.8, 130.9, 128.3, 128.1, 127.5, 127.4, 126.3, 118.2, 117.6, 115.0, 114.6, 114.2, 111.1, 72.9, 72.1, 60.6, 55.4, 54.1, 40.3, 39.2 (extra peaks at 132.8 and 114.2 are present due to apparent slow bond rotation); IR (film) 2923, 2859, 1504 cm\(^{-1}\); MS (ESI+) 465.2536 (465.2537 calcd for C\textsubscript{31}H\textsubscript{32}N\textsubscript{2}O\textsubscript{2}, M + H\(^{+}\)).
(S)-(+-)-Methyl 3-(4-methoxybenzyl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (6a). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 4-bromoanisole (60 mg, 0.32 mmol) using NaO\textsubscript{t}Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 40 mg (51%) of the title compound as a colorless oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 5.8 and 12.1 min), [\alpha]\textsuperscript{23}D +48.8 (c 0.86, CH\textsubscript{2}Cl\textsubscript{2}); \textsuperscript{1}H NMR (700 MHz, d\textsubscript{8}-toluene, 95 °C) δ 7.01–6.95 (m, 2 H), 6.92 (d, \textit{J} = 8.2 Hz, 2 H), 6.88–6.83 (m, 2 H), 6.69 (d, \textit{J} = 8.5 Hz, 1 H), 4.82 (d, \textit{J} = 16.6 Hz, 1 H), 4.73 (s, br, 1 H), 4.34 (d, \textit{J} = 16.7 Hz, 1 H), 3.60 (s, 3 H), 3.49 (s, 3 H), 2.74–2.67 (m, 2 H), 2.67 (d, \textit{J} = 13.6 Hz, 1 H), 2.35 (dd, \textit{J} = 9.0, 13.5, Hz 1 H); \textsuperscript{13}C NMR (175 MHz, d\textsubscript{8}-toluene, 95 °C) δ 159.6, 156.4, 134.2, 134.9, 131.6, 130.8, 129.8, 127.3, 126.9, 126.8, 114.9, 55.3, 52.7, 52.4, 44.3, 38.4, 32.5; IR (film) 2952, 1695 cm\textsuperscript{-1}; MS (ESI+) 312.1589 (312.1594 calcd for C\textsubscript{19}H\textsubscript{21}NO\textsubscript{3}, M + H\textsuperscript{+}).

(\textit{S})-(+-)-Methyl 3-(2-methoxybenzyl)-3,4-dihydroisoquinoline-2(1H)-carboxylate (6b). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 2-iodoanisole (85 mg, 0.32 mmol) using NaO\textsubscript{t}Bu (31 mg, 0.32
mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 33 mg (42%) of the title compound as a white solid, mp 74-77 °C. This material was judged to be 80:20 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 5.7 and 8.2 min), [α]D 23 +77.0 (c 0.67, CH2Cl2); 1H NMR (700 MHz, d8-toluene, 95 °C) δ 7.02–6.92 (m, 5 H), 6.88–6.82 (m, 2 H), 6.73 (t, J = 7.5 Hz, 1 H), 6.56 (d, J = 8.1 Hz, 1 H), 4.88 (s, br, 1 H), 4.80 (d, J = 16.8 Hz, 1 H), 4.32 (d, J = 16.8 Hz, 1 H), 3.44 (s, 3 H), 3.41 (s, 3 H), 2.80–2.72 (m, 2 H), 2.64 (dd, J = 8.1, 13.2 Hz, 1 H), 2.46 (d, J = 15.9 Hz, 1 H); 13C NMR (175 MHz, d8-toluene, 95 °C) δ 159.1, 156.7, 134.3, 134.2, 131.9, 130.0, 127.2, 126.9, 126.8, 121.3, 111.5, 55.6, 52.4, 51.3, 44.3, 33.7, 33.4 (two aromatic carbon signals are missing due to incidental equivalence); IR (film) 2951, 1698 cm⁻¹; MS (ESI+) 312.1593 (312.1594 calcd for C19H21NO3, M + H⁺).

(S)-(+-)-Methyl 3-[4-(trifluoromethyl)benzyl]-3,4-dihydroisoquinoline-2(1H)-carboxylate (6c). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 4-bromobenzotrifluoride (72 mg, 0.32 mmol) using NaOTBu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 63 mg (72%) of the title compound as a colorless oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 4.4 and 9.3 min), [α]D 23 +39.5 (c 1.24, CH2Cl2); 1H NMR (700 MHz, d8-toluene, 95 °C) δ 7.32 (d, J = 7.8 Hz, 2 H), 7.03–6.94 (m, 2 H), 6.92 (d, J = 7.8 Hz, 2 H), 6.87–6.79 (m, 2 H), 4.67 (d, J = 16.6 Hz, 1 H), 4.58 (s, br, 1 H), 4.19 (d, J = 16.7 Hz, 1 H), 3.47 (s, 3 H), 2.68–2.62 (m, 2 H), 2.34–2.26 (m, 2 H); 13C NMR (175 MHz, d8-toluene, 95 °C) δ 156.3, 143.8, 133.9, 133.5, 130.3,
129.7, 127.5, 127.2, 126.8, 125.8, 152.5, 152.3, 44.3, 39.1, 32.6 (two aromatic carbon signals are missing due to incidental equivalence); IR (film) 2954, 1695 cm\(^{-1}\). MS (ESI\(^{+}\)) 350.1365 (350.1362 calcd for C\(_{19}\)H\(_{18}\)F\(_3\)NO\(_2\), M + H\(^{+}\)).

