Supporting Information

Kinetic Resolution of Propargylamines via a Highly Enantioselective Non-enzymatic N-Acylation Process

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General: The reactions were run under argon atmosphere in oven-dried glassware unless otherwise specified. Dichloromethane was distilled from calcium hydride. THF and Et₂O were distilled from sodium/benzophenone. DMF was distilled under vacuum over MgSO₄, and pyridine was stored over NaOH pellets. Analytical thin layer chromatography (TLC) was performed on silica gel plates (Merck 60F₂₅₄) visualized either with a UV lamp (254 nm) or by using solutions of p-anisaldehyde/sulfuric acid/acetic acid in EtOH, phosphomolybdic acid in EtOH or KMnO₄/K₂CO₃/AcOH in water followed by heating. Flash chromatographies were performed on silica gel (60-230 mesh mesh). All the reactions were carried out under N₂ atmosphere. Organic extracts were dried over anhydrous Na₂SO₄. Infrared spectra (IR) were recorded on a Bruker TENSOR™ 27 (IRTF) and wave-numbers are indicated in cm⁻¹. ¹H NMR spectra were recorded on a Bruker AVANCE 400 at 400 MHz in CDCl₃ and data are reported as follows: chemical shift in parts per million from tetramethylsilane as an internal standard, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of nonequivalent resonances), integration. ¹³C NMR spectra were recorded at 100 MHz in CDCl₃ (unless otherwise specified) and data were reported as follows: chemical shift in parts per million from tetramethylsilane with the solvent as an internal indicator (CDCl₃ d 77.0 ppm), multiplicity with respect to proton (deduced from DEPT experiments, s = quaternary C, d = CH, t = CH₂, q = CH₃). Mass spectra (MS) were recorded using a Hewlett-Packard tandem 5890A/5971 GCMS (70 eV). High-Resolution Mass Spectra were performed by "Groupe de Spectrométrie de masse de l'Université Pierre et Marie Curie (Paris)". The enantiomeric excesses were determined by supercritical fluid chromatography (SFC) analysis on chiral phase.
**General procedure for the synthesis of the propargyl alcohol precursors:** To a solution of alkyne (10 mmol, 1 equiv.) in THF (28 mL) at 0 °C was added a solution of n-butyllithium (2.5 M in THF, 4 mL, 10 mmol, 1 equiv.) drop-wise. After stirring for 30 min at the same temperature, the aldehyde (12 mmol, 1.2 equiv.) was added and the mixture was stirred for an additional 30 min before a saturated aqueous solution of NH₄Cl (50 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with EtOAc (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced. The crude residue was purified by flash column chromatography over silica gel to afford the pure propargyl alcohol.

**General procedure for the synthesis of the phthalimide precursors:** To a solution of propargyl alcohol (10 mmol, 1 equiv.) in THF (100 mL) at 0 °C were added triphenylphosphine (2.89 g, 11 mmol, 1.1 equiv.), phthalimide (1.62 g, 11 mmol, 1.1 equiv.) and a solution of DEAD (40 %wt in toluene, 5.9 mL, 13 mmol, 1.3 equiv.). The reaction mixture was then stirred for 20 h at room temperature before the solvent was evaporated and the triphenylphosphate oxide precipitated by addition of a 1:1 mixture of Et₂O/PE (40 mL). The precipitate was filtered through a plug of Celite® and the solvent was evaporated under reduced pressure to afford a crude residue which was purified by flash column chromatography over silica gel.

**General procedure for the Sonogashira coupling with aryl bromides:** To a solution of terminal alkyne (2.5 mmol, 1 equiv.) and aryl bromide (5 mmol, 2 equiv.) in a freshly degased 3:1 MeCN/Et₃N (12 mL) mixture (solvents were degased separately) were added CuI (88 mg, 0.125 mmol, 5 mol %) and PdCl₂(PPh₃)₂ (24 mg, 0.125 mmol, 5 mol %). The reaction mixture was then heated under microwave irradiation (closed vessel, 400 W, 100 °C) for 1 h, time after the
solvents were removed under reduced pressure and the residue was purified by flash column chromatography over silica gel.

**General procedure for the Sonogashira coupling with vinyl iodides:** To a solution of terminal alkyne (0.6 mmol, 1 equiv.) and (E)-1-iodobut-1-ene (658 mg, 3.6 mmol, 6 equiv.) in a freshly degased 1:1 DMF/Et$_3$N (20 mL) mixture (solvents were degased separately) were added CuI (15 mg, 0.08 mmol, 13 mol %) and PdCl$_2$(PPh$_3$)$_2$ (21 mg, 0.03 mmol, 5 mol %). The reaction mixture was then stirred at room temperature for 3 h, time after which the organic layer was separated and the aqueous phase was extracted twice with EtOAc (2 x 20 mL). The combined organic layers were dried over anhydrous Na$_2$SO$_4$, filtered and concentrated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel.

**General procedure for the cleavage of the phthalimide:** To a solution of phthalimide (3 mmol, 1 equiv.) in EtOH (26 mL) was added hydrazine hydrate (0.9 mL, 18 mmol, 6 equiv.) drop-wise and the resulting mixture was stirred at reflux for 3 h. The formed pasty precipitate was then filtered through a plug of Celite®, washed with Et$_2$O (20 mL) and the solvent was evaporated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel to afford the desired pure propargyl amine.

**General procedure for the kinetic resolution of propargyl amines:** To a solution of propargyl amine (0.14 mmol, 1 equiv.) in THF (1.2 mL) at –20 °C was added Aliquat™ 336 (0.8 mL). A solution of (1S,2S)-1 (0.07 mmol, 0.5 equiv.) in THF (0.6 mL) was then added during 1 h using a syringe pump and the resulting mixture was stirred overnight at the same temperature. Evaporation of the solvent and purification of the residue by flash column chromatography over silica gel gave the corresponding enantio-enriched acetamide.
4-Phenylbut-3-yn-2-ol

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\begin{align*}
\text{IR (neat): } & \quad 3349, 2961, 2872, 2200, 1489, 1383, 1026, 754, 689 \text{ cm}^{-1}. \\
\text{HRMS (ESI) m/z: } & \quad \text{calcd for C}_{12}\text{H}_{14}\text{NaO}[\text{M + Na}]^{+}: 197.0937, \text{ found: 197.0782.}
\end{align*}
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1H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.43-7.36\) (m, 2H, H\(_{\text{Ph}}\)), 7.30-7.22 (m, 3H, H\(_{\text{Ph}}\)), 4.73 (qd, \(J = 6.6, 1.7\) Hz, 1H, H\(_1\)), 2.37 (s, 1H, OH), 1.52 (dd, \(J = 6.6, 1.8\) Hz, 3H, H\(_8\)). 13C NMR (100 MHz, CDCl\(_3\)) \(\delta 131.7\) (d, C\(_{\text{Ph}}\)), 128.5 (d, C\(_{\text{Ph}}\)), 128.4 (d, C\(_{\text{Ph}}\)), 122.7 (s, C\(_4\)), 91.1 (s, C\(_2\)), 84.1 (s, C\(_3\)), 58.9 (d, C\(_1\)), 24.4 (q, C\(_8\)).

1-Phenylpent-1-yn-3-ol

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\begin{align*}
\text{IR (neat): } & \quad 3367, 2208, 1598, 1158 \text{ cm}^{-1}. \\
\text{HRMS (ESI) m/z: } & \quad \text{calcd for C}_{12}\text{H}_{14}\text{NaO}[\text{M + Na}]^{+}: 197.0937, \text{ found: 197.0782.}
\end{align*}
\]

1H NMR (400 MHz, CDCl\(_3\)) \(\delta 7.48-7.41\) (m, 2H, H\(_{\text{Ph}}\)), 7.35-7.28 (m, 3H, H\(_{\text{Ph}}\)), 4.57 (t, \(J = 6.4\) Hz, 1H, H\(_1\)), 1.95 (s, 1H, OH), 1.88-1.79 (m, 2H, H\(_8\)), 1.09 (t, \(J = 7.4\) Hz, 3H, H\(_9\)). 13C NMR (100 MHz, CDCl\(_3\)) \(\delta 131.7\) (d, C\(_{\text{Ph}}\)), 128.4 (d, C\(_{\text{Ph}}\)), 122.7 (s, C\(_4\)), 91.1 (s, C\(_2\)), 84.1 (s, C\(_3\)), 64.2 (d, C\(_1\)), 31.0 (t, C\(_8\)), 9.5 (q, C\(_9\)).

4-Methyl-1-phenylpent-1-yn-3-ol

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\begin{align*}
\text{IR (neat): } & \quad 3349, 2961, 2872, 2200, 1489, 1383, 1026, 754, 689 \text{ cm}^{-1}. \\
\text{HRMS (ESI) m/z: } & \quad \text{calcd for C}_{12}\text{H}_{14}\text{NaO}[\text{M + Na}]^{+}: 197.0937, \text{ found: 197.0782.}
\end{align*}
\]

5-Methyl-1-phenylhex-1-yn-3-ol³

IR (neat): 3338, 2956, 2931, 2870, 2202, 1664, 1489, 1443, 1367, 1132, 1057, 1027, 755, 690 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.47-7.41 (m, 2H, H₉), 7.34-7.29 (m, 3H, H₈), 4.66 (t, J = 7.2 Hz, 1H, H₁), 2.11 (s, 1H, OH), 1.93 (dseptapp, J = 13.5, 6.7 Hz, 1H, H₉), 1.80-1.63 (m, 2H, H₈), 0.99 (dd, J = 6.6, 5.9 Hz, 6H, H₁₀).

¹³C NMR (100 MHz, CDCl₃) δ 131.8 (d, C₇), 128.5 (d, C₈), 128.4 (d, C₉), 122.8 (s, C₄), 90.5 (s, C₂), 84.9 (s, C₃), 61.6 (d, C₁), 47.0 (s, C₉), 25.0 (d, C₉), 22.7 (q, C₁₀).

1,4-Diphenylbut-3-yn-2-ol⁴

IR (neat): 3339, 3029, 2924, 2203, 1664, 1598, 1489, 1442, 1028, 753, 690 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.45-7.40 (m, 2H, H₉), 7.38-7.27 (m, 8H, H₈), 4.83 (t, J = 6.3 Hz, 1H, H₁), 3.14 (d, J = 6.12 Hz, 1H, H₃), 3.13 (d, J = 6.48 Hz, 1H, H₈'), 2.04 (s, 1H, OH).

