Electronic Supplementary Information

Chemoselective Reductive Alkynylation of Tertiary Amides by Ir and Cu(I) Bis-metal Sequential Catalysis

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Table 1. The structures of the amides used

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1m: P = Boc
1n: P = Cbz
Table 2. The structures of the alkynes used

\[ \begin{align*}
2a & \quad \begin{array}{c}
\text{Ph} \\
\equiv \\
H
\end{array} \\
2b & \quad \begin{array}{c}
\text{EtO} \\
\equiv \\
\text{EtO}
\end{array} \\
2c & \quad \begin{array}{c}
\text{(Me}_3\text{Si)} \\
\equiv \\
H
\end{array} \\
2d & \quad \begin{array}{c}
\text{MeO}_2\text{C} \\
\equiv \\
H
\end{array} \\
2e & \quad \begin{array}{c}
\text{H} \\
\equiv \\
\text{O}
\end{array}
\end{align*} \]
**In situ** $^1$H NMR Spectra of the reaction

$^1$H NMR spectra (400 MHz) of starting material, intermediates in D$_8$-toluene. (A) 1v in D$_8$-toluene; (B) 1v and (Me$_2$HSi)$_2$O in D$_8$-toluene; (C) 1v, (Me$_2$HSi)$_2$O (2.0 equiv), and [IrCl(CO)(PPh$_3$)$_2$] (1 mol %) in D$_8$-toluene, 10 min.

$^1$H NMR spectra (400 MHz) of starting material, intermediates in D$_8$-toluene. (A’) 1b in D$_8$-toluene; (B’) 1b and (Me$_2$HSi)$_2$O in D$_8$-toluene; (C’) 1b, (Me$_2$HSi)$_2$O (1.2 equiv), and [IrCl(CO)(PPh$_3$)$_2$] (1 mol %) in D$_8$-toluene, 30 min.
**Experimental Procedures**

**General Methods.** Melting points were determined on a Büchi M560 Automatic Melting Point apparatus and are uncorrected. Infrared spectra were measured with a Nicolet Avatar 360 FT-IR spectrometer using film KBr pellet techniques. NMRSpectra were recorded on a Bruker AV 400 or AC 500 spectrometer at 25 °C in the solvents indicated. Chemical shifts (δ) are reported in ppm and respectively referenced to internal standard Me₄Si and solvent signals (Me₄Si, 0 ppm for ¹H NMR and CDCl₃, 77.0 ppm for ¹³C NMR). Mass spectra were recorded on a Bruker Dalton ESquire 3000 plus LC-MS apparatus (ESI direct injection). HRMS spectra were recorded on a 7.0T FT-MS apparatus. Silica gel (300-400 mesh) was used for flash column chromatography, eluting (unless otherwise stated) with EtOAc/ n-hexane mixture. Toluene were distilled over sodium benzophenone ketyl under N₂.

**N,N-Dimethyl-1,3-diphenylprop-2-yn-1-amine (3a)¹¹**

Following the general procedure, the reaction of tert-amide 1a (149 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), the known propargylic amine 3a¹¹(209 mg, yield: 89%) as a pale yellow oil; IR (film)νmax: 2939, 1597, 1488, 1450, 1325, 1021, 755, 694cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 2.32 (s, 6H), 4.82 (s, 1H), 7.27-7.38 (m, 6H), 7.51-7.53 (m, 2H), 7.60-7.62 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 41.6 (2C), 62.2, 84.8, 88.3, 123.2, 127.6, 128.1 (2C), 128.2 (2C), 128.3 (2C), 128.4, 131.8, 138.7 ppm; MS (ESI) m/z 236 (M+H⁺).

**N,N-Dimethyl-3-phenyl-1-(p-tolyl)prop-2-yn-1-amine (3b)**

Following the general procedure, the reaction of tert-amide 1a (149 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), the known propargylic amine 3a¹¹(209 mg, yield: 89%) as a pale yellow oil; IR (film)νmax: 2939, 1597, 1488, 1450, 1325, 1021, 755, 694cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 2.32 (s, 6H), 4.82 (s, 1H), 7.27-7.38 (m, 6H), 7.51-7.53 (m, 2H), 7.60-7.62 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 41.6 (2C), 62.2, 84.8, 88.3, 123.2, 127.6, 128.1 (2C), 128.2 (2C), 128.3 (2C), 128.4, 131.8, 138.7 ppm; MS (ESI) m/z 236 (M+H⁺).
Following the general procedure, the reaction of tert-amide 1b (163 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (elucent: EtOAc/n-hexane = 1: 100), the propargylic amine 3b (224 mg, yield: 90%) as a pale yellow oil; IR (film) \( \nu_{\text{max}} \): 2939, 1597, 1488, 1450, 1325, 1021, 755, 694 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \): 2.31 (s, 6H), 2.35 (s, 3H), 4.78 (s, 1H), 7.17 (d, \( J = 7.8 \) Hz, 2H), 7.31-7.33 (m, 3H), 7.47-7.53 (m, 4H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \): 21.1, 41.6 (2C), 61.9, 85.1, 88.1, 123.2, 128.1, 128.2 (2C), 128.3 (2C), 128.9 (2C), 131.8 (2C), 135.7, 137.3 ppm; MS (ESI) \( m/z \) 250 (M+H\(^+\)); HRMS (ESI) \( m/z \) calcd for [C\(_{18}\)H\(_{20}\)N]\(^+\)(M + H\(^+\)): 250.1590; found: 250.1591.