\((S)-(+)\)-Methyl 3-[3-(4-fluorophenoxy)benzyl]-3,4-dihydroisoquinoline-2(1H)-carboxylate (6d). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and 3-bromo-4'-fluorodiphenyl ether (85 mg, 0.32 mmol) using NaO\(_{t}\)Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 60 mg (61%) of the title compound as colorless oil. This material was judged to be 93:7 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 1 mL/min, \(\lambda\) 215 nm, RT = 7.7 and 13.3 min), \([\alpha]_{D}^{23}\) +48.3 (c 1.18, CH\(_2\)Cl\(_2\)); \(^1\)H NMR (700 MHz, d\(_8\)-toluene, 95 °C) \(\delta\) 7.02–6.93 (m, 3 H), 6.83–6.68 (m, 9 H), 4.68 (d, \(J = 16.8\) Hz, 1 H), 4.62 (s, br, 1 H), 4.20 (d, \(J = 16.6\) Hz, 1 H), 3.46 (s, 3 H), 2.71–2.62 (m, 2 H), 2.39 (d, \(J = 15.9\) Hz, 1 H), 2.31 (dd, \(J = 8.5, 13.5\) Hz, 1 H); \(^13\)C NMR (175 MHz, d\(_8\)-toluene, 95 °C) \(\delta\) 159.8 (d, \(J = 241\) Hz), 158.8, 156.3, 154.2, 141.8, 134.0, 133.6, 130.2, 129.7, 127.4, 127.0, 126.8, 124.9, 121.1 (d, \(J = 7\) Hz), 120.3, 117.2, 116.8 (d, \(J = 23\) Hz), 52.5, 44.3, 39.2, 32.8 (one aliphatic carbon signal is missing due to incidental equivalence); IR (film) 2952, 1695, 1500 cm\(^{-1}\); MS (ESI\(^{+}\)) 392.1658 (392.1656 calcd for C\(_{24}\)H\(_{22}\)FNO\(_2\), M + H\(^{+}\)).
(S)-(+) -Methyl 3-benzyl-3,4-dihydroisoquinoline-2(1H)-carboxylate (6e). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and bromobenzene (51 mg, 0.32 mmol) using NaO'Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 48 mg (68%) of the title compound as a colorless oil. This material was judged to be 94:6 er by chiral HPLC analysis (Chiracel ODH, 15 cm x 4.6 mm, 5% IPA/Hexanes, 0.5 mL/min, λ 215nm, RT = 9.9 and 18.5 min), [α]_{23}^{20} +63.2 (c 1.28, CH_{2}Cl_{2}); {^1}H NMR (700 MHz, d8-toluene, 90 °C) δ 7.10–7.03 (m, 2 H), 7.03–6.95 (m, 5 H), 6.87–6.81 (m, 2 H), 4.72 (d, \( J = 16.7 \) Hz, 1 H), 4.66 (s, br, 1 H), 4.24 (d, \( J = 16.7 \) Hz, 1 H), 3.49 (s, 3 H), 2.72 (dd, \( J = 5.8, 13.3 \) Hz, 1 H), 2.67 (dd, \( J = 5.6, 15.7 \) Hz, 1 H), 2.41 (d, \( J = 15.7 \) Hz, 1 H), 2.37 (dd, \( J = 8.9, 13.5 \) Hz, 1 H); {^{13}}C NMR (125 MHz, d8-toluene, 90 °C) δ 156.3, 139.7, 134.1, 133.8, 130.0, 129.7, 129.0, 127.3, 126.9, 126.9, 126.8, 52.6, 52.4, 44.3, 39.3, 32.5; IR (film) 2953 1697 cm^{-1}; MS (ESI+) 282.1491 (282.1489 calcd for C_{18}H_{19}NO_2, M + H^+).