¹³C NMR (100 MHz, CDCl₃) δ 136.6 (s, C₉), 131.8 (d, C₈), 130.0 (d, C₉), 128.6 (d, C₈), 128.5 (d, C₉), 128.4 (d, C₈), 127.1 (d, C₉), 122.6 (s, C₄), 89.5 (s, C₃), 85.9 (s, C₃), 63.8 (d, C₁), 44.3 (t, C₈).

1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-ol

IR (neat): 3000, 2933, 2835, 2199, 1609, 1510, 1303, 1247, 1171, 1028, 829, 755, 689 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, J = 8.4 Hz, 2H, H₉), 7.52-7.47 (m, 2H, H₈), 7.37 7.31 (m, 3H, H₈), 6.94 (d, J = 8.8 Hz, 2H, H₁₀), 5.66 (s, 1H, H₁), 3.83 (s, 3H, H₁₂), 2.46 (s, 1H, OH).

¹³C NMR (100 MHz, CDCl₃) δ 159.8 (s, C₁₁), 133.1 (s, C₈), 131.8 (d, C₈), 128.7 (d, C₈), 128.4 (d, C₉), 128.3 (d, C₉), 122.6 (s, C₄), 89.5 (s, C₃), 85.9 (s, C₃), 63.8 (d, C₁), 44.3 (t, C₈).

C(H), 128.3 (d, C9), 122.6 (s, C4), 114.1 (s, C10), 89.1 (s, C2), 86.6 (s, C3), 64.8 (d, C1), 55.4 (q, C12).


4-(p-Tolyl)but-3-yn-2-ol

IR (neat): 3352, 2199, 1668, 1509, 1282, 1103, 1034, 933, 815 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.34 (d, J = 8.1 Hz, 2H, H5 or H6), 7.11 (d, J = 7.8 Hz, 2H, H5 or H6), 4.77 (q, J = 6.6 Hz, 1H, H1), 2.69 (s br, 1H, OH), 2.34 (s, 3H, H8), 1.56 (d, J = 6.6 Hz, 3H, H9).

¹³C NMR (100 MHz, CDCl₃) δ 138.4 (s, C7), 131.6 (d, C5 or C6), 129.0 (d, C5 or C6), 119.6 (s, C4), 90.5 (s, C2), 84.1 (s, C3), 58.8 (d, C1), 24.4 (q, C9), 21.5 (q, C8). HRMS (ESI) m/z: calcd for C11H10NaO [M + Na]+: 181.0624, found: 181.0626.

4-(2,4-Difluorophenyl)but-3-yn-2-ol

IR (neat): 3320, 2985, 1618, 1503, 1425, 1265, 1143, 1094, 969, 849, 814, 612 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.44-7.36 (m, 1H, H9), 6.87-6.79 (m, 2H, H6 + H8), 4.78 (q, J = 6.6 Hz, 1H, H1), 2.08 (s br, 1H, OH), 1.57 (d, J = 6.6 Hz, 3H, H10). ¹³C NMR (100 MHz, CDCl₃) δ 163.2 (s, J = 253.0, 12.0 Hz, C5 or C7), 162.9 (s, J = 250.0, 12.0 Hz, C5 or C7), 134.5 (d, J = 10.0, 3.0 Hz, C9), 111.7 (d, J = 22.0, 4.0 Hz, C6 or C8), 107.6 (s, J = 16.0, 4.0 Hz, C4), 104.4 (d, J = 26.0, 3.0 Hz, C6 or C8), 96.0 (s, J = 3.0, 2.0 Hz, C2), 76.6 (s, C3), 59.0 (d, C1), 24.3 (q, C10).


4-[3-(Trifluoromethyl)phenyl]but-3-yn-2-ol
IR (neat): 3317, 2984, 1432, 1331, 1236, 1166, 1124, 1071, 946, 901, 800, 694, 657 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.69 (s, 1H, H₅), 7.64-7.51 (m, 2H, H₇ + H₉), 7.48-7.37 (m, 1H, H₈), 4.78 (q, J = 6.6 Hz, 1H, H₁), 1.57 (d, J = 6.6 Hz, 3H, H₁₁). ¹³C NMR (100 MHz, CDCl₃) δ 134.8 (d, C₇ or C₉), 131.0 (s, J = 32.0 Hz, C₆), 129.0 (d, C₈), 128.6 (d, C₅), 125.1 (d, C₇ or C₉), 123.8 (s, J = 270.0 Hz, C₁₀), 123.7 (s, C₄), 92.6 (s, C₂), 82.7 (s, C₃), 58.9 (d, C₁), 24.4 (q, C₁₁).

8-[(tert-Butyldiphenylsilyloxy)oct-5-yn-4-ol

IR (neat): 3392, 2959, 2214, 1673, 1427, 1361, 1106, 822, 736, 700, 612 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.69 (s, 1H, H₅), 7.64-7.51 (m, 2H, H₇ + H₉), 7.48-7.37 (m, 1H, H₈), 4.78 (q, J = 6.6 Hz, 1H, H₁), 1.57 (d, J = 6.6 Hz, 3H, H₁₁). ¹³C NMR (100 MHz, CDCl₃) δ 134.8 (d, C₇ or C₉), 131.0 (s, J = 32.0 Hz, C₆), 129.0 (d, C₈), 128.6 (d, C₅), 125.1 (d, C₇ or C₉), 123.8 (s, J = 270.0 Hz, C₁₀), 123.7 (s, C₄), 92.6 (s, C₂), 82.7 (s, C₃), 58.9 (d, C₁), 24.4 (q, C₁₁).

8-[(tert-Butyldiphenylsilyloxy)oct-5-yn-4-ol

IR (neat): 3392, 2959, 2214, 1673, 1427, 1361, 1106, 822, 736, 700, 612 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.69 (s, 1H, H₅), 7.64-7.51 (m, 2H, H₇ + H₉), 7.48-7.37 (m, 1H, H₈), 4.78 (q, J = 6.6 Hz, 1H, H₁), 1.57 (d, J = 6.6 Hz, 3H, H₁₁). ¹³C NMR (100 MHz, CDCl₃) δ 134.8 (d, C₇ or C₉), 131.0 (s, J = 32.0 Hz, C₆), 129.0 (d, C₈), 128.6 (d, C₅), 125.1 (d, C₇ or C₉), 123.8 (s, J = 270.0 Hz, C₁₀), 123.7 (s, C₄), 92.6 (s, C₂), 82.7 (s, C₃), 58.9 (d, C₁), 24.4 (q, C₁₁).

8-(Benzyloxy)oct-5-yn-4-ol

IR (neat): 3395, 2958, 2870, 1454, 1362, 1098, 1026, 736, 697 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.28 (m, 5H, H₉), 4.56 (s, 2H, H₆), 4.34 (tt, J = 6.6, 1.9 Hz, 1H, H₁), 3.59 (d, J = 7.0 Hz, 2H, H₅), 2.60 (s, 1H, OH), 2.53 (td, J = 7.0, 2.0 Hz, 2H, H₄), 1.79-1.57 (m, 2H, H₁), 1.54-1.42 (m, 2H, H₁₁), 0.95 (t, J = 7.4 Hz, 3H, H₁₃). ¹³C NMR (100 MHz, CDCl₃) δ 138.0 (s, C₇), 128.5 (d, Cₙ), 127.8 (2d, Cₙ), 82.7 (s, C₂), 81.8 (s, C₃), 72.9 (t, C₆), 68.4 (t, C₅), 62.3 (s, C₁₀), 40.1 (t, C₁₁), 20.1 (t, C₄), 18.5 (t, C₁₂), 13.8 (q, C₁₃). HRMS (ESI) m/z: calcd for C₁₅H₂₀NaO₂Si [M + Na]⁺: 255.13556, found: 255.13557.
2-(4-Phenylbut-3-yn-2-yl)isoindoline-1,3-dione

IR (neat): 3064, 2926, 1777, 1703, 1488, 1384, 1337, 1147, 1065, 879, 760, 713 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.85 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{11}\) or H\(_{12}\)), 7.70 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{11}\) or H\(_{12}\)), 7.47-7.38 (m, 2H, H\(_{11}\) or H\(_{12}\)), 7.29-7.23 (m, 3H, H\(_{Ph}\)), 5.42 (q, \(J = 7.1\) Hz, 1H, H\(_1\)), 1.78 (d, \(J = 7.2\) Hz, 3H, H\(_8\)).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 167.1 (s, C\(_9\)), 134.2 (s, C\(_{11}\) or C\(_{12}\)), 132.1 (d, C\(_{Ph}\)), 132.0 (s, C\(_{10}\)), 128.5 (d, C\(_{Ph}\)), 128.3 (d, C\(_{Ph}\)), 123.5 (s, C\(_{11}\) or C\(_{12}\)), 122.6 (s, C\(_4\)), 86.7 (s, C\(_2\)), 83.0 (s, C\(_3\)), 37.8 (s, C\(_1\)), 20.4 (s, C\(_8\)).

HRMS (ESI) \(m/z\): calcd for C\(_{18}\)H\(_{13}\)NaNO\(_2\) [M + Na]\(^+\): 298.0838, found: 298.0842.

2-(1-Phenylpent-1-yn-3-yl)isoindoline-1,3-dione

IR (neat): 3025, 2973, 2879, 1774, 1703, 1466, 1384, 1357, 1146, 1077, 896, 751, 712, 688, 644 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.85 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{12}\) or H\(_{13}\)), 7.71 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{12}\) or H\(_{13}\)), 7.47-7.38 (m, 2H, H\(_{Ph}\)), 7.31-7.20 (m, 3H, H\(_{Ph}\)), 5.16 (t, \(J = 7.9\) Hz, 1H, H\(_1\)), 2.32-2.04 (m, 2H, H\(_8\)), 1.05 (t, \(J = 7.4\) Hz, 3H, H\(_9\)).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 167.3 (s, C\(_{10}\)), 134.15 (d, C\(_{12}\) or C\(_{13}\)), 132.0 (d, C\(_{11}\)), 131.0 (d, C\(_{Ph}\)), 128.4 (d, C\(_{Ph}\)), 128.3 (d, C\(_{Ph}\)), 123.5 (d, C\(_{12}\) or C\(_{13}\)), 122.7 (s, C\(_4\)), 85.8 (s, C\(_2\)), 83.8 (s, C\(_3\)), 43.9 (d, C\(_1\)), 27.2 (t, C\(_8\)), 11.1 (q, C\(_9\)).