\( N,N\)-Dibenzyl-4,4-dimethyl-1-phenylpent-1-yn-3-amine (3c)

Following the general procedure, the reaction of tertiary amide 1c (281 mg, 1.0 mmol) with phenylacetylene 2b (0.13 mL, 1.2 mmol) gave, after FC (elucent: EtOAc/n-hexane = 1: 100), propargylic amine 3c (220 mg, yield: 60%) as a pale yellow oil; IR (film) \( \nu_{\text{max}} \): 2955, 1601, 1495, 1453, 1357, 1258, 1030, 800, 749, 691 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \): 0.97 (s, 9H), 3.37 (s, 1H), 3.45 (d, \( J = 14.0 \) Hz, 2H), 3.95 (d, \( J = 14.0 \) Hz, 2H), 7.22-7.25 (m, 2H), 7.30-7.35 (m, 7H), 7.42-7.44 (m, 4H), 7.50-7.52 (m, 2H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \): 27.9 (3C), 36.2, 57.9 (2C), 62.2, 86.6, 86.7, 123.8, 126.9, 127.8 (2C), 128.0 (2C), 128.1 (4C), 128.3 (4C), 129.1 (2C), 131.8, 140.0 ppm; MS (ESI) \( m/z \)368 (M+H\(^+\)); HRMS (ESI) \( m/z \) calcd for [C\(_{27}\)H\(_{30}\)N]\(^+\)(M + H\(^+\)): 368.2373; found: 368.2376.

1-((3\(r\),5\(r\),7\(r\))-Adamantan-1-yl)-N,\(N\)-dimethyl-3-phenylprop-2-yn-1-amine (3d)
Following the general procedure, the reaction of tert-amide 1d (207 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 80), propargylic amine 3d (234 mg, yield: 80%) as a pale yellow oil; IR (film) $\nu_{\text{max}}$: 2901, 1492, 1447, 1341, 1021, 752, 688 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.63-1.78 (m, 12H), 2.00 (s, 3H), 2.36 (s, 6H), 3.04 (s, 1H), 7.28-7.33 (m, 3H), 7.45-7.47 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 28.7, 37.2 (2C), 38.2 (3C), 39.9 (3C), 45.2 (3C), 69.3, 85.1, 88.1, 123.8, 127.7 (2C), 128.2 (2C), 131.7 ppm; MS (ESI) m/z 294 (M+H$^+$); HRMS (ESI) m/z calcd for [C$_{21}$H$_{28}$N]$^+$ (M + H$^+$): 294.2216; found: 294.2216.

$N,N$-Diisopropyl-1,3-diphenylprop-2-yn-1-amine (3e)$^{[2]}$

Following the general procedure, the reaction of tert-amide 1e (205 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), the known propargylic amine 3e$^{[2]}$ (195 mg, yield: 67%) as a pale yellow oil; IR (film) $\nu_{\text{max}}$: 2952, 1601, 1492, 1447, 1380, 1181, 758, 710, 685 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.05 (d, $J = 6.7$ Hz, 3H), 1.29 (d, $J = 6.7$ Hz, 3H), 3.16-3.26 (m, 2H), 5.02 (s, 1H), 7.22-7.35 (m, 6H), 7.46-7.49 (m, 2H), 7.73-7.75 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 20.7 (2C), 23.8 (2C), 46.6 (2C), 50.5, 85.9, 91.6, 123.9, 126.8, 127.8 (2C), 127.9 (4C), 128.3 (2C), 131.3, 142.2 ppm; MS (ESI) m/z 292 (M+H$^+$).

$N$-Allyl-$N$-(1,3-diphenylprop-2-yn-1-yl)prop-2-en-1-amine (3f)$^{[3]}$

Following the general procedure, the reaction of tert-amide 1f (201 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), the known propargylic amine 3f$^{[3]}$ (244 mg, yield: 85%) as a pale yellow oil; IR (film) $\nu_{\text{max}}$: 3077, 2923, 2814, 1642, 1594, 1485, 1444, 1267, 1114,
992, 970, 922, 755, 694 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 3.02-3.08 (m, 2H), 3.26-3.29 (m, 2H), 5.10-5.14 (m, 3H), 5.25-5.29 (m, 2H), 5.81-5.91 (m, 2H), 7.24-7.37 (m, 6H), 7.52-7.53 (m, 2H), 7.67-7.70 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 53.5 (2C), 56.6, 117.3 (2C), 123.3, 127.3 (2C), 127.4 (4C), 128.1 (2C), 128.3 (2C), 131.8 (2C), 136.5 (2C), 139.3 ppm; MS (ESI) $m/z$ 288 (M+H$^+$).