(Z,S)-(+) -Methyl 3-[4-(trifluoromethyl)benzyl]-3,4-dihydroisoquinoline-2(1H)-carboxylate (6f). General Procedure D was employed for the coupling of (2-allylbenzyl)carbamate (51 mg, 0.25 mmol) and (Z)-1-bromo-1-butene (43 mg, 0.32 mmol) using NaO'Bu (31 mg, 0.32 mmol) as the base and a reaction temperature of 90 °C for 2 h. This procedure afforded 36 mg (57%) of the title compound as a colorless oil.
This material was judged to be 93:7 er by chiral HPLC analysis (ODH, 15 cm x 4.6 mm, 1% IPA/Hexanes, 1 mL/min, λ 215 nm, RT = 6.7 and 22.1 min), [α]_{D}^{23} +46.7 (c 1.12, CH₂Cl₂); (The mixture was found to exist as a 2.5:1 mixture of rotomers in the nmr, with most of the minor rotomer peaks appearing partially in the major rotomer peaks, however coupling constants given are all for the major rotomer) ¹H NMR (700 MHz, d8-toluene, 95 °C) δ 6.98–6.93 (m, 2 H), 6.88–6.84 (m, 2 H), 6.83–6.79 (m, 2 H), 5.38–5.33 (m, 1 H), 5.30–5.25 (m, 1 H), 4.78 (d, J = 16.7 Hz, 1 H), 4.48 (s, br, 1 H), 4.19 (d, J = 16.7 Hz, 1 H), 3.55 (s, 3 H), 2.78 (dd, J = 6.1, 15.6 Hz, 1 H), 2.46 (d, J = 15.7 Hz, 1 H), 2.20–2.15 (m, 1 H), 2.00–1.95 (m, 1 H), 1.90-1.84 (m, 0.63 H), 1.80 (quin, J = 7.30 Hz, 1.45 H), 0.87 (t, J = 7.5 Hz, 0.76 H), 0.80 (t, J = 7.5 Hz, 2.22 H); ¹³C NMR (125 MHz, d8-toluene, 95 °C) δ 156.4, 134.5, 134.1, 133.9, 129.7, 127.2, 126.9, 126.6, 125.7, 52.6, 51.1, 44.1, 33.1, 30.7, 21.2, 14.5; IR (film) 2958, 1699 cm⁻¹; MS (ESI⁺) 260.1644 (260.1645 calcd for C₁₆H₂₁NO₂, M + H⁺).

Confirmation of product 6f structure.

The NMR spectrum of 6f was complicated due to apparent rotomers. In order to rule out the presence of E:Z alkene stereoisomers the product was reduced with LiAlH₄ to form the analogous N-methyl isoquinoline derivative S₄.

(Z,S)-(−)-2-methyl-3-(pent-2-en-1-yl)-1,2,3,4-tetrahydroisoquinoline (S₄). A flame-dried round-bottom flask equipped with a stirbar was cooled under a stream of nitrogen and charged with 6f (35 mg, 0.136 mmol) and THF (4 mL). The resulting solution was cooled to 0°C and after five minutes of stirring LiAlH₄ (0.27 mL, 0.27 mmol, 1.0M in THF) was added dropwise. The resulting solution was heated to reflux for 1.5 h then cooled to rt and quenched with 0.1 mL H₂O followed by 0.1 mL of a 15% aqueous
NaOH solution. The mixture was filtered and the solid was washed with ether (2 x 5) mL. The combined organic solutions were dried over anhydrous MgSO₄, filtered, and concentrated in vacuo. The crude material was purified by flash chromatography on silica gel using hexanes/EtOAc as the eluent to afford 20 mg (69%) of the product as a clear oil; [α]²³D −24.7 (c 1.86, CH₂Cl₂). ¹H NMR (700 MHz, CDCl₃) δ 7.13–7.09 (m, 2 H), 7.08–7.05 (m, 1 H), 7.04–7.00 (m, 1 H), 5.53–5.48 (m, 1 H), 5.44–5.39 (m, 1 H), 3.82 (d, J = 15.5 Hz, 1 H), 3.68 (d, J = 15.6 Hz, 1 H), 2.79–2.72 (m, 1 H), 2.69–2.58 (m, 2 H), 2.36 (s, 3 H), 2.32–2.27 (m, 1 H), 2.10–2.04 (m, 1H), 2.03 (quint, J = 7.5 Hz, 2 H), 0.97 (t, J = 7.5 Hz, 3 H); ¹³C NMR (175 MHz, CDCl₃) δ 134.3, 134.0, 133.7, 128.8, 126.2, 126.1, 125.6, 125.4, 59.1, 56.4, 41.0, 32.3, 28.3, 20.7, 14.2; IR (film) 2960, 1456 cm⁻¹; MS (ESI+ 216.1747 (216.1747 calcd for C₁₅H₂₁N, M + H⁺).
Results of a screen of chiral ligands in the reaction of 1b with 1-bromo-4-tert-butylbenzene.