HRMS (ESI) \(m/z\): calcd for C\(_{19}\)H\(_{15}\)NaNO\(_2\) [M + Na]\(^+\): 312.0995, found: 312.0999.
2-(4-Methyl-1-phenylpent-1-yn-3-yl)isoindoline-1,3-dione

**IR** (neat): 2965, 2872, 1776, 1709, 1467, 1380, 1348, 1330, 1077, 882, 756, 714, 690, 640 cm$^{-1}$.

**$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.88 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{12}$ or H$_{13}$), 7.74 (dd, $J = 5.4$, 3.1 Hz, 2H, H$_{12}$ or H$_{13}$), 7.49-7.41 (m, 2H, H$_{Ph}$), 7.32-7.24 (m, 3H, H$_{Ph}$), 4.88 (d, $J = 10.3$ Hz, 1H, H$_1$), 2.76-2.61 (m, 1H, H$_8$), 1.27 (d, $J = 6.7$ Hz, 3H, H$_9$), 0.94 (d, $J = 6.7$ Hz, 3H, H$_9$).

**$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ 167.4 (s, C$_{10}$), 134.2 (d, C$_{12}$ or C$_{13}$), 131.9 (d, C$_{Ph}$), 131.8 (s, C$_{11}$), 128.4 (d, C$_{Ph}$), 128.3 (d, C$_{Ph}$), 123.5 (d, C$_{12}$ or C$_{13}$), 122.8 (s, C$_2$), 85.5 (s, C$_3$), 49.2 (d, C$_1$), 31.8 (d, C$_8$), 20.4 (q, C$_9$), 19.3 (q, C$_9$). **HRMS (ESI) m/z**: calcd for C$_{20}$H$_{17}$NaNO$_2$ [M + Na]$^+$: 326.1151, found: 326.1152.

2-(5-Methyl-1-phenylhex-1-yn-3-yl)isoindoline-1,3-dione

**IR** (neat): 2952, 2927, 2869, 1777, 1707, 1490, 1468, 1380, 1358, 1148, 1080, 876, 761, 718, 693, 647 cm$^{-1}$. **$^1$H NMR** (400 MHz, CDCl$_3$) $\delta$ 7.85 (dd, $J = 5.4$, 3.1 Hz, 2H, H$_{13}$ or H$_{14}$), 7.71 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{13}$ or H$_{14}$), 7.47-7.37 (m, 2H, H$_{Ph}$), 7.33-7.20 (m, 3H, H$_{Ph}$), 5.34 (t, $J = 8.0$ Hz, 1H, H$_1$), 2.15-1.98 (m, 2H, H$_8$), 1.73 (sept, $J = 6.6$ Hz, 1H, H$_9$), 0.98 (dd, $J = 8.3$, 6.6 Hz, 6H, H$_{10}$). **$^{13}$C NMR** (100 MHz, CDCl$_3$) $\delta$ 167.3 (s, C$_{11}$), 134.2 (d, C$_{13}$ or C$_{14}$), 132.0 (s, C$_{12}$), 132.0 (d, C$_{Ph}$), 128.4 (d, C$_{Ph}$), 128.3 (d, C$_{Ph}$), 123.5 (d, C$_{13}$ or C$_{14}$), 122.7 (s, C$_2$), 86.1 (s, C$_3$), 83.5 (s, C$_3$), 42.2 (t, C$_8$), 40.8 (d, C$_1$), 25.4 (d, C$_9$), 22.3 (q, C$_{10}$). **HRMS (ESI) m/z**: calcd for C$_{21}$H$_{19}$NaNO$_2$ [M + Na]$^+$: 340.1308, found: 340.1312.
2-(1,4-Diphenylbut-3-yn-2-yl)isoindoline-1,3-dione

IR (neat): 3028, 2914, 1777, 1703, 1489, 1383, 1355, 1104, 1070, 955, 882, 867, 756, 721, 691, 647 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.81 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{15}$ or H$_{16}$), 7.68 (dd, $J = 5.5$, 3.1 Hz, 2H, H$_{15}$ or H$_{16}$), 7.46-7.39 (m, 2H, H$_{15}$ or H$_{16}$), 7.31-7.16 (m, 8H, H$_{Ph}$), 7.31-7.16 (m, 8H, H$_{Ph}$), 5.51 (t, $J = 8.1$ Hz, 1H, H$_1$), 3.52 (d, $J = 8.1$ Hz, 2H, H$_8$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.1 (s, C$_{13}$), 136.6 (s, C$_9$), 134.1 (d, C$_{15}$ or C$_{16}$), 131.9 (d, C$_{Ph}$), 131.8 (s, C$_{14}$), 129.3 (d, C$_{Ph}$), 128.6 (d, C$_{Ph}$), 128.5 (d, C$_{Ph}$), 128.3 (d, C$_{Ph}$), 127.1 (d, C$_{Ph}$), 123.5 (d, C$_{15}$ or C$_{16}$), 122.5 (s, C$_4$), 85.5 (s, C$_2$), 84.2 (s, C$_3$), 43.7 (d, C$_1$), 39.7 (t, C$_5$). HRMS (ESI) $m/z$: calcd for C$_{24}$H$_{17}$NaNO$_2$ [M + Na]$^+$: 374.1152, found: 374.1159.

2-(1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-yl)isoindoline-1,3-dione

IR (neat): 2932, 2837, 1768, 1711, 1610, 1510, 1377, 1326, 1250, 1174, 1104, 1031, 889, 838, 713, 690 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.85 (dd, $J = 5.4$, 3.1 Hz, 2H, H$_{15}$ or H$_{16}$), 7.71 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{15}$ or H$_{16}$), 7.67 (d, $J = 8.6$ Hz, 2H, H$_{9}$), 7.56-7.49 (m, 2H, H$_{Ph}$), 7.35-7.30 (m, 3H, H$_{Ph}$), 6.90 (d, $J = 8.8$ Hz, 2H, H$_{10}$), 6.51 (s, 1H, H$_1$), 3.80 (s, 3H, H$_{12}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.8 (s, C$_{13}$), 159.7 (s, C$_{11}$), 134.2 (d, C$_{15}$ or C$_{16}$), 123.1 (s, C$_{14}$), 132.1 (d, C$_{Ph}$), 129.6 (d, C$_9$), 129.0 (s, C$_8$), 128.7 (d, C$_{Ph}$), 128.4 (d, C$_{Ph}$), 123.6 (d, C$_{15}$ or C$_{16}$), 122.6 (s, C$_4$), 114.0 (d, C$_{10}$), 85.7 (s, C$_2$), 84.3 (s, C$_3$), 55.4 (q, C$_{12}$), 44.9 (d, C$_1$). HRMS (ESI) $m/z$: calcd for C$_{24}$H$_{17}$NaNO$_3$ [M + Na]$^+$: 390.1101, found: 390.1102.
2-(But-3-yn-2-yl)isoindoline-1,3-dione

IR (neat): 3266, 1775, 1710, 1385, 1334, 1141, 1059, 999, 881, 714, 678, 614 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.84 (dd, J = 5.5, 3.0 Hz, 2H, H₇ or H₈), 7.72 (dd, J = 5.4, 3.1 Hz, 2H, H₇ or H₈), 5.20 (qd, J = 7.2, 2.5 Hz, 1H, H₁), 2.35 (d, J = 2.5 Hz, 1H, H₃), 1.70 (d, J = 7.2 Hz, 3H, H₄). ¹³C NMR (100 MHz, CDCl₃) δ 166.9 (s, C₅), 134.2 (d, C₇ or C₈), 131.9 (s, C₆), 123.5 (d, C₇ or C₈), 81.2 (d, C₃), 71.3 (s, C₂), 36.9 (d, C₁), 20.1 (q, C₄). HRMS (ESI) m/z: calcd for C₁₂H₉NaNO₂ [M + Na]⁺: 222.0525, found: 222.0528.

2-[4-(p-Tolyl)but-3-yn-2-yl]isoindoline-1,3-dione

IR (neat): 1778, 1702, 1509, 1388, 1358, 1150, 1066, 1005, 880, 816, 720, 618 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.86 (dd, J = 5.4, 3.1 Hz, 2H, H₁₂ or H₁₃), 7.72 (dd, J = 5.5, 3.0 Hz, 2H, H₁₂ or H₁₃), 7.33 (d, J = 8.1 Hz, 2H, H₅ or H₆), 7.08 (d, J = 7.9 Hz, 2H, H₅ or H₆), 5.43 (q, J = 7.1 Hz, 1H, H₁), 2.32 (s, 3H, H₈), 1.79 (d, J = 7.2 Hz, 3H, H₉). ¹³C NMR (100 MHz, CDCl₃) δ 167.1 (s, C₁₀), 138.5 (s, C₇), 134.1 (d, C₁₂ or C₁₃), 132.0 (d, C₁₁), 131.8 (d, C₅ or C₆), 129.0 (d, C₅ or C₆), 123.4 d, C₁₂ or C₁₃), 119.5 (s, C₄), 86.0 (s, C₂), 83.1 (s, C₃), 37.8 (d, C₁), 21.5 (q, C₈), 20.4 (q, C₉). HRMS (ESI) m/z: calcd for C₁₉H₁₅NaNO₂ [M + Na]⁺: 312.0995, found: 312.0996.
2-[4-(2,4-Difluorophenyl)but-3-yn-2-yl]isoindoline-1,3-dione

IR (neat): 2989, 1777, 1709, 1503, 1381, 1143, 1088, 969, 855, 720, 614 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.87 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{13}\) or H\(_{14}\)), 7.74 (dd, \(J = 5.5, 3.1\) Hz, 2H, H\(_{13}\) or H\(_{14}\)), 7.47-7.36 (m, 1H, H\(_9\)), 6.86-6.76 (m, 2H, H\(_6\) + H\(_8\)), 5.45 (q, \(J = 7.1\) Hz, 1H, H\(_1\)), 1.80 (d, \(J = 7.2\) Hz, 3H, H\(_{10}\)).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 167.0 (s, C\(_{11}\)), 163.4 (s, \(J = 253.0, 12.0\) Hz, C\(_5\) or C\(_7\)), 162.8 (s, \(J = 251.0, 12.0\) Hz, C\(_5\) or C\(_7\)), 134.8 (d, \(J = 10.0, 3.0\) Hz, C\(_9\)), 134.3 (d, C\(_{13}\) or C\(_{14}\)), 132.0 (s, C\(_{12}\)), 123.6 (d, C\(_{13}\) or C\(_{14}\)), 111.5 (d, \(J = 22.0, 4.0\) Hz, C\(_6\) or C\(_8\)), 107.6 (s, \(J = 16.0\) Hz, C\(_4\)), 104.3 (d, \(J = 26.0, 25.0\) Hz, C\(_6\) or C\(_8\)), 91.6 (s, C\(_2\)), 75.6 (s, C\(_3\)), 37.8 (d, C\(_1\)), 20.3 (q, C\(_{10}\)).