$N$-Benzyl-4,4-diethoxy-$N$-methyl-1-phenylbut-2-yn-1-amine (3g)

Following the general procedure, the reaction of $tert$-amide 1g (225 mg, 1.0 mmol) with 3,3-diethoxyprop-1-yne 2b (0.17 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), propargylic amine 3g (307 mg, yield: 91%) as a pale yellow oil; IR (film) $\nu_{\text{max}}$: 2978, 1450, 1328, 1053, 1011, 736, 694 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.29 (t, $J$ = 7.0 Hz, 6H), 2.18 (s, 3H), 3.55 (d, $J$ = 13.2 Hz, 1H), 3.65-3.73 (m, 2H), 3.81-3.90 (m, 2H), 4.78 (s, 1H), 5.48 (d, $J$ = 1.1 Hz, 1H), 7.24-7.38 (m, 8H), 7.59-7.61 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 15.2 (2C), 38.0, 59.1, 60.8, 60.9, 80.6, 84.0, 91.5, 127.1, 127.5, 128.1 (2C), 128.2 (2C), 128.3 (2C), 138.4, 139.1 ppm; MS (ESI) $m/z$ 338 (M+H$^+$); HRMS (ESI) $m/z$ calcd for [C$_{22}$H$_{28}$NO$_2$]$^+(M + H^+)$: 338.2115; found: 338.2115.

1-(Furan-2-yl)-$N,N$-dimethyl-3-phenylprop-2-yn-1-amine (3h)

Following the general procedure, the reaction of $tert$-amide 1h (139 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 90), propargylic amine 3h (180 mg, yield: 80%) as a pale yellow oil; IR (film) $\nu_{\text{max}}$: 2936, 1700, 1594, 1485, 1181, 1072, 758, 691 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 2.35 (s, 6H), 4.89 (s, 1H), 6.35 (dd, $J$ = 1.9, 3.2 Hz, 1H), 6.49-6.50 (m, 1H), 7.31-7.34 (m, 3H), 7.42-7.43 (m, 1H), 7.49-7.51 (m, 2H) ppm; $^{13}$C NMR (100
MHz, CDCl3): δ 41.3 (2C), 56.2, 82.6, 86.2, 108.9, 109.9, 122.7, 128.2 (2C), 128.3 (2C), 131.8, 142.6, 151.9 ppm; MS (ESI) m/z 226 (M+H+); HRMS (ESI) m/z calcd for [C15H16NO]+(M + H+): 226.1226; found: 226.1228.

1-(Benzo[b]thiophen-2-yl)-N, N-dimethyl-3-phenylprop-2-yn-1-amine (3i)

Following the general procedure, the reaction of tert-amide 1i (205 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 80), propargylic amine 3i (236 mg, yield: 81%) as a white solid; Mp70-72 °C; IR (film) νmax: 2938, 1715, 1590, 1491, 1180, 1135, 1052, 768, 698 cm⁻¹. ¹H NMR (400 MHz, CDCl3): δ 2.40 (s, 6H), 5.08 (d, J = 1.1Hz, 1H), 7.27-7.36 (m, 5H), 7.49 (s, 1H), 7.55-7.57 (m, 2H), 7.71-7.80 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl3): δ 41.6 (2C), 58.7, 83.4, 87.9, 122.3, 122.6, 122.8, 123.5 (2C), 124.1 (2C), 128.3, 128.4, 131.9, 139.3, 140.3, 145.1 ppm; MS (ESI) m/z 292 (M+H+); HRMS (ESI) m/z calcd for [C19H18NS]+(M + H+): 292.1154; found: 292.1156.

N, N-Dimethyl-1-(4-nitrophenyl)-3-phenylprop-2-yn-1-amine (3j)

Following the general procedure, the reaction of tert-amide 1j (194 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 20), propargylic amine 3j (216 mg, yield: 77%) as a pale yellow oil; IR (film) νmax: 2952, 1745, 1488, 1203, 758, 691 cm⁻¹. ¹H NMR (400 MHz, CDCl3): δ 2.32 (s, 6H), 4.90 (s, 1H), 7.34-7.38 (m, 3H), 7.51-7.56 (m, 2H), 7.81-7.85 (m, 2H), 8.21-8.24 (m, 2H) ppm; ¹³C NMR (100 MHz, CDCl3): δ 41.7 (2C), 58.7, 83.0, 89.5, 122.5, 123.4, 128.4 (2C), 128.6 (2C), 129.2 (2C), 131.8 (2C), 146.3, 147.5 ppm; MS
(ESI) m/z 281 (M+H+); HRMS (ESI) m/z calcd for [C_{17}H_{17}N_{2}O_{2}]^+(M + H+): 281.1285; found: 281.1280.

2-(1-(Dimethylamino)-3-phenylprop-2-yn-1-yl)benzonitrile (3k)

Following the general procedure, the reaction of tert-amide 1k (174 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 40), propargylic amine 3k (208 mg, yield: 80%) as a pale yellow oil; IR (film) $\tilde{\nu}_{\text{max}}$: 2944, 2224, 1705, 1597, 1320, 1017, 758, 691 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 2.35 (s, 6H), 5.09 (s, 1H), 7.35-7.42 (m, 4H), 7.53-7.61 (m, 3H), 7.69-7.71 (m, 1H), 7.84-7.86 (m, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 41.5 (2C), 60.5, 82.7, 89.4, 113.3, 117.6, 122.6, 128.1, 128.4 (2C), 128.5 (2C), 129.1, 131.8, 132.1, 133.4, 143.0 ppm; MS (ESI) m/z 261 (M+H$^+$).