84% yield, 54.46 er

78% yield, 59.41 er

(R)-(S)-Josiphos
25% yield, 62.38 er

(R)-Siphos-PE
80% yield, 68.32 er

(Ra-S)-Ph-Bn-SIPHOX
65% yield, 83.17 er

(S)-Phanephos
86% yield, 64.38 er
Assignment of Absolute Stereochemistry.

The absolute stereochemistry of product 2j was established by single crystal x-ray analysis as shown below. The stereochemistry of all other products was assigned based on analogy to 2j.

References

Phosphorus-31

Sample Name:

Data Collected on:
yh. che. jls. with adw/tauare/500

Archive Directory:

File Name: MB-NMR-186703

Phase sequence: proton signal

Sample: (dml)

Data collected on: Dec 10 2013

---

Phosphorus-31

Sample Name:

Data Collected on:
yh. che. jls. with adw/tauare/500

Archive Directory:

File Name: MB-NMR-186703

Phase sequence: proton signal

Sample: (dml)

Data collected on: Dec 10 2013
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**Phenyl-

Sample Name:

Data Collected on:

pH 7.0, 20°C, 99% DMSO/700

Sample directory:

File Name: SBB-6-177800

Pulse sequence: HETCOR (ipap)

Element: C

Data collected on: Jun 11 2013

**Phenyl-

Sample Name:

Data Collected on:

pH 7.0, 20°C, 99% DMSO/700

Sample directory:

File Name: SBB-6-177800

Pulse sequence: HETCOR (ipap)

Element: C

Data collected on: Jun 11 2013
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PDA Multi 1/254nm 4nm

S 94
Shimadzu LC solution Analysis Report

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1 PDA Multi 1/254nm 4nm

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PDA Ch1 254nm 4nm
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Software Version: 6.0.1

File Name: S2021010005

Spectrum 1

Spectrum 2

S 102
==== Shimadzu LCsolution Analysis Report ====  

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==== Shimadzu LCsolution Analysis Report ====

Acquired by: Admin
Sample Name: CHIRAL-BAH-V-175(2):0.80%IPA-0.10mL-min-ODH.lcd
Sample ID:
Tray #: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-V-175(2):0.80%IPA-0.10mL-min-ODH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 1/10/2014 1:00:16 PM
Data Processed: 1/10/2014 1:45:08 PM

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PDA Multi 1

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==== Shimadzu LCsolution Analysis Report ====

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Sample Name: RAC-BAH-V-151(III)-1.00%IPA-0.20mL_min-ODH
Sample ID: <SAMPLE>
Tray #: 1
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Injection Volume: 1 uL
Data File Name: RAC-BAH-V-151(III)-1.00%IPA-0.20mL_min-ODH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name:
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Data Acquired: 6/25/2013 7:51:01 AM

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PDA Multi 1

PDA Ch1 254nm 444nm
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-V-175(6)-1.00%IPA-0.20mL_min-ODH
Sample ID: <SAMPLE>
Tray #: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-V-175(6)-1.00%IPA-0.20mL_min-ODH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 7/8/2013 10:03:46 AM

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-V-150(II)-0.90/IPA-0.10 mL/min-ODH
Sample ID: 1
Tray#: 1
Vial #: 1
Injection Volume: 1 mL
Data File Name: CHIRAL-BAH-V-150(II)-0.90/IPA-0.10 mL/min-ODH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 1/8/2014 1:40:15 PM
Data Processed: 1/8/2014 2:31:20 PM

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-6-184(1)-5.00%IPA-1.00mL-min-ADH
Sample ID:
Tray #: 1
Val # : 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-6-184(1)-5.00%IPA-1.00mL-min-ADH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: Default.lcr
Report File Name: Default.lcr
Data Acquired: 1/15/2014 2:01:33 PM
Data Processed: 1/15/2014 2:39:14 PM

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[Graph showing chromatogram data]

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-5-186(1)-5.00%IPA-1.00mL-min-ADH
Sample ID: 1
Tray #: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-5-186(1)-5.00%IPA-1.00mL-min-ADH.icd
Method File Name: Cyclic Urea Method.icm
Batch File Name: Default.icr
Report File Name: Default.icr
Data Acquired: 1/15/2014 3:37:40 PM
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== Shimadzu LCsolution Analysis Report ==

Acquired by: Admin
Sample Name: RAC-BAH-6-180(2)-0.40%IPA-1.10mL-min-ADH
Sample ID: 1
Tray #: 1
Vial #: 1
Injection Volume: 1 mL
Data File Name: RAC-BAH-6-180(2)-0.40%IPA-1.10mL-min-ADH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: Default.lcr
Report File Name: Default.lcr
Data Acquired: 1/14/2014 5:23:26 PM
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<Chromatogram>