HRMS (ESI) \(m/z\): calcd for C\(_{18}\)H\(_{11}\)F\(_2\)NaNO\(_2\) [M + Na]\(^+\): 334.0650, found: 334.0652.

2-[4-(3-(Trifluoromethyl)phenyl)but-3-yn-2-yl]isoindoline-1,3-dione

IR (neat): 2960, 2940, 2890, 1776, 1707, 1386, 1357, 1330, 1169, 115, 1071, 879, 798, 718, 694, 618 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.88 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{13}\) or H\(_{15}\)), 7.75 (dd, \(J = 5.4, 3.1\) Hz, 2H, H\(_{14}\) or H\(_{15}\)), 7.70 (s, 1H, H\(_5\)), 7.61 (d, \(J = 7.7\) Hz, 1H, H\(_6\)), 7.55 (d, \(J = 7.9\) Hz, 1H, H\(_7\)), 7.42 (t, \(J = 7.8\) Hz, 1H, H\(_8\)), 5.45 (q, \(J = 7.2\) Hz, 1H, H\(_1\)), 1.81 (d, \(J = 7.2\) Hz, 3H, H\(_{10}\)).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 167.1 (s, C\(_{12}\)), 135.1 (d, C\(_9\)), 134.3 (d, C\(_{14}\) or C\(_{15}\)), 132.0 (s, C\(_{13}\)), 130.4 (s, \(J = 32.0\) Hz, C\(_6\)), 128.9 (d, C\(_8\)), 128.6 (d, C\(_5\)), 125.1 (d, \(J = 4.0\) Hz, C\(_7\)), 123.8 (s, \(J = 271.0\) Hz, C\(_{10}\)), 123.6 (s, C\(_4\)), 123.6 (d, C\(_{14}\) or C\(_{15}\)), 88.4 (s, C\(_2\)), 81.6 (s, C\(_3\)), 37.7 (d, C\(_1\)), 20.3 (q, C\(_{11}\)).

HRMS (ESI) \(m/z\): calcd for C\(_{19}\)H\(_{12}\)F\(_3\)NaNO\(_2\) [M + Na]\(^+\): 366.0712, found: 366.0720.
2-[(tert-Butyldiphenylsilyl)oxy]oct-5-yn-4-yl]isoindoline-1,3-dione

IR (neat): 2959, 2858, 1714, 1382, 1353, 1107, 700, 613 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.84 (dd, J = 5.5, 3.0 Hz, 2H, H₁₇ or H₁₈), 7.71 (dd, J = 5.4, 3.1 Hz, 2H, H₁₇ or H₁₈), 7.69-7.45 (m, 4H, H₆), 7.46-7.35 (m, 6H, H₆), 5.03 (tt, J = 7.9, 2.2 Hz, 1H, H₁), 3.75 (t, J = 6.9 Hz, 2H, H₅), 2.48 (td, J = 6.9, 2.2 Hz, 2H, H₄), 2.12-1.95 (m, 2H, H₁₁), 0.93 (t, J = 7.4 Hz, 3H, H₁₄). ¹³C NMR (100 MHz, CDCl₃) δ 167.4 (s, C₁₅), 135.7 (d, C₆), 134.1 (d, C₁₇ or C₁₈), 133.7 (s, C₆), 132.0 (s, C₁₆), 129.7 (d, C₆), 127.8 (d, C₆), 123.4 (d, C₁₇ or C₁₈), 81.1 (s, C₂), 77.8 (s, C₃), 62.4 (t, C₃), 41.9 (d, C₁), 35.7 (t, C₁₂), 26.8 (q, C₁₁), 19.8 (t, C₁₃), 19.3 (s, C₁₀), 13.5 (q, C₁₄). HRMS (ESI) m/z: calcd for C₃₂H₃₅NaNO₃Si [M + Na]⁺: 532.2278, found: 532.2271.

2-[(Benzyloxy)oct-5-yn-4-yl]isoindoline-1,3-dione

IR (neat): 2960, 2872, 1709, 1383, 1353, 1101, 1072, 716, 697 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.84 (dd, J = 5.5, 3.0 Hz, 2H, H₁₆ or H₁₇), 7.71 (dd, J = 5.5, 3.0 Hz, 2H, H₁₆ or H₁₇), 7.37-7.22 (m, 5H, H₆), 5.04 (tt, J = 7.9, 2.1 Hz, 1H, H₁), 4.54 (s, 2H, H₆), 3.59 (t, J = 7.1 Hz, 2H, H₃), 2.52 (td, J = 7.1, 2.2 Hz, 2H, H₄), 2.13-1.94 (m, 2H, H₁₁), 1.50-1.30 (m, 2H, H₁₂), 0.94 (t, J = 7.4 Hz, 3H, H₁₃). ¹³C NMR (100 MHz, CDCl₃) δ 167.3 (s, C₁₄), 138.2 (s, C₇), 134.0 (d, C₁₆ or C₁₇), 131.9 (s, C₁₃), 128.4 (d, C₆), 127.7 (d, C₆), 127.6 (d, C₆), 123.4 (d, C₁₆ or C₁₇), 80.7 (s, C₂), 77.8 (s, C₃), 73.0 (t, C₆), 68.4 (t, C₃), 41.8 (d, C₁), 35.7 (t, C₁₁), 20.2 (t, C₄), 19.7 (t, C₁₂), 13.4 (q, C₁₃). HRMS (ESI) m/z: calcd for C₂₃H₂₃NaNO₃ [M + Na]⁺: 384.1570, found: 384.1571.
2-[4-(Naphthalen-1-yl)but-3-yn-2-yl]isoindoline-1,3-dione

**IR** (neat): 3057, 2985, 2937, 1776, 1380, 1348, 1147, 1115, 1039, 879, 801, 774, 714, 615 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.43-8.39 (m, 1H, H\(_{12}\)), 7.90 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{17}\) or H\(_{18}\)), 7.85-7.79 (m, 2H, H\(_7\) + H\(_8\)), 7.74 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{17}\) or H\(_{18}\)), 7.68 (dd, \(J = 7.2, 1.2\) Hz, 1H, H\(_8\) or H\(_9\)), 7.61 (ddd, \(J = 8.3, 6.9, 1.3\) Hz, 1H, H\(_{11}\)), 7.52 (ddd, \(J = 8.1, 6.9, 1.3\) Hz, 1H, H\(_{10}\)), 7.40 (dd, \(J = 8.3, 7.2\) Hz, 1H, H\(_5\) or H\(_6\)), 5.60 (q, \(J = 7.1\) Hz, 1H, H\(_1\)), 1.91 (d, \(J = 7.1\) Hz, 3H, H\(_{14}\)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 167.2 (s, C\(_{15}\)), 134.2 (d, C\(_{17}\) or C\(_{18}\)), 133.7 (s, C\(_{13}\) or C\(_8\)), 133.2 (s, C\(_{13}\) or C\(_8\)), 132.1 (s, C\(_{16}\)), 130.8 (d, C\(_5\) or C\(_6\)), 129.0 (d, C\(_7\) or C\(_9\)), 128.3 (d, C\(_7\) or C\(_9\)), 127.0 (d, C\(_{11}\)), 126.5 (d, C\(_{12}\) or C\(_{10}\)), 126.4 (d, C\(_{12}\) or C\(_{10}\)), 125.2 (d, C\(_5\) or C\(_6\)), 123.6 (d, C\(_{17}\) or C\(_{18}\)), 120.3 (s, C\(_4\)), 91.6 (s, C\(_2\)), 81.3 (s, C\(_3\)), 38.1 (d, C\(_1\)), 20.7 (q, C\(_{14}\)). HRMS (ESI) \(m/\ell\): calcld for C\(_{22}\)H\(_{15}\)NaNO\(_2\) [M + Na]\(^+\): 348.0995, found: 348.0996.

2-[4-(Naphthalen-2-yl)but-3-yn-2-yl]isoindoline-1,3-dione

**IR** (neat): 3057, 1777, 1714, 1467, 1382, 1351, 1147, 1064, 879, 818, 719 cm\(^{-1}\). \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.98 (d, \(J = 0.8\) Hz, 1H, H\(_{13}\)), 7.89 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{17}\) or H\(_{18}\)), 7.81-7.72 (m, 5H, H\(_6\) + H\(_8\) + H\(_{11}\) + H\(_{17}\) or H\(_{18}\)), 7.51-7.45 (m, 3H, H\(_5\), H\(_9\), H\(_{10}\)), 5.50 (q, \(J = 7.2\) Hz, 1H, H\(_1\)), 1.85 (d, \(J = 7.2\) Hz, 3H, H\(_{14}\)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 167.2 (s, C\(_{15}\)), 134.2 (d, C\(_{17}\) or C\(_{18}\)), 133.0 (s, C\(_{12}\)), 133.0 (s, C\(_7\)), 132.1 (d, C\(_{16}\)), 132.0 (d, C\(_{13}\)), 128.7 (d, C\(_5\), C\(_9\) or C\(_{10}\)), 128.0 (d, C\(_6\), C\(_8\) or C\(_{11}\)), 127.9 (d, C\(_6\), C\(_8\) or C\(_{11}\)), 127.8 (d, C\(_6\), C\(_8\) or C\(_{11}\)), 126.8 (d, C\(_5\), C\(_9\) or C\(_{10}\)), 126.6 (d, C\(_5\), C\(_9\) or C\(_{10}\)), 123.5 (d, C\(_{17}\) or C\(_{18}\)), 120.0 (s, C\(_4\)), 87.0 (s, C\(_2\)), 83.3 (s, C\(_3\)), 37.9 (d, C\(_1\)), 20.5 (q, C\(_{14}\)). HRMS (ESI) \(m/\ell\): calcld for C\(_{22}\)H\(_{15}\)NaNO\(_2\) [M + Na]\(^+\): 348.0995, found: 348.0997.
2-[4-(4-Methoxyphenyl)but-3-yn-2-yl]isoindoline-1,3-dione