4-(1-(Dimethylamino)-3-phenylprop-2-yn-1-yl)benzaldehyde (3l)

Following the general procedure, the reaction of tert-amide 1l (354 mg, 2.0 mmol) with phenylacetylene 2a (0.26 mL, 2.4 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 70; 1: 5), propargylic amine 3l (336 mg, yield: 64%), and propargylic amine 3l’ (42 mg, yield: 8%).

3l: pale yellow oil; IR (film) $\tilde{\nu}_{\text{max}}$: 2943, 2824, 1703, 1607, 1492, 1021, 790, 758, 688, 598 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 2.33 (s, 6H), 4.88 (s, 1H), 7.34-7.36 (m, 3H), 7.52-7.55 (m, 2H), 7.80-7.90 (m, 4H), 10.0 (s, 1H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 41.6 (2C), 62.0, 83.6, 89.1, 122.7, 128.3, 128.4 (2C), 129.0 (2C), 129.6 (2C), 131.8 (2C), 135.8, 145.7, 191.9 ppm; MS (ESI) m/z 264 (M+H$^+$); HRMS (ESI) m/z calcd for [C$_{18}$H$_{18}$NO]$^+$(M + H$^+$): 264.1383; found: 264.1384.
(4-(1-(Dimethylamino)-3-phenylprop-2-yn-1-yl)phenyl)methanol (3l’): pale yellow oil; IR (film) $\nu_{\text{max}}$: 3240, 2936, 2858, 2773, 1485, 752, 688 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 2.18 (s, 1H), 2.30 (s, 6H), 4.68 (s, 2H), 4.80 (s, 1H), 7.32-7.36 (m, 5H), 7.50-7.51 (m, 2H), 7.58-7.60 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 41.5 (2C), 62.0, 65.0, 84.7, 88.4, 123.1, 126.8, 128.2 (2C), 128.3 (2C), 128.7 (2C), 131.8 (2C), 138.0, 140.5 ppm; MS (ESI) $m/z$ 266 (M$^+$H$^+$). The structure of this side-product was confirmed by HMBC spectrum (cf. ESI p.44).

**tert-Butyl 4-(1,3-diphenylprop-2-yn-1-yl)piperazine-1-carboxylate (3m)**

Following the general procedure, the reaction of *tert*-amide 1m (290 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), propargylic amine 3m (346 mg, yield: 92%) as a white solid; Mp 86-87 °C; IR (film) $\nu_{\text{max}}$: 2935, 2807, 1702, 1496, 1363, 1167, 1044, 756, 692 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.45 (s, 9H), 2.56-2.58 (m, 4H), 3.40-3.49 (m, 4H), 4.84 (s, 1H), 7.29-7.39 (m, 6H), 7.49-7.51 (m, 2H), 7.62-7.64 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 28.4 (3C), 43.6 (2C), 49.2 (2C), 61.8, 79.5, 84.9, 88.5, 122.9, 127.7 (2C), 128.2 (2C), 128.3 (2C), 128.5 (2C), 131.8 (2C), 137.9, 154.8 ppm; MS (ESI) $m/z$ 377 (M$^+$H$^+$); HRMS (ESI) $m/z$ calcd for [C$_{24}$H$_{29}$N$_2$O$_2$]$^+$ (M$^+$H$^+$): 377.2224; found: 377.2226.

**Benzyl 4-(1,3-diphenylprop-2-yn-1-yl)piperazine-1-carboxylate (3n)**

Following the general procedure, the reaction of *tert*-amide 1n (324mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), propargylic amine 3n (390 mg, yield: 95%) as a white solid; Mp
111-113 °C; IR (film) $\nu_{\text{max}}$: 2933, 1703, 1597, 1239, 1178, 1072, 1021, 755, 694 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 2.59 (br s, 4H), 3.48-3.58 (m, 4H), 4.85 (s, 1H), 5.12 (s, 2H), 7.28-7.39 (m, 11H), 7.48-7.51 (m, 2H), 7.62-7.63 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 43.9 (2C), 49.1 (2C), 61.7, 67.0, 84.6, 88.6, 122.8, 127.8, 127.9 (2C), 128.2 (4C), 128.3 (4C), 128.4 (2C), 131.8 (2C), 136.7, 137.7, 155.2 ppm; MS (ESI) $m/z$ 411 (M+H$^+$); HRMS (ESI) $m/z$ calcd for [C$_{27}$H$_{27}$N$_2$O$_2$]$^+(M + H^+)$: 411.2067; found: 411.2070.

**tert-Butyl (1-(1,3-diphenylprop-2-yn-1-yl)piperidin-4-yl)carbamate (3o)**

Following the general procedure, the reaction of tert-amide 1o (304 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), propargylic amine 3o (347 mg, yield: 89%) as a white solid; Mp 132-134 °C; IR (film) $\nu_{\text{max}}$: 3401, 2954, 1697, 1418, 1248, 1181, 1130, 1072, 758, 694 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.32-1.56 (m, 11H), 1.86-2.00 (m, 2H), 2.30-2.35 (m, 1H), 2.59-2.66 (m, 2H), 2.89-2.92 (m, 1H), 3.48 (br s, 1H), 4.43 (s, 1H), 4.82 (s, 1H), 7.27-7.37 (m, 6H), 7.48-7.51 (m, 2H), 7.60-7.62 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 28.4 (3C), 32.6, 33.0, 46.2, 47.9, 50.8, 61.7, 79.1, 85.3, 88.2, 123.0, 127.6 (2C), 128.1 (2C), 128.3 (2C), 128.4 (2C), 131.7 (2C), 138.4, 155.1 ppm; MS (ESI) $m/z$391 (M+H$^+$); HRMS (ESI) $m/z$ calcd for [C$_{25}$H$_{31}$N$_2$O$_2$]$^+(M + H^+)$: 391.2380; found: 391.2383.