PDA Multi 9

PDA Multi 1:254nm 4nm

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-182(1)-0.40%IPA-1.10mL-min-ADH
Sample ID:
Tray #: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: CHIRAL-BAH-6-182(1)-0.40%IPA-1.10mL-min-ADH.lcd
Method File Name: Cyclic Urea Method Icm
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 1/14/2014 6:07:27 PM
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==== Shimadzu LCsolution Analysis Report====

Acquired by: Admin
Sample Name: RAC-BAH-6-180(1)-8.00%IPA-1.00mL-min-ADH
Sample ID:
Tray #:
Vial #:
Injection Volume: 1 μL
Data File Name: RAC-BAH-6-180(1)-8.00%IPA-1.00mL-min-ADH.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 1/16/2014 10:04:39 AM
Data Processed: 1/16/2014 10:27:15 AM

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PDA Multi 1
== Shimadzu LCsolution Analysis Report ==

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-182(3)-8.00%IPA-1.00mL-min-ADH
Sample ID:
Tray #: 1
Vial #: 1
Injection Volume: 1 μL
Data File Name: CHIRAL-BAH-6-182(3)-8.00%IPA-1.00mL-min-ADH.lcd
Method File Name: Cyclic Urea Method icm
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 1/16/2014 10:51:31 AM
Data Processed: 1/19/2014 11:25:02 AM

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[Insert image of chromatogram]

Peak Table

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==== Shimadzu LCsolution Analysis Report ==== 

Acquired by: Admin  
Sample Name: RAC-BAH-6-180(3)-0.80IPA-0.20mL-min-ADH  
Sample ID: 1  
Tray #: 1  
Vial #: 1  
Injection Volume: 1 uL  
Data File Name: RAC-BAH-6-180(3)-0.80IPA-0.20mL-min-ADH.lcd  
Method File Name: Cyclic Urea Method lcm  
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Report File Name: Default.lcr  
Data Acquired: 1/14/2014 1:01:51 PM  
Data Processed: 1/14/2014 1:32:16 PM  

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==== Shimadzu LC solution Analysis Report ====  

Acquired by: Admin  
Sample Name: CHIRAL-BAH-6-182(2)-0.80%IPA-0.20mL-min-ADH  
Sample ID:   
Tray#: 1  
Vial #: 1  
Injection Volume: 1 uL  
Data File Name: CHIRAL-BAH-6-182(2)-0.80%IPA-0.20mL-min-ADH.lcd  
Method File Name: Cyclic Urea Method.lcm  
Batch File Name:   
Report File Name: Default.lcr  
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Data Processed: 1/14/2014 4:45:54 PM  

<Chromatogram>

![Chromatogram Image]

**PDA Multi 1**

**PeakTable**

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== Shimadzu LCsolution Analysis Report ==

Acquired by: Admin
Sample Name: RAC-BAH-6-184(2)-0.80%IPA-0.20mL.min-ADH
Tray#: 1
Val #: 1
Injection Volume: 1.0 µL
Data File Name: RAC-BAH-6-184(2)-0.80%IPA-0.20mL.min-ADH.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name:
Report File Name: Default.icr
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==== Shimadzu LCsolution Analysis Report====

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-182(5)-0.80%IPA-0.20mL-min-ADH
Sample ID:
Tray #:
Vial #:
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-6-182(5)-0.80%IPA-0.20mL-min-ADH.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name:
Report File Name: Default.icr
Data Acquired: 1/15/2014 10:26:04 AM
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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-8-12(A)-1.0%IPA-0.75mL_min-ADH-frac28
Sample ID: 
Tray #: 1
Val #: 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-8-12(A)-1.0%IPA-0.75mL_min-ADH-frac28.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name: 
Report File Name: Default.icr
Data Acquired: 7/30/2014 12:39:14 PM
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== Shimadzu LCsolution Analysis Report ==

Acquired by: Admin
Sample Name: CHIRAL-BAH-8-18(II)-1.00%IPA-0.75mL_min-ADH1
Sample ID:
Tray #: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-8-18(II)-1.00%IPA-0.75mL_min-ADH1.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name:
Report File Name: Default.icr
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Data Processed: 7/31/2014 11:34:09 PM

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PDA Multi 1

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PDA Ch1 254nm-4nm

PeakTable

S 131
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-7-46(1)-0.80%IPA-0.150ml_min-ADH
Sample ID:
Tray #:
Well #:
Injection Volume: 1 uL
Data File Name: RAC-BAH-7-46(1)-0.80%IPA-0.150ml_min-ADH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name:
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==== Shimadzu LCsolution Analysis Report ====

Acquired by: Admin
Sample Name: CHIRAL-BAH-7-46(3)-0.80%IPA-0.150ml_min-ADH
Sample ID: 1
Tray #: 1
Vail #: 1
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-7-46(3)-0.80%IPA-0.150ml_min-ADH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 3/3/2014 10:52:41 AM

<Chromatogram>

![Chromatogram Image]