IR (neat): 2937, 2235, 1777, 1703, 1383, 1356, 1290, 1251, 1173, 1154, 1072, 1029, 1016, 880, 828, 719, 615 cm$^{-1}$. \textsuperscript{1}H NMR (400 MHz, CDCl$_3$) δ 7.86 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{12}$ or H$_{13}$), 7.72 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{12}$ or H$_{13}$), 7.40-7.33 (m, 2H, H$_5$ or H$_6$), 6.84-6.76 (m, 2H, H$_5$ or H$_6$), 5.42 (q, $J = 7.1$ Hz, 1H, H$_1$), 3.79 (s, 3H, H$_8$), 1.79 (d, $J = 7.2$ Hz, 3H, H$_9$). \textsuperscript{13}C NMR (100 MHz, CDCl$_3$) δ 167.2 (s, C$_{10}$), 159.8 (s, C$_7$), 134.1 (d, C$_{12}$ or C$_{13}$), 133.4 (d, C$_5$ or C$_6$), 132.1 (s, C$_{11}$), 123.5 (d, C$_{12}$ or C$_{13}$), 114.8 (s, C$_4$), 113.9 (d, C$_5$ or C$_6$), 85.3 (s, C$_2$), 82.9 (s, C$_3$), 55.4 (q, C$_8$), 37.9 (d, C$_1$), 20.50 (q, C$_9$). HRMS (ESI) m/z: calcd for C$_{19}$H$_{15}$NaNO$_3$ [M + Na]$^+$: 328.0944, found: 328.0944.

2-[4-(3-Methoxyphenyl)but-3-yn-2-yl]isoindoline-1,3-dione

IR (neat): 3062, 2934, 1775, 1704, 1605, 1575, 1481, 1466, 1384, 1355, 1289, 1205, 1046, 877, 721, 682 cm$^{-1}$. \textsuperscript{1}H NMR (400 MHz, CDCl$_3$) δ 7.85 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{14}$ or H$_{15}$), 7.71 (dd, $J = 5.4$, 3.1 Hz, 2H, H$_{14}$ or H$_{15}$), 7.21-7.13 (m, 1H, H$_{Ph}$), 7.02 (dt, $J = 7.6$, 1.2 Hz, 1H, H$_{Ph}$), 6.96-6.94 (m, 1H, H$_{Ph}$), 6.84 (ddd, $J = 8.3$, 2.6, 1.0 Hz, 1H, H$_{Ph}$), 5.43 (q, $J = 7.2$ Hz, 1H, H$_1$), 3.77 (s, 3H, H$_{10}$), 1.79 (d, $J = 7.2$ Hz, 3H, H$_{11}$). \textsuperscript{13}C NMR (100 MHz, CDCl$_3$) δ 167.1 (s, C$_{12}$), 159.3 (s, C$_6$), 134.2 (d, C$_{14}$ or C$_{15}$), 132.0 (d, C$_{13}$), 129.3 (d, C$_{Ph}$), 124.5 (d, C$_{Ph}$), 123.6 (s, C$_4$), 123.5 (d, C$_{14}$ or C$_{15}$), 116.6 (d, C$_{Ph}$), 115.3 (d, C$_{Ph}$), 86.5 (s, C$_2$), 82.9 (s, C$_3$), 55.4 (q, C$_{10}$), 37.8 (d, C$_1$), 20.4 (q, C$_{11}$). HRMS (ESI) m/z: calcd for C$_{19}$H$_{15}$NaNO$_3$ [M + Na]$^+$: 328.0944, found: 328.0947.
2-[4-(2-Methoxyphenyl)but-3-yn-2-yl]isoindoline-1,3-dione

IR (neat): 3009, 2942, 2838, 1773, 1495, 1388, 1265, 1149, 1023, 880, 752, 727, 616 cm$^{-1}$.  $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.87 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{14}$ or H$_{15}$), 7.72 (dd, $J = 5.4$, 3.1 Hz, 2H, H$_{14}$ or H$_{15}$), 7.44 (dd, $J = 7.6$, 1.7 Hz, 1H, H$_6$ or H$_9$), 7.30-7.23 (m, 1H, H$_7$ or H$_8$), 6.88 (td, $J = 7.5$, 1.0 Hz, 1H, H$_7$ or H$_8$), 6.84 (d, $J = 8.4$ Hz, 1H, H$_6$ or H$_9$), 5.50 (q, $J = 7.1$ Hz, 1H, H$_1$), 3.86 (s, 3H, H$_{10}$), 1.81 (d, $J = 7.1$ Hz, 3H, H$_{11}$).  $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 167.1 (s, C$_{12}$), 160.2 (s, C$_5$), 134.3 (d, C$_6$ or C$_9$), 134.1 (d, C$_{14}$ or C$_{15}$), 132.2 (s, C$_{13}$), 130.0 (d, C$_7$ or C$_8$), 123.5 (d, C$_{14}$ or C$_{15}$), 120.5 (d, C$_7$ or C$_8$), 111.9 (s, C$_4$), 110.8 (d, C$_6$ or C$_9$), 90.8 (s, C$_2$), 79.5 (s, C$_3$), 55.9 (q, C$_{10}$), 38.2 (d, C$_1$), 20.6 (q, C$_{11}$).  HRMS (ESI) $m/z$: calcd for C$_{19}$H$_{15}$NaNO$_3$ [M + Na]$^+$: 328.0944, found: 328.0943.

2-[4-(Pyridin-2-yl)but-3-yn-2-yl]isoindoline-1,3-dione

IR (neat): 3071, 1778, 1703, 1580, 1461, 1386, 1351, 1150, 880, 781, 712, 616 cm$^{-1}$.  $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.54 (d, $J = 4.8$ Hz, 1H, H$_8$), 7.85 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{12}$ or H$_{13}$), 7.72 (dd, $J = 5.5$, 3.0 Hz, 2H, H$_{12}$ or H$_{13}$), 7.63 (td, $J = 7.7$, 1.8 Hz, 1H, H$_6$), 7.44 (dt, $J = 7.9$, 1.0 Hz, 1H, H$_3$), 7.22 (ddd, $J = 7.6$, 4.9, 1.2 Hz, 1H, H$_7$), 5.46 (q, $J = 7.2$ Hz, 1H, H$_1$), 1.81 (d, $J = 7.2$ Hz, 3H, H$_9$).  $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 166.9 (s, C$_{10}$), 149.8 (d, C$_8$), 142.7 (s, C$_4$), 136.4 (d, C$_6$), 134.2 (d, C$_{12}$ or C$_{13}$), 132.0 (s, C$_{11}$), 127.5 (d, C$_3$), 123.5 (d, C$_{12}$ or C$_{13}$), 123.2 (d, C$_7$), 87.0 (s, C$_2$), 82.1 (s, C$_3$), 37.6 (d, C$_1$), 20.1 (q, C$_9$).  HRMS (ESI) $m/z$: calcd for C$_{17}$H$_{13}$N$_2$O$_2$ [M]$^+$: 277.0972, found: 277.0975.
(E)-2-(Oct-5-en-3-yn-2-yl)isoindoline-1,3-dione

IR (neat): 2971, 1777, 1715, 1384, 1353, 1141, 880, 721 cm\(^{-1}\). \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.86 (dd, \(J = 5.5, 3.0\) Hz, 2H, H\(_{11}\) or H\(_{12}\)), 7.72 (dd, \(J = 5.4, 1.8\) Hz, 1H, H\(_4\)), 5.32 (qd, \(J = 7.1, 2.0\) Hz, 1H, H\(_1\)), 2.16-2.04 (m, 2H, H\(_6\)), 1.71 (d, \(J = 7.1\) Hz, 3H, H\(_8\)), 0.99 (t, \(J = 7.4\) Hz, 3H, H\(_7\)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 167.2 (s, C\(_9\)), 147.2 (d, C\(_5\)), 134.1 (d, C\(_{11}\) or C\(_{12}\)), 132.1 (s, C\(_{10}\)), 123.5 (d, C\(_{11}\) or C\(_{12}\)), 108.1 (d, C\(_4\)), 85.1 (s, C\(_2\)), 81.8 (s, C\(_3\)), 37.9 (d, C\(_1\)), 26.2 (t, C\(_6\)), 20.4 (q, C\(_8\)), 12.9 (q, C\(_7\)). HRMS (ESI) m/z: calcd for C\(_{16}\)H\(_{15}\)NaNO\(_2\) [M + Na\(^+\)]: 276.0995, found: 276.1002.

4-Phenylbut-3-yn-2-amine\(^5\) (2a)

\(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.44-7.38 (m, 2H, H\(_{Ph}\)), 7.32-7.26 (m, 3H, H\(_{Ph}\)), 3.92 (q, \(J = 6.8\) Hz, 1H, H\(_1\)), 1.70 (s, 2H, NH\(_2\)), 1.44 (d, \(J = 6.8\) Hz, 3H, H\(_8\)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 131.6 (d, C\(_{Ph}\)), 128.3 (s, C\(_{Ph}\)), 128.0 (s, C\(_{Ph}\)), 123.3 (s, C\(_4\)), 94.0 (s, C\(_2\)), 81.6 (s, C\(_3\)), 39.4 (s, C\(_1\)), 24.5 (s, C\(_8\)).

1-Phenylpent-1-yn-3-amine (2b)

IR (neat): 3369, 2964, 2930, 1598, 1489, 1443, 1369, 1316, 871, 843, 754, 690 cm\(^{-1}\). \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.45-7.36 (m, 2H, H\(_{Ph}\)), 7.31-7.23 (m, 3H, H\(_{Ph}\)), 3.71 (dd, \(J = 7.5, 5.9\) Hz, 1H, H\(_1\)), 1.77-1.60 (m, 4H, H\(_8\) + NH\(_2\)), 1.07 (t, \(J = 7.4\) Hz, 3H, H\(_3\)). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 131.6 (d, C\(_{Ph}\)), 128.3 (d, C\(_{Ph}\)), 128.0 (d, C\(_{Ph}\)), 123.4 (s, C\(_4\)), 92.9 (s, C\(_2\)), 82.6 (s, C\(_2\)), 45.6

(d, C_1), 31.3 (t, C_8), 10.5 (q, C_9). **HRMS (ESI) m/z**: calcd for C_{11}H_{14}N [M]^+: 160.1121, found: 160.1118.