**1-(1,3-Diphenylprop-2-yn-1-yl)-4-((1,1,3,3-tetramethyldisiloxanyl)oxy)piperidine (3p)**

![Diagram of 1-(1,3-Diphenylprop-2-yn-1-yl)-4-((1,1,3,3-tetramethyldisiloxanyl)oxy)piperidine (3p)](image)
Following the general procedure, the reaction of tert-amide 1p (205 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 40), propargylic amine 3p (215 mg, yield: 52%) as a pale yellow oil; IR (film) $\nu_{\text{max}}$: 2952, 1097, 1485, 1258, 1069, 909, 797, 758, 694 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 0.09 (s, 6H), 0.17 (s, 3H), 0.18 (s, 3H), 1.54-1.86 (m, 4H), 2.28-2.33 (m, 1H), 2.52-2.58 (m, 1H), 2.68-2.70 (m, 1H), 2.89-2.92 (m, 1H), 3.75-3.80 (m, 1H), 4.68-4.72 (m, 1H), 4.83 (s, 1H), 7.29-7.37 (m, 6H), 7.49-7.53 (m, 2H), 7.63-7.64 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ −0.5 (2C), 0.7 (2C), 34.8, 35.2, 45.9 (2C), 61.7, 68.6, 85.8, 88.0, 123.3, 127.5 (2C), 128.1 (2C), 128.3 (2C), 128.4 (2C), 131.8 (2C), 138.7 ppm; MS (ESI) $m/z$ 414 (M$^+$H$^+$); HRMS (ESI) $m/z$ calcld for [C$_{24}$H$_{34}$NO$_2$Si$_2$]$^+$(M + H$^+$): 414.2123; found: 414.2124.

**Methyl (S,S)-1-(1, 3-diphenylprop-2-yn-1-yl)pyrrolidine-2-carboxylate (3q)**

Following the general procedure, the reaction of tert-amide 1q (233 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave a diastereomeric mixture ($dr = 20:1$, determined by $^1$H NMR of the crude product), after FC (eluent: EtOAc/n-hexane = 1: 80), the major diastereomeric propargylic amine 3q$^{[4]}$ (239 mg, yield: 75%) as a pale yellow oil; [\(\alpha\)]$^D_{26}$ $-$107.8 (c 1.8, CHCl$_3$); IR (film) $\nu_{\text{max}}$: 2949, 1745, 1488, 1447, 1271, 1200, 758, 697 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.76-1.85 (m, 2H), 2.01-2.03 (m, 2H), 2.67-2.77 (m, 2H), 3.77 (dd, $J = 8.9, 7.1$ Hz, 1H) superposed with 3.77 (s, 3H), 5.24 (s, 1H), 7.27-7.38 (m, 6H), 7.50-7.52 (m, 2H), 7.67-7.69 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 23.2, 29.3, 47.4, 51.9, 57.3, 63.1, 85.2, 87.9, 123.0, 127.6 (2C), 128.2 (3C), 128.3 (3C), 131.8 (2C), 138.9, 174.5 ppm; MS (ESI) $m/z$ 320 (M+H$^+$).

**Methyl (S,S)-1-(1’-phenyl-3’-(trimethylsilyl)prop-2-yn-1’-yl)pyrrolidine-2-carboxylate (3r)**
Following the general procedure, the reaction of tert-amide 1q (233 mg, 1.0 mmol) with ethynyltrimethylsilane 2c (0.17 mL, 1.2 mmol) gave a diastereomeric mixture \((dr = 17:1, \text{determined by }^1\text{H NMR of the crude product}), \text{after FC (eluent: EtOAc/n-hexane }= 1:100), \text{the major diastereomeric propargylic amine }3r \text{ (246 mg, yield: 78\%) as a pale yellow oil; }[^\alpha]D_{26} -101.3 \text{ (c 1.6, CHCl}_3); \text{IR (film) }\nu_{\text{max}}: 2955, 1745, 1450, 1197, 1127, 995, 851, 758 \text{cm}^{-1}. \text{^1H NMR (400 MHz, CDCl}_3): \delta 0.23 \text{ (s, 9H), 1.72-1.82 (m, 2H), 1.98-2.19 (m, 2H), 2.59-2.64 (m, 2H), 3.67 (dd, } J = 9.0, 6.8 \text{ Hz, 1H) superposed with 3.76 (s, 3H), 5.01 (s, 1H), 7.24-7.35 \text{ (m, 3H), 7.59-7.61 (m, 2H) ppm; }^{13}\text{C NMR (100 MHz, CDCl}_3): \delta 0.2 (3C), 23.2, 29.3, 47.1, 51.8, 57.4, 63.0, 92.4, 101.5, 127.5, 128.1 (2C), 128.2 (2C), 138.6, 174.5 \text{ ppm; MS (ESI) } m/ z \text{ 316 (M+H\textsuperscript{+}); HRMS (ESI) } m/z \text{ calcd for [C}_{18}H_{26}NO_2Si]^+(M + H\textsuperscript{+}): 316.1727; \text{found: 316.1726.}