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-6-201(1)-2.50%IPA-0.200mL_min-luxamyllose.lcd
Tray #: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: RAC-BAH-6-201(1)-2.50%IPA-0.200mL_min-luxamyllose.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: Default.lcm
Report File Name: Data Acquired: 2/3/2014 1:55:36 PM
Data Processed: 2/3/2014 3:05:38 PM

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Peak Table

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-7-12-2.50%/IPA-0.20mL_min-luxamylose
Sample ID:
Tray #:
Vial #:
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-7-12-2.50%/IPA-0.20mL_min-luxamylose.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name:
Report File Name: Default.icr
Data Acquired: 2/5/2014 2:48:02 PM
Data Processed: 2/5/2014 3:34:27 PM

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![Chromatogram Image]

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==== Shimadzu LCsolution Analysis Report ====  

Acquired by: Admin  
Sample Name: RAC-BAH-6-172(1)-1.0%IPA-1.00mL-min-ADH  
Sample ID:  
Tray#: 1  
Vial #: 1  
Injection Volume: 1 uL  
Data File Name: RAC-BAH-6-172(1)-1.0%IPA-1.00mL-min-ADH.icm  
Method File Name: Cyclic Urea Method.icm  
Batch File Name:  
Report File Name: Default.icr  
Data Acquired: 3/13/2014 10:42:36 AM  
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PDA Multi 1  

--- Peak Table ---  

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-172(3)-1.0%IPA-1.00mL-min-ADH
Sample ID: 1
Tray #: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-6-172(3)-1.0%IPA-1.00mL-min-ADH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 1/3/2014 11:04:35 AM
Data Processed: 1/3/2014 11:44:24 AM

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PDA Multi 1/254nm 4nm

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==== Shimadzu LCsolution Analysis Report ====  

Acquired by: Admin  
Sample Name: CHIRAL-BAH-6-208(3)-3.00%IPA-1.00mL-min-ADH  
Sample ID:  
Tray #: 1  
Vial #: 1  
Injection Volume: 1 uL  
Data File Name: CHIRAL-BAH-6-208(3)-3.00%IPA-1.00mL-min-ADH.lcd  
Method File Name: Cyclic Urea Method.lcm  
Batch File Name:  
Report File Name: Default.lcr  
Data Acquired: 1/28/2014 6:34:23 PM  
Data Processed: 1/28/2014 7:17:08 PM  

<Chromatogram>
<Chromatogram>
== Shimadzu LCsolution Analysis Report ==

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-195(4)-3.00%IPA-0.250mL_min-luxamyllose
Sample ID: 1
Tray#: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: CHIRAL-BAH-6-195(4)-3.00%IPA-0.250mL_min-luxamyllose.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: Default.lcr
Report File Name: Default.lcr
Data Acquired: 1/31/2014 9:58:24 PM
Data Processed: 1/31/2014 10:33:09 PM

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![Chromatogram Image]

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PDA Multi 1/254nm 1nm
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-7-2(2)-2.00%IPA-1.00mL-min-ADH
Sample ID:
Tray#:
Vial #:
Injection Volume: 1.0uL
Data File Name: RAC-BAH-7-2(2)-2.00%IPA-1.00mL-min-ADH.lcd
Method File Name: Cyclic Urea Method.lcr
Batch File Name:
Report File Name: Default.lcr
Data Acquired: 1/29/2014 3:02:32 PM
Data Processed: 1/29/2014 3:30:05 PM

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-7-9(3)-2.00%IPA-1.00mL_min-ADH
Sample ID:
Tray #: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: CHIRAL-BAH-7-9(3)-2.00%IPA-1.00mL_min-ADH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: Default.lcr
Report File Name: 
Data Acquired: 2/4/2014 12:06:47 PM

<Chromatogram>

1 PDA Multi 1/254nm 4mm
PDA Multi 1

Peak Table

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**** Shimadzu LCsolution Analysis Report ****

Acquired by: Admin
Sample Name: RAC-BAH-6-199(5)-1.00%IPA-1.00ml_min-ADH
Sample ID: 1
Tray #: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-6-199(5)-1.00%IPA-1.00ml_min-ADH.lcd
Method File Name: Cyclic Urea Method.icm
Batch File Name: Default.icr
Report File Name: Default.icr
Data Acquired: 2/26/2014 8:46:21 AM
Data Processed: 2/26/2014 9:36:40 AM

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![Chromatogram Image]

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==== Shimadzu LCsolution Analysis Report ==== 

Acquired by: Admin  
Sample Name: CHIRAL-BAH-7-9(1)-1.00%IPA-1.00ml_min-ADH  
Sample ID: 1  
Tray #: 1  
Vial #: 1  
Injection Volume: 1 uL  
Data File Name: CHIRAL-BAH-7-9(1)-1.00%IPA-1.00ml_min-ADH001.lcd  
Method File Name: Cyclic Urea Method.icm  
Batch File Name: Default.icr  
Report File Name: Default.icr  
Data Acquired: 2/26/2014 5:00:56 PM  
Data Processed: 2/26/2014 5:38:37 PM