4-Methyl-1-phenylpent-1-yn-3-amine (2c)

![Diagram of 4-Methyl-1-phenylpent-1-yn-3-amine (2c)]

**IR** (neat): 2959, 2870, 1597, 1489, 1443, 1367, 1069, 855, 754, 690 cm\(^{-1}\). **\(^1\)H NMR** (400 MHz, CDCl\(_3\) \(\delta\) 7.45-7.39 (m, 2H, H\(_{Ph}\)), 7.32-7.27 (m, 3H, H\(_{Ph}\)), 3.63 (d, \(J = 5.4\) Hz, 1H, H\(_1\)), 1.92-1.82 (m, 1H, H\(_8\)), 1.74 (s, 2H, NH\(_2\)), 1.07 (d, \(J = 2.2\) Hz, 3H, H\(_9\)), 1.06 (d, \(J = 2.3\) Hz, 3H, H\(_9\)). **\(^{13}\)C NMR** (100 MHz, CDCl\(_3\) \(\delta\) 131.7 (d, C\(_{Ph}\)), 128.3 (d, C\(_{Ph}\)), 128.0 (d, C\(_{Ph}\)), 123.5 (s, C\(_4\)), 91.5 (s, C\(_2\)), 83.4 (s, C\(_3\)), 50.2 (d, C\(_1\)), 34.7 (d, C\(_8\)), 19.4 (q, C\(_9\)), 17.8 (q, C\(_9\)). **HRMS (ESI) m/z**: calcd for C\(_{12}\)H\(_{16}\)N [M]^+: 174.1277, found: 174.1274.

5-Methyl-1-phenylhex-1-yn-3-amine (2d)

![Diagram of 5-Methyl-1-phenylhex-1-yn-3-amine (2d)]

**IR** (neat): 2955, 2926, 2869, 1598, 1489, 1443, 1366, 1069, 914, 868, 821, 754, 690 cm\(^{-1}\). **\(^1\)H NMR** (400 MHz, CDCl\(_3\) \(\delta\) 7.45-7.37 (m, 2H, H\(_{Ph}\)), 7.32-7.25 (m, 3H, H\(_{Ph}\)), 3.81 (dd, \(J = 8.4, 6.7\) Hz, 1H, H\(_1\)), 1.99-1.84 (m, 1H, H\(_8\)), 1.72 (s, 2H, NH\(_2\)), 1.63-1.49 (m, 2H, H\(_8\)), 0.97 (dd, \(J = 6.6, 5.7\) Hz, 6H, H\(_{10}\)). **\(^{13}\)C NMR** (100 MHz, CDCl\(_3\) \(\delta\) 131.6 (d, C\(_{Ph}\)), 128.3 (d, C\(_{Ph}\)), 128.0 (d, C\(_{Ph}\)), 123.4 (s, C\(_4\)), 93.3 (s, C\(_2\)), 82.5 (s, C\(_3\)), 47.4 (t, C\(_8\)), 42.5 (d, C\(_1\)), 25.5 (d, C\(_8\)), 23.0 (q, C\(_{10}\)), 22.3 (q, C\(_{10'}\)). **HRMS (ESI) m/z**: calcd for C\(_{13}\)H\(_{16}\)N [M]^+: 188.1434, found: 188.1434.

1,4-Diphenylbut-3-yn-2-amine (2e)

![Diagram of 1,4-Diphenylbut-3-yn-2-amine (2e)]

**IR** (neat): 3028, 2921, 1734, 1597, 1489, 1442, 1071, 1028, 840, 750, 691 cm\(^{-1}\). **\(^1\)H NMR** (400 MHz, CDCl\(_3\) \(\delta\) 7.42-7.36 (m, 2H, H\(_{Ph}\)), 7.36-7.32 (m, 4H, H\(_{Ph}\)), 7.32-7.24 (m, 4H,
H$_{Ph}$), 4.05 (t, $J = 6.6$ Hz, 1H, H$_1$), 3.05 (dd, $J = 13.2, 6.4$ Hz, 1H, H$_8$), 2.98 (dd, $J = 13.2, 6.7$ Hz, 1H, H$_8$), 1.72 (s, 2H, NH$_2$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 137.6 (s, C$_9$), 131.6 (d, C$_{Ph}$), 129.8 (d, C$_{Ph}$), 128.5 (d, C$_{Ph}$), 158.4 (d, C$_{Ph}$), 128.2 (d, C$_{Ph}$), 126.9 (d, C$_{Ph}$), 123.2 (s, C$_4$), 92.0 (s, C$_2$), 83.7 (s, C$_3$), 45.5 (d, C$_1$), 44.4 (t, C$_8$).

HRMS (ESI) m/z: calcd for C$_{16}$H$_{16}$N [M]$^+$: 222.1277, found: 222.1275.

1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-amine$_6$ (2f)

IR (neat): 2929, 2835, 1607, 1509, 1245, 832, 755, 691 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.54 (d, $J = 8.7$ Hz, 2H, H$_9$), 7.49-7.43 (m, 2H, H$_{Ph}$), 7.34-7.29 (m, 3H, H$_{Ph}$), 6.92 (d, $J = 8.8$ Hz, 2H, H$_{10}$), 4.99 (s, 1H, H$_1$), 3.82 (s, 3H, H$_{12}$), 2.77 (s, 2H, NH$_2$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.2 (s, C$_{11}$), 134.4 (s, C$_8$), 131.7 (d, C$_{Ph}$), 128.4 (d, C$_{Ph}$), 128.3 (d, C$_{Ph}$), 128.1 (d, C$_9$), 123.2 (s, C$_4$), 114.1 (d, C$_{10}$), 91.6 (s, C$_2$), 84.3 (s, C$_3$), 55.4 (q, C$_{12}$), 47.5 (d, C$_1$). HRMS (ESI) m/z: calcd for C$_{16}$H$_{15}$NaNO [M + Na]$^+$: 260.1046, found: 260.1047.

4-(Naphthalen-1-yl)but-3-yn-2-amine (2g)

IR (neat): 3056, 2970, 2927, 1585, 1394, 1370, 1310, 1119, 858, 798, 771 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.35-8.31 (m, 1H, H$_{12}$), 7.87-7.83 (m, 1H, H$_7$ or H$_9$), 7.81 (d, $J = 8.3$ Hz, 1H, H$_7$ or H$_9$), 7.65 (dd, $J = 7.1, 1.2$ Hz, 1H, H$_5$ or H$_6$), 7.58 (ddd, $J = 8.3, 6.8, 1.4$ Hz, 1H, H$_{11}$), 7.52 (dd, $J = 8.1, 6.8, 1.4$ Hz, 1H, H$_{10}$), 7.42 (dd, $J = 8.3, 7.2$ Hz, 1H, H$_5$ or H$_6$), 4.10 (q, $J = 6.8$ Hz, 1H, H$_1$), 1.81 (s, 2H, NH$_2$), 1.57 (d, $J = 6.8$ Hz, 3H, H$_{14}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 133.4 (s, C$_{13}$ or C$_8$), 133.2 (s, C$_{13}$ or C$_8$), 130.3 (d, C$_{Ar}$), 128.5 (d, C$_{Ar}$), 128.3 (d, C$_{Ar}$), 126.7 (d, C$_{Ar}$), 126.4 (d, C$_{Ar}$), 126.2 (d, C$_{12}$), 125.3 (d, C$_{Ar}$), 120.9 (s, C$_4$), 99.1 (s, C$_2$), 79.7 (s, C$_3$), 39.8 (d, C$_1$), 24.8 (q, C$_{14}$). HRMS (ESI) m/z: calcd for C$_{14}$H$_{11}$ [MH-NH$_3$]$^+$: 179.0855, found: 179.0856.

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4-(Naphthalen-2-yl)but-3-yn-2-amine (2h)

IR (neat): 3265, 3057, 2976, 1634, 1544, 1493, 1369, 1259, 112, 859, 818, 742, 606 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.94 (s, 1H, H₁), 7.84 - 7.73 (m, 3H, H₆, H₈, H₁₁), 7.51 - 7.44 (m, 3H, H₅, H₉, H₁₀), 3.99 (q, J = 6.8 Hz, 1H, H₁), 1.84 (s, 2H, NH₂), 1.50 (d, J = 6.8 Hz, 3H, H₁₄).

¹³C NMR (100 MHz, CDCl₃) δ 133.1 (s, C₇ or C₁₂), 132.8 (s, C₇ or C₁₂), 131.4 (d, C₁₃), 128.6 (d, C₅, C₉ or C₁₀), 128.0 (d, C₆, C₈ or C₁₁), 127.8 (d, C₆, C₈ or C₁₁), 127.8 (d, C₆, C₈ or C₁₁) 126.6 (d, C₅, C₉ or C₁₀), 126.6 (d, C₅, C₉ or C₁₀), 120.6 (s, C₄), 94.0 (s, C₂), 82.2 (s, C₃), 39.6 (d, C₁), 24.5 (q, C₁₄). HRMS (ESI) m/z: calcd for C₁₄H₁₄N [M⁺]: 196.1121, found: 196.1118.

4-(4-Methoxyphenyl)but-3-yn-2-amine (2i)

IR (neat): 3365, 2969, 2930, 2836, 1606, 1507, 1442, 1287, 1244, 1173, 1029, 829 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.36 - 7.30 (m, 2H, H₅ or H₆), 6.84 - 6.78 (m, 2H, H₅ or H₆), 3.90 (q, J = 6.7 Hz, 1H, H₁), 3.78 (s, 3H, H₈), 1.72 (s, 2H, NH₂), 1.42 (d, J = 6.8 Hz, 3H, H₉).