Methyl (S,S)-1-(1’-(4’’-bromophenyl)-3’-phenylprop-2-yn-1’-yl)pyrrolidine-2-carboxylate (3s)

Following the general procedure, the reaction of tert-amide 1r (311 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave a mixture \((dr = 15:1, \text{determined by }^1\text{H NMR of the crude product}), \text{after FC (eluent: EtOAc/n-hexane }= 1:100), \text{the major diastereomeric propargylic amine }3s \text{ (314 mg, yield: 79\%) as a pale yellow oil; }[^\alpha]D_{26} -85.5 \text{ (c 2, CHCl}_3); \text{IR (film) }\nu_{\text{max}}: 2949, 1738, 1488, 1264, 1203, 761, 691 \text{ cm}^{-1}. \text{^1H NMR (400 MHz, CDCl}_3): \delta 1.76-1.85 \text{ (m, 2H), 2.00-2.24 (m, 2H), 2.63-2.73 (m, 2H), 3.75 (dd, } J = 9.0, 7.1 \text{ Hz, 1H) superposed with 3.77 (s, 3H), 5.20 (s, 1H), 7.33-7.35 \text{ (m, 3H), 7.47-7.58 (m, 6H) ppm; }^{13}\text{C NMR (100 MHz, CDCl}_3): \delta 23.2, 29.3, 47.2, 51.9, 56.6, 63.0, 84.5, 88.3, 121.5, 122.7, 128.3 (2C), 128.4 (2C), 129.9 (2C), 131.3 (2C), 131.8, 138.1, 174.4 \text{ ppm; MS (ESI) } m/z \text{ 398 (M+H\textsuperscript{+}); HRMS (ESI) } m/z \text{ calcd for [C}_{21}H_{21}BrNO_2]^+(M + H\textsuperscript{+}): 398.0750; \text{found: 398.0751.}
Methyl (S,S)-1-(1’-(4’’-cyanophenyl)-3’-phenylprop-2-yn-1’-yl)pyrrolidine-2-carboxylate (3t)

Following the general procedure, the reaction of tert-amide 1s (258 mg, 1.0 mmol) with phenylacetylene 2a (0.13 mL, 1.2 mmol) gave a diastereomeric mixture (dr = 17:1, determined by $^1$H NMR from crude product), after FC (eluent: EtOAc/n-hexane = 1: 50), the major diastereomeric propargylic amine 3t (293 mg, yield: 85%) as a pale yellow oil; [α]$_D^{26}$ –103.3 (c = 2, CHCl$_3$); IR (film) $\nu_{\text{max}}$: 2949, 2215, 1741, 1607, 1457, 1200, 1133, 758, 691 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 1.78-1.85 (m, 2H), 2.00-2.26 (m, 2H), 2.57-2.75 (m, 2H), 3.77 $\text{dd, } J = 9.1, 7.1 \text{ Hz, } 1\text{H}$ superposed with 3.79 (s, 3H), 5.30 (s, 1H), 7.35-7.37 (m, 3H), 7.50-7.53 (m, 2H), 7.65-7.67 (m, 2H), 7.83-7.85 (m, 2H) ppm; $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 23.2, 29.2, 47.1, 51.9, 56.8, 63.0, 83.5, 88.9, 111.4, 118.8, 122.3, 128.4 (2C), 128.6 (2C), 128.9 (2C), 131.8 (2C), 132.0, 144.5, 174.2 ppm; MS (ESI) $m/z$ 345 (M+H$^+$); HRMS (ESI) $m/z$ calcd for [C$_{22}$H$_{21}$N$_2$O$_2$]$^+(M+H^+)$: 345.1598; found: 345.1597.

N-Benzyl-1,1-diethoxy-N-methyloctadec-2-yn-4-amine (3u)

Following the general procedure (except the 2.0 equiv TMDS was used), the reaction of tert-amide 1t (1380 mg, 4 mmol) with 3,3-diethoxyprop-1-yne 2b (0.68 mL, 4.8 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), propargylic amine 3s (1716 mg, yield: 94%) as a colourless oil; IR (film) $\nu_{\text{max}}$: 2930, 2856, 1604, 1460, 1325, 1133, 1053, 701 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 0.88 (t, $J = 6.8$ Hz, 3H), 1.24-1.28 (m, 28H), 1.37-1.45 (m, 2H), 1.63-1.70 (m, 2H), 2.20 (s, 3H), 3.42-3.46 (m, 2H), 4.53-4.55 (m, 2H), 4.88-4.90 (m, 2H), 5.30-5.32 (s, 3H), 7.35-7.37 (m, 3H), 7.50-7.53 (m, 2H), 7.65-7.67 (m, 2H), 7.83-7.85 (m, 2H) ppm; MS (ESI) $m/z$ 477 (M+H$^+$); HRMS (ESI) $m/z$ calcd for [C$_{35}$H$_{47}$N$_2$O$_2$]$^+(M+H^+)$: 477.3554; found: 477.3550.
3.59-3.68 (m, 3H), 3.75-3.83 (m, 2H), 3.61 (d, J = 1.2 Hz, 1H), 7.21-7.34 (m, 5H) ppm; ¹³C NMR (100 MHz, CDCl₃, some peaks overlapped): δ 14.1, 15.1, 22.7, 26.4, 29.2, 29.3, 29.5, 29.6, 29.7, 31.9, 33.6, 37.7, 55.4, 59.1, 60.7, 80.9, 83.5, 91.5, 126.9, 128.2, 128.9, 139.3 ppm; MS (ESI) m/z 458 (M+H⁺); HRMS (ESI) m/z calcd for [C₃₀H₅₂NO₂]⁺(M + H⁺): 458.3993; found: 458.3996.