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Automated NMR tuning parameters
Sample Name:

Data Collected on:
- Agilent 600 MHz spectrometer
- Sample directory:

File Name: SBB-R-114-1284

Data collected on: Apr 27 2011

4f

![NMR Spectrum](image)

Automated NMR tuning parameters
Sample Name:

Data Collected on:
- Agilent 600 MHz spectrometer
- Sample directory:

File Name: SBB-R-114-1284

Data collected on: Apr 27 2011

![NMR Spectrum](image)
Shimadzu LC solution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-7-114(1)-1.0ml_min-0.50%IPA-ADH
Sample ID: 1
Tray #: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-7-114(1)-1.0ml_min-0.50%IPA-ADH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 4/26/2014 11:28:56 AM

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PDA Multi 1

1 PDA Multi 1/254nm 4nm

Peak Table

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### Shimadzu LCsolution Analysis Report ###

- **Acquired by:** Admin
- **Sample Name:** CHIRAL-BAH-7-114(2)-1.0ml_min-0.50%IPA-ADH
- **Sample ID:** 1
- **Tray #:** 1
- **Vial #:** 1
- **Injection Volume:** 1 μL
- **Data File Name:** RAC-BAH-7-114(2)-1.0ml_min-0.50%IPA-ADH.lcd
- **Method File Name:** Cyclic Urea Method.lcm
- **Report File Name:** Default ICU
- **Data Acquired:** 4/26/2014 11:10:53 AM
- **Data Processed:** 4/26/2014 11:24:11 AM

#### Chromatogram ####

![Chromatogram Image]

### Peak Table ###

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==== Shimadzu LCsolution Analysis Report ====

Acquired by: Admin
Sample Name: RAC-BAH-8-11-A-1.0%IPA-1.0mL.min-ADH
Sample ID: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-8-11-A-1.0%IPA-1.0mL.min-ADH1.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 7/29/2014 10:55:19 AM
Data Processed: 7/29/2014 11:09:34 AM

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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-6-54(I)-5.0%IPA-0.50ml_min-ODH
Sample ID: <SAMPLE>
Tray#: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: RAC-BAH-6-54(I)-5.0%IPA-0.50ml_min-ODH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 9/16/2013 4:31:42 PM
Data Processed: 9/16/2013 4:50:39 PM

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Peak Table

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</table>

S 160
#### Shimadzu LCsolution Analysis Report ####

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-106(2)-5.0%IPA-0.50mL_min-ODH
Sample ID: 
Tray#: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: CHIRAL-BAH-6-106(2)-5.0%IPA-0.50mL_min-ODH
Method File Name: Cyclic Urea Method-JB.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 10/29/2013 12:10:26 PM
Data Processed: 10/29/2013 12:36:41 PM

@ 90 °C

**<Chromatogram>**

![Chromatogram](image)

**PeakTable**

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<tbody>
<tr>
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<td>42747337</td>
<td>1030380</td>
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</tbody>
</table>
== Shimadzu LCsolution Analysis Report ==

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-112(1)-5.00%IPA-0.50ml_min-ODH
Sample ID: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: CHIRAL-BAH-6-112(1)-5.00%IPA-0.50ml_min-ODH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default lcf
Data Processed: 2/25/2014 4:09:19 PM

<Chromatogram>

@ 70 °C

PeakTable

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<th>Area</th>
<th>Height</th>
<th>Area %</th>
<th>Height %</th>
</tr>
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<tbody>
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<td>821378</td>
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<td>274461</td>
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</tbody>
</table>
### Shimadzu LCsolution Analysis Report

- **Acquired by:** Admin
- **Sample Name:** RAC-BAH-6-91(l)-5.0%IPA-1.0mL_min-ODH.lcd
- **Sample ID:** 
- **Tray #:** 1
- **Vial #:** 1
- **Injection Volume:** 1 μL
- **Data File Name:** RAC-BAH-6-91(l)-5.0%IPA-1.0mL_min-ODH.lcd
- **Method File Name:** Cyclic Urea Method-JB.lcm
- **Batch File Name:** 
- **Report File Name:** Default.Icr
- **Data Acquired:** 10/15/2013 1:33:22 PM
- **Data Processed:** 10/15/2013 1:56:19 PM

---

**<Chromatogram>**

![Chromatogram Image]

**PDA Multi**

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<td><strong>Total</strong></td>
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<td><strong>1380873</strong></td>
<td>100.000</td>
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</tbody>
</table>
**** Shimadzu LCsolution Analysis Report ****