¹³C NMR (100 MHz, CDCl₃) δ 159.4 (s, C₇), 133.0 (d, C₅ or C₆), 115.4 (s, C₄), 113.9 (d, C₅ or C₆), 92.5 (s, C₂), 81.4 (s, C₃), 55.3 (q, C₈), 39.5 (d, C₁), 24.7 (q, C₉). HRMS (ESI) m/z: calcd for C₁₁H₁₃NaNO [M + Na⁺]: 198.0889, found: 198.0887.

4-(3-Methoxyphenyl)but-3-yn-2-amine (2j)

IR (neat): 3366, 2970, 2931, 1597, 1574, 1481, 1426, 1315, 1285, 1201, 1163, 1039, 854, 780, 686 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 7.22 - 7.15 (m, 1H, H₉), 6.99 (dt, J = 7.6, 1.2 Hz, 1H, H₉), 6.94 - 6.92 (m, 1H, H₉), 6.84 (ddd, J = 8.3, 2.6, 1.0 Hz, 1H, H₉), 3.91 (q, J = 6.8 Hz, 1H, H₁), 3.78
(s, 3H, H$_{10}$), 1.77 (s, 2H, NH$_2$), 1.43 (d, J = 6.8 Hz, 3H, H$_{11}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.3 (s, C$_6$), 129.4 (d, C$_{Ph}$), 124.3 (s, C$_4$), 124.1 (d, C$_{Ph}$), 116.5 (d, C$_{Ph}$), 114.6 (d, C$_{Ph}$), 93.8 (s, C$_2$), 81.5 (s, C$_3$), 55.3 (q, C$_{10}$), 39.4 (d, C$_1$), 24.6 (q, C$_{11}$). HRMS (ESI) m/z: calcd for C$_{11}$H$_{14}$NO [M]$^+$: 176.1070, found: 176.1068.

4-(2-Methoxyphenyl)but-3-yn-2-amine (2k)

IR (neat): 2970, 2931, 1595, 1491, 1434, 1259, 1118, 1022, 750 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.37 (dd, J = 7.6, 1.7 Hz, 1H, H$_6$ or H$_9$), 7.25 (ddd, J = 8.3, 7.4, 1.7 Hz, 1H, H$_7$ or H$_8$), 6.91-6.81 (m, 2H, H$_6$ or H$_9$ + H$_7$ or H$_8$), 3.96 (q, J = 6.8 Hz, 1H, H$_1$), 3.85 (s, 3H, H$_{10}$), 1.81 (s, 2H, NH$_2$), 1.45 (d, J = 6.8 Hz, 3H, H$_{11}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.8 (s, C$_5$), 133.7 (d, C$_6$ or C$_9$), 129.5 (d, C$_7$ or C$_8$), 120.5 (d, C$_7$ or C$_8$), 112.3 (s, C$_4$), 110.6 (d, C$_6$ or C$_9$), 98.2 (s, C$_2$), 77.7 (s, C$_3$), 55.8 (q, C$_{10}$), 39.6 (d, C$_1$), 24.5 (q, C$_{11}$). HRMS (ESI) m/z: calcd for C$_{11}$H$_{14}$NO [M]$^+$: 176.1070, found: 176.1068.

4-(p-Tolyl)but-3-yn-2-amine (2l)

IR (neat): 2971, 2925, 1509, 1448, 1369, 1312, 1118, 1066, 878, 814 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.32-7.27 (m, 2H, H$_5$ or H$_6$), 7.11-7.07 (m, 2H, H$_5$ or H$_6$), 3.91 (q, J = 6.8 Hz, 1H, H$_1$), 2.33 (s, 3H, H$_8$), 1.86 (s, 2H, NH$_2$), 1.43 (d, J = 6.8 Hz, 3H, H$_9$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 138.1 (s, C$_7$), 131.5 (d, C$_5$ or C$_6$), 129.1 (d, C$_5$ or C$_6$), 120.2 (s, C$_4$), 93.3 (s, C$_2$), 81.7 (s, C$_3$), 39.5 (d, C$_1$), 24.6 (q, C$_9$), 21.5 (q, C$_8$). HRMS (ESI) m/z: calcd for C$_{11}$H$_{14}$N [M]$^+$: 160.1121, found: 160.1118.
4-(2,4-Difluorophenyl)but-3-yn-2-amine (2m)

IR (neat): 2976, 2932, 1617, 1590, 1502, 1265, 1143, 1098, 969, 850, 813, 651, 612 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.40-7.32 (m, 1H, H\(_9\)), 6.86-6.77 (m, 2H, H\(_6\)+H\(_8\)), 3.94 (q, \(J = 6.8\) Hz, 1H, H\(_1\)), 2.06 (s br, 2H, NH\(_2\)), 1.45 (d, \(J = 6.8\) Hz, 3H, H\(_{10}\)).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 163.1 (s, \(J = 252.0, 12.0\) Hz, C\(_5\) or C\(_7\)), 162.5 (s, \(J = 250.0, 11.0\) Hz, C\(_5\) or C\(_7\)), 134.3 (d, \(J = 10.0, 3.0\) Hz, C\(_9\)), 111.5 (d, \(J = 22.0, 4.0\) Hz, C\(_6\) or C\(_8\)), 108.1 (s, \(J = 16.0, 4.0\) Hz, C\(_4\)), 104.3 (d, C\(_6\) or C\(_8\)), 98.9 (s, \(J = 3.0, 2.0\) Hz, C\(_2\)), 75.1 (s, C\(_3\)), 39.6 (d, C\(_1\)), 24.4 (q, C\(_{11}\)).

HRMS (ESI) m/z: calcd for C\(_{10}\)H\(_{10}\)F\(_2\)N [M]\(^+\): 182.0776, found: 182.0776.

4-[3-(Trifluoromethyl)phenyl]but-3-yn-2-amine (2n)

IR (neat): 2976, 1588, 1486, 1431, 1331, 1166, 1123, 1070, 899, 800, 695, 656 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.66 (s, 1H, H\(_5\)), 7.55 (d, \(J = 7.7\) Hz, 1H, H\(_5\)), 7.52 (d, \(J = 7.9\) Hz, 1H, H\(_7\)), 7.40 (t, \(J = 7.8\) Hz, 1H, H\(_8\)), 3.93 (q, \(J = 6.8\) Hz, 1H, H\(_1\)), 1.76 (s, 2H, NH\(_2\)), 1.44 (d, \(J = 6.8\) Hz, 3H, H\(_{11}\)).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 134.7 (d, C\(_9\)), 130.9 (s, \(J = 32\) Hz, C\(_6\)), 128.8 (d, C\(_8\)), 128.5 (d, \(J = 4.0\) Hz, C\(_5\)), 124.6 (d, \(J = 4.0\) Hz, C\(_7\)), 124.3 (s, C\(_4\)), 123.8 (s, \(J = 271.0\) Hz, C\(_{10}\)), 95.6 (s, C\(_2\)), 80.3 (s, C\(_3\)), 39.5 (d, C\(_1\)), 24.4 (q, C\(_{11}\)).

HRMS (ESI) m/z: calcd for C\(_{11}\)H\(_{11}\)F\(_3\)N [M]\(^+\): 214.0838, found: 214.0836.

4-(Pyridin-2-yl)but-3-yn-2-amine (2o)

IR (neat): 3277, 2974, 2929, 2229, 1582, 1463, 1427, 1368, 1312, 1265, 1071, 889, 846, 777, 740 cm\(^{-1}\).

\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.55 (ddd, \(J = 4.9, 1.8, 1.0\) Hz, 1H, H\(_5\)), 7.62 (td, \(J = 7.7\),
1.8 Hz, 1H, H₆), 7.38 (dt, J = 7.8, 1.1 Hz, 1H, H₃), 7.20 (ddd, J = 7.6, 4.9, 1.2 Hz, 1H, H₇), 3.94 (q, J = 6.8 Hz, 1H, H₁), 1.93 (s, 2H, NH₂), 1.47 (d, J = 6.8 Hz, 3H, H₈). ¹³C NMR (100 MHz, CDCl₃) δ 150.0 (d, C₈), 143.4 (s, C₄), 136.3 (d, C₆), 127.0 (d, C₅), 122.9 (d, C₇), 93.9 (s, C₂), 81.4 (s, C₃), 39.4 (d, C₁), 24.0 (q, C₀). HRMS (ESI) m/z: calcd for C₉H₁₁N₂ [M]⁺: 147.0917, found: 147.0914.

(E)-Oct-5-en-3-yn-2-amine (2p)

IR (neat): 3283, 2966, 2933, 2874, 1657, 1552, 1376, 1085, 956 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 6.14 (dt, J = 15.9, 6.6 Hz, 1H, H₅), 5.47 (dq app, J = 15.9, 1.8 Hz, 1H, H₄), 3.82 (qd, J = 6.7, 1.6 Hz, 1H, H₁), 2.50 (s br, 2H, NH₂), 2.16-2.07 (m, 2H, H₁), 1.37 (d, J = 6.8 Hz, 3H, H₈), 1.01 (t, J = 7.5 Hz, 3H, H₇). ¹³C NMR (100 MHz, CDCl₃) δ 145.9 (d, C₅), 108.5 (d, C₄), 92.4 (s, C₂), 80.5 (s, C₃), 39.4 (d, C₁), 26.2 (t, C₆), 24.59 (q, C₈), 13.1 (q, C₇). HRMS (ESI) m/z: calcd for C₈H₁₄N [M]⁺: 124.1121, found: 124.1116.

8-[(tert-Butyldiphenylsilyl)oxy]oct-5-yn-4-amine (2r)

IR (neat): 2957, 2931, 2857, 1472, 1107, 822, 700, 612 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.76-7.66 (m, 4H, H₉Ph), 7.48-7.35 (m, 6H, H₈Ph), 3.78 (t, J = 7.0 Hz, 2H, H₅), 3.54 (t, J = 6.6 Hz, 1H, H₁), 2.48 (td, J = 7.0, 2.0 Hz, 2H, H₄), 1.76 (s, 2H, NH₂), 1.62-1.40 (m, 4H, H₁₂+H₁₃), 1.08 (s, 9H, H₁₁), 0.93 (t, J = 7.2 Hz, 3H, H₁₄). ¹³C NMR (100 MHz, CDCl₃) δ 135.7 (d, C₉Ph), 133.8 (s, C₆), 129.8 (d, C₈Ph), 127.8 (d, C₇Ph), 84.7 (s, C₂), 79.6 (s, C₃), 62.8 (t, C₅), 43.5 (d, C₁), 40.6 (t, C₁₂), 26.9 (q, C₁₁), 23.0 (t, C₄), 19.4 (t, C₁₃), 19.3 (s, C₁₀), 13.9 (q, C₁₄). HRMS (ESI) m/z: calcd for C₂₄H₃₄NOSi [M]⁺: 380.2404, found: 380.2410.
8-(Benzyloxy)oct-5-yn-4-amine (6)

IR (neat): 2957, 2931, 2868, 1605, 1573, 1454, 1361, 1099, 735, 697 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.40-7.25 (m, 5H, Haryl), 4.56 (s, 2H, H₆), 3.63-3.48 (m, 3H, H₁ + H₃), 2.51 (td, J = 7.1, 2.0 Hz, 2H, H₄), 1.69 (s, 2H, NH₂), 1.59-1.40 (m, 4H, H₁₁ + H₁₂), 0.93 (t, J = 7.2 Hz, 3H, H₁₃).