1-Methyl-2-tetradecylpyrrolidine (4)[⁵]

A suspension of 3u (457 mg, 1.0 mmol) and 10% Pd/C (45 mg) in MeOH (10 mL) containing concentrated HCl (0.3 mL) was stirred under a hydrogen atmosphere (1 atm, ballon) at RT for 24 h. The catalyst was filtered off, and the filtrate was concentrated under reduced pressure. The residue was purified by flash column chromatography (FC) on silica gel (eluent: DCM/MeOH = 20: 1). The fractions were collected, and concentrated. To the residue were added CH₂Cl₂ (5 mL) and Et₃N (1 mL), and the mixture was stirred for 2 h at RT before treating with a saturated aqueous Na₂CO₃ solution. The resulting mixture was extracted with CH₂Cl₂ (3 × 5 mL). The combined organic layers were washed with brine, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to afford (±)-bgugaine (4) [⁴] (267 mg, yield: 95%) as a pale yellow oil. IR (film) νmax: 2901, 2843, 2360, 1488, 1447, 1027, 749, 688 cm⁻¹. ¹H NMR (400 MHz, CDCl₃): δ 0.88 (t, J = 7.0 Hz, 3H), 1.18-1.33 (m, 25H), 1.38-1.48 (m, H), 1.64-1.70 (m, 2H), 1.71-1.81 (m, 1H), 1.88-1.99 (m, 2H), 2.11 (dd, J = 17.8, 9.4 Hz, 1H), 2.30 (s, 3H), 3.03-3.08 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃,some peaks overlapped): δ 14.1, 21.8, 22.6, 26.7, 29.3, 29.6, 29.7, 30.0, 31.9, 33.8, 40.4, 57.3, 66.4 ppm; MS (ESI) m/z 282 (M+H⁺).

Methyl 4-(dibenzylamino)-6-methylhept-2-ynoate (3v)
Following the general procedure (except the 2.0 equiv TMDS was used), the reaction of tert-amide 1u (1405 mg, 5 mmol) with methyl propiolate 2d (0.55 mL, 6.0 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), propargylic amine 3v (1535 mg, yield: 88%) as a pale yellow oil; IR (film) \( \nu_{\text{max}} \): 2955, 2222, 1716, 1450, 1072, 749, 694 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)): \( \delta \) 0.63 (d, \( J = 6.6 \) Hz, 3H), 0.78 (d, \( J = 6.6 \) Hz, 3H), 1.45-1.52 (m, 1H), 1.66-1.73 (m, 1H), 1.80-1.90 (m, 1H), 3.39 (d, \( J = 13.7 \) Hz, 2H), 3.60 (t, \( J = 7.6 \) Hz, 1H), 3.81 (s, 3H), 3.85 (d, \( J = 13.7 \) Hz, 2H), 7.21-7.37 (m, 10H) ppm; \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 21.6 (2C), 22.7, 24.4, 41.9, 49.6, 52.7, 54.9, 76.9, 87.5, 127.1, 128.2 (2C), 128.3 (4C), 128.8 (4C), 139.0, 154.1 ppm; MS (ESI) \( m/z \) 350 (M+H\(^+\)); HRMS (ESI) \( m/z \) calcd for [C\(_{23}\)H\(_{28}\)NO\(_2\)]\(^+(\text{M + H}^+\))\: 350.2115; found: 350.2112.

**4-(3-(Dibenzylamino)pent-1-yn-1-yl)benzaldehyde (3w)**

Following the general procedure (except the 2.0 equiv TMDS was used), the reaction of tert-amide 1v (253 mg, 1.0 mmol) with 4-ethynylbenzaldehyde 2e (157 mg, 1.2 mmol) gave, after FC (eluent: EtOAc/n-hexane = 1: 100), propargylic amine 3w (246 mg, yield: 67%) as a white solid. Mp 79-81\(^\circ\)C; IR (film) \( \nu_{\text{max}} \): 3439, 2885,1697, 1639, 1450, 1136, 1075, 697 cm\(^{-1}\). \(^1\)H NMR (500 MHz, CDCl\(_3\)): \( \delta \) 1.01 (t, \( J = 7.3 \) Hz, 3H), 1.73-1.89 (m,2H), 3.48 (d, \( J = 13.7 \) Hz, 2H), 3.05 (t, \( J = 7.6 \) Hz, 1H), 3.90 (d, \( J = 13.7 \) Hz, 2H), 7.20-7.25 (m, 2H), 7.31-7.34 (m, 4H), 7.41-7.43 (m, 4H), 7.63 (d, \( J = 8.0 \) Hz, 2H), 7.85 (d, \( J = 8.0 \) Hz, 2H), 10.0 (s, 1H) ppm; \(^{13}\)C NMR (125 MHz, CDCl\(_3\)): \( \delta \) 11.2, 26.8, 54.1, 55.0 (2C), 84.7, 92.7, 126.9, 128.8 (2C), 128.8 (2C), 129.5 (2C),
129.9 (4C), 132.4 (4C), 135.2 (2C), 139.6, 191.5 ppm; MS (ESI) m/z 368 (M+H⁺); HRMS (ESI) m/z calcd for [C26H26NO]+(M+H⁺): 368.2009; found: 368.2010.