Acquired by: Admin
Sample Name: CHIRAL-BAH-691(II)-5.0%IPA-1.0ml_min-ODH
Sample ID: 1
Tray#: 1
Yail #: 1
Injection Volume: 1 μL
Data File Name: CHIRAL-BAH-691(II)-5.0%IPA-1.0ml_min-ODH.lcd
Method File Name: Cyclic Urea Method.JB.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 10/15/2013 1:57:19 PM
Data Processed: 10/15/2013 2:27:52 PM

<Chromatogram>

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</thead>
<tbody>
<tr>
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</tbody>
</table>
\textbf{Shimadzu LCsolution Analysis Report}

Acquired by: Admin
Sample Name: RAC-BAH-6-107(4)-5.0\%IPA-1.0mL_min-ODH
Sample ID: 
Tray #: 1
Vail #: 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-6-107(4)-5.0\%IPA-1.0mL_min-ODH.lcd
Method File Name: Cyclic Urea Method-JB.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 10/29/2013 2:44:32 PM
Data Processed: 10/29/2013 3:06:03 PM

\textbf{Chromatogram}

\begin{center}
\includegraphics[width=\textwidth]{chromatogram.png}
\end{center}

\begin{center}
\textbf{Peak Table}
\begin{tabular}{|c|c|c|c|c|}
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Peak & Ret Time & Area & Height & Area\% & Height\% \\
\hline
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2 & 8.152 & 30059949 & 1179440 & 31.737 & 42.223 \\
\hline
Total & & 58100760 & 2793344 & 100.000 & 100.000 \\
\hline
\end{tabular}
\end{center}
==== Shimadzu LCsolution Analysis Report ==== 

Acquired by : Admin
Sample Name  : CHIRAL-BAH-6-107(3)-5.0%IPA-1.0mL_min-ODH
Sample ID    : 
Tray #       : 1
Vial #       : 1
Injection Volume : 1.0uL
Data File Name : CHIRAL-BAH-6-107(3)-5.0%IPA-1.0mL_min-ODH.lcd
Method File Name : Cyclic Urea Method-J5.lcm
Batch File Name : Default.lcr
Report File Name : 
Data Acquired : 10/28/2013 2:22:07 PM
Data Processed : 10/28/2013 3:32:09 PM

<Chromatogram>

![Chromatogram Image]

mAU

<table>
<thead>
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<th>Height</th>
<th>Area %</th>
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</thead>
<tbody>
<tr>
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</table>
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-6-67-paraCF3-5.0%IPA-1.0mL_min-ODH
Sample ID:
Tray#: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-6-67-paraCF3-5.0%IPA-1.0mL_min-ODH.idc
Method File Name: Cyclic Urea Method-JB.idc
Batch File Name: 
Report File Name: Default.idc
Data Acquired: 10/25/2013 11:21:59 AM
Data Processed: 10/25/2013 12:31:16 PM

<Chromatogram>
Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: CHIRAL-BAH-6-108-2-5.0%IPA-1.00ml_min-ODH
Sample ID: 
Tray #: 1
Vial #: 1
Injection Volume: 1 µL
Data File Name: CHIRAL-BAH-6-108-2-5.0%IPA-1.00ml_min-ODH.lcd
Method File Name: Cyclic Urea Method.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 10/30/2013 11:25:48 AM
Data Processed: 10/30/2013 11:45:19 AM

<Chromatogram>

Peak Table

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<th>Height</th>
<th>Area %</th>
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</thead>
<tbody>
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<td>247088</td>
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Shimadzu LCsolution Analysis Report

Acquired by: Admin
Sample Name: RAC-BAH-6-107-(II)-1.0%IPA-1.0mL_min-ODH
Sample ID: 1
Tray#: 1
Vial #: 1
Injection Volume: 1 uL
Data File Name: RAC-BAH-6-107-(II)-1.0%IPA-1.0mL_min-ODH.lcd
Method File Name: Cyclic Urea Method-JB.lcm
Batch File Name: 
Report File Name: Default.lcr
Data Acquired: 10/25/2013 2:12:44 PM
Data Processed: 10/25/2013 3:22:45 PM

<Chromatogram>

Peak Table

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</table>
==== Shimadzu LCsolution Analysis Report ====  

Acquired by: Admin  
Sample Name: CHIRAL-BAH-7-14(2)-1.00%IPA-1.00mL_min-ADH  
Sample ID:  
Tray #: 1  
Val #: 1  
Injection Volume: 1 ul  
Data File Name: CHIRAL-BAH-7-14(2)-1.00%IPA-1.00mL_min-ADH.lcd  
Method File Name: Cyclic Urea Method.icm  
Batch File Name: Default.lcr  
Report File Name:  
Data Acquired: 2/5/2014 1:31:16 PM  
Data Processed: 2/6/2014 1:58:24 PM  

<Chromatogram>