Reference

The $^1$H NMR and $^{13}$C NMR spectra of compound 3a
The $^1$H NMR and $^{13}$C NMR spectra of compound 3b
The $^1$H NMR and $^{13}$C NMR spectra of compound 3c
The $^1$H NMR and $^{13}$C NMR spectra of compound 3d
The $^1$H NMR and $^{13}$C NMR spectra of compound 3e
The $^1$H NMR and $^{13}$C NMR spectra of compound 3f
The $^1$H NMR and $^{13}$C NMR spectra of compound 3g.
The $^1$H NMR and $^{13}$C NMR spectra of compound 3h
The $^1$H NMR and $^{13}$C NMR spectra of compound 3i
The $^1$H NMR and $^{13}$C NMR spectra of compound 3j
The $^1$H NMR and $^{13}$C NMR spectra of compound 3k
The $^1$H NMR and $^{13}$C NMR spectra of compound 3l
The $^1$H NMR and $^{13}$C NMR spectra of compound 31
The $^1$H NMR and $^{13}$C NMR spectra of compound 3m
The $^1$H NMR and $^{13}$C NMR spectra of compound 3n
The $^1$H NMR and $^{13}$C NMR spectra of compound 30
The $^1$H NMR and $^{13}$C NMR spectra of compound 3p
The $^1$H NMR and $^{13}$C NMR spectra of compound 3q
The $^1$H NMR and $^{13}$C NMR spectra of compound 3r
The $^1$H NMR and $^{13}$C NMR spectra of compound 3s
The $^1$H NMR and $^{13}$C NMR spectra of compound 3t
The $^1$H NMR and $^{13}$C NMR spectra of compound 3u
The $^1$H NMR and $^{13}$C NMR spectra of compound 4
The $^1$H NMR and $^{13}$C NMR spectra of compound 3v
The $^1$H NMR and $^{13}$C NMR spectra of compound 3w
HMBC Spectrum of compound 3l'
Table 1. Comparison of the data of the diagnostic protons at the stereogenic centres of the known diastereomers A, B, and C with those of the major diastereomers 3q – 3t (1H NMR, CDCl₃)

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<th>Compound</th>
<th>Chemical Structure</th>
<th>(MHz)</th>
<th>Proton Data</th>
<th>Reference</th>
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<td>A (300 MHz)</td>
<td><img src="image" alt="Structure A" /></td>
<td>3.73-3.78 (dd, J = 9.0, 6.9 Hz, 1H)</td>
<td>5.27 (s, 1H)</td>
<td>1. V. K.-Y. Lo, Y. Liu, M.-K. Wong and C.-M. Che, Org. Lett., 2006, 8, 1529-1532.</td>
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<tr>
<td>B (300 MHz)</td>
<td><img src="image" alt="Structure B" /></td>
<td>3.57-3.61 (dd, J = 9.3, 4.5 Hz, 1H)</td>
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<td>C (300 MHz)</td>
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<td>3.74-3.78 (m, 1H)</td>
<td>3.78 (s, 3H)</td>
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<td>3q (400 MHz)</td>
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<td>3.77 (dd, J = 8.9, 7.1 Hz, 1H) superposed with 3.77 (s, 3H)</td>
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<td><img src="image" alt="Structure 3r" /></td>
<td>3.67 (dd, J = 9.0, 6.8 Hz, 1H) superposed with 3.76 (s, 3H)</td>
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<td>3.77 (dd, J = 9.1, 7.1 Hz, 1H) superposed with 3.79 (s, 3H)</td>
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Reference:


Table 2. Comparison of data of the diagnostic carbon at the stereogenic centre of the known diastereomers\(^1\) A, B, and C with those of our major diastereomers 3q – 3t (\(^{13}\)C NMR, CDCl\(_3\))

<table>
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<th>(\text{Ph}^\text{N}^\text{CO}_2\text{Me})</th>
<th>(\text{Ph}^\text{N}^\text{CO}_2\text{Me})</th>
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<tr>
<td>A (125 MHz)</td>
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<td>(63.1)</td>
<td>(63.1)</td>
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<tr>
<td>B (125 MHz)</td>
<td>(60.85)</td>
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<td>(63.0)</td>
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<td>C (75 MHz)</td>
<td>(63.1)</td>
<td>(63.0)</td>
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Reference:


$^1$H NMR Spectrum of the crude 3q

$^1$H NMR Spectrum of the crude 3r
$^1$H NMR Spectrum of the crude 3s

$^1$H NMR Spectrum of the crude 3t