¹³C NMR (100 MHz, CDCl₃) δ 138.24 (s, C₇), 128.50 (d, Caryl), 127.76 (2d, 2Caryl), 84.79 (s, C₂), 79.24 (s, C₃), 73.02 (t, C₆), 68.78 (t, C₅), 43.50 (d, C₁), 40.61 (t, C₁₁), 20.23 (t, C₄), 19.39 (t, C₁₂), 13.92 (q, C₁₃). HRMS (ESI) m/z: calcd for C₁₅H₂₂NO [M⁺]: 232.1696, found: 232.1699.
\(N-(4\text{-Phenylbut-3-yn-2-yl})\text{acetamide (3a)}\)

\[\alpha\] = -163 (c 0.6, CHCl\(_3\), 91\% ee). \textbf{IR (neat):} 3290, 3057, 2981, 2932, 1643, 1542, 1372, 1134, 757, 691 cm\(^{-1}\). \textbf{\textit{H NMR} (400 MHz, CDCl\(_3\))}: 7.46-7.38 (m, 2H, H\(_{\text{Ph}}\)), 7.35-7.28 (m, 3H, H\(_{\text{Ph}}\)), 5.84 (s, 1H, NH), 5.05 (dq, \(J = 13.8, 6.9\) Hz, 1H, H\(_1\)), 2.02 (s, 3H, H\(_{10}\)), 1.49 (d, \(J = 6.8\) Hz, 3H, H\(_8\)).

\textbf{\textit{C NMR} (100 MHz, CDCl\(_3\)):} 169.15 (s, C\(_9\)), 131.76 (d, C\(_{\text{Ph}}\)), 128.4 (d, C\(_{\text{Ph}}\)), 128.3 (d, C\(_{\text{Ph}}\)), 122.7 (s, C\(_4\)), 89.5 (s, C\(_2\)), 82.2 (s, C\(_3\)), 37.7 (d, C\(_1\)), 23.3 (q, C\(_8\) or C\(_{10}\)), 22.6 (q, C\(_8\) or C\(_{10}\)). \textbf{HRMS (ESI)} \textit{m/z}: calcd for C\(_{12}\)H\(_{13}\)NaNO \([\text{M} + \text{Na}]^+: 210.0889\), found: 210.0887.

\textbf{SFC: OD-H}, Pressure = 150 bar, CO\(_2\)/\(i\)-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, t\(R\) = 2.4 min and t\(R\) = 2.8 min (major).
\[ \text{N-(1-Phenylpent-1-yn-3-yl)acetamide (3b)} \]

\[ \alpha \] \text{D} – 106 (c 1.2, CHCl}_3, 92\% \text{ ee).} \text{ IR (neat): } 3288, 3053, 2974, 2929, 1637, 1542, 1490, 1367, 1301, 1275, 1118, 948, 758, 715, 688, 600 \text{ cm}^{-1}. \text{ } \text{H NMR (400 MHz, CDCl}_3\text{)} \delta 7.46-7.38 (m, 2H, H}_{\text{Ph}}, 7.35-7.27 (m, 3H, H}_{\text{Ph}}, 5.98 (d, J = 7.4 \text{ Hz, 1H, NH}), 4.93 (td, J = 7.8, 6.0 \text{ Hz, 1H, H}_1), 2.03 (s, 3H, H}_{11}, 1.84-1.70 (m, 2H, H}_8, 1.06 (t, J = 7.4 \text{ Hz, 3H, H}_9). \text{ 13C NMR (100 MHz, CDCl}_3\text{)} \delta 169.2 (s, C}_{10}, 131.8 (d, C}_{\text{Ph}}, 128.5 (d, C}_{\text{Ph}}, 128.4 (d, C}_{\text{Ph}}, 122.8 (s, C}_{4}, 88.4 (s, C}_{2}, 83.23 (s, C}_{3}, 43.5 (d, C}_{1}, 29.3 (t, C}_{8}, 23.4 (q, C}_{11}, 10.2 (q, C}_{9}). \text{ HRMS (ESI) m/z: calcd for C}_{13}H_{15}NaNO [M + Na]^+: 224.1046, found: 224.1040. \text{ SFC: OD-H, Pressure = 150 bar, CO}_2/i-\text{PrOH = 92:8, Flow rate = 5 mL/min, UV = 254 nm, } t_R = 2.9 \text{ min and } t_R = 3.1 \text{ min (major).} \]
**N-(4-Methyl-1-phenylpent-1-yn-3-yl)acetamide (3c)**

$$\text{[a]}^{20\text{D}} = -135 \ (c \ 1.0, \ \text{CHCl}_3, \ 90\% \ \text{ee})$$

**IR (neat):** 3268, 2962, 2929, 1645, 1539, 1490, 1371, 1282, 1090, 755, 691, 601 cm\(^{-1}\).  **\(^1\)H NMR** (400 MHz, CDCl\(_3\)) \(\delta\) 7.47-7.36 (m, 2H, H\(_{\text{Ph}}\)), 7.34-7.23 (m, 3H, H\(_{\text{Ph}}\)), 6.18 (s br, 1H, NH), 4.88 (dd, \(J = 8.7, 5.5\) Hz, 1H, H\(_1\)), 2.03 (s, 3H, H\(_{11}\)), 2.02-1.94 (m, 1H, H\(_3\)), 1.04 (d, \(J = 6.7\) Hz, 6H, H\(_9\)).  **\(^13\)C NMR** (100 MHz, CDCl\(_3\)) \(\delta\) 169.3 (s, C\(_{10}\)), 131.8 (d, C\(_{\text{Ph}}\)), 128.3 (2d, C\(_{\text{Ph}}\)), 122.8 (s, C\(_4\)), 87.1 (s, C\(_2\)), 83.8 (s, C\(_3\)), 47.8 (d, C\(_1\)), 33.1 (d, C\(_8\)), 23.3 (q, C\(_{11}\)), 19.1 (q, C\(_9\)), 17.8 (q, C\(_9\)).  **HRMS (ESI) m/z:** calcd for C\(_{14}\)H\(_{18}\)NO [M]\(^+\): 216.1383, found: 216.1380.

**SFC:** OD-H, Pressure = 150 bar, CO\(_2\)/i-PrOH = 91:9, Flow rate = 5 mL/min, UV = 254 nm, t\(R\) = 2.7 min and t\(_R\) = 2.9 min (major).
N-(5-Methyl-1-phenylhex-1-yn-3-yl)acetamide (3d)

\[ \alpha \] D –120 (c 0.7, CHCl₃, 87% ee). IR (neat): 3261, 2956, 2870, 1647, 1541, 1490, 1370, 1287, 1146, 1087, 755, 690, 604 cm⁻¹. ¹H NMR (400 MHz, CDCl₃) δ 7.47-7.36 (m, 2H, H₇Ph), 7.35-7.28 (m, 3H, H₈Ph), 5.77 (d, \( J = 8.2 \) Hz, 1H, NH), 5.03 (dd, \( J = 16.1, 7.9 \) Hz, 1H, H₁), 2.03 (s, 3H, H₁₂), 1.92-1.79 (m, 1H, H₀), 1.68-1.57 (m, 2H, H₆), 0.99 (d, \( J = 6.6 \) Hz, 6H, H₁₀). ¹³C NMR (100 MHz, CDCl₃) δ 169.02 (s, C₁₁), 131.85 (d, C₇Ph), 128.42 (2d, C₈Ph), 122.83 (s, C₄), 88.84 (s, C₂), 82.98 (s, C₃), 45.38 (t, C₈), 40.65 (d, C₁), 25.39 (d, C₀), 23.47 (q, C₁₀), 22.97 (q, C₁₂), 22.14 (q, C₁₀'). HRMS (ESI) m/z: calcd for C₁₅H₁₉NaNO [M + Na]⁺: 252.1359, found: 252.1350. SFC: OD-H, Pressure = 150 bar, CO₂/i-PrOH = 92:8, Flow rate = 1 mL/min, UV = 254 nm, tR = 13.8 min and tR = 14.5 min (major).
N-(1,4-Diphenylbut-3-yn-2-yl)acetamide (3e)

$\left[\alpha\right]_{D}^{20} = -56 \ (c \ 0.9, \ \text{CHCl}_3, \ 86\% \ ee)$. IR (neat): 3258, 3057, 3031, 1636, 1540, 1490, 1427, 1372, 1288, 1269, 1097, 950, 913, 748, 689, 606 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36-7.17 (m, 10H, H$_{\text{Ph}}$), 5.79 (d, $J$ = 8.2 Hz, 1H, NH), 5.19 (ddd, $J$ = 8.3, 7.2, 4.9 Hz, 1H, H$_1$), 3.04 (dd, $J$ = 13.2, 4.9 Hz, 1H, H$_8$), 2.96 (dd, $J$ = 13.2, 7.1 Hz, 1H, H$_9$), 1.93 (s, 3H, H$_{14}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.1 (s, C$_{13}$), 136.6 (s, C$_9$), 131.7 (d, C$_{\text{Ph}}$), 130.1 (d, C$_{\text{Ph}}$), 128.5 (d, C$_{\text{Ph}}$), 128.4 (2d, C$_{\text{Ph}}$), 127.0 (d, C$_{\text{Ph}}$), 122.6 (s, C$_4$), 87.8 (s, C$_2$), 84.5 (s, C$_3$), 43.2 (d, C$_1$), 41.5 (t, C$_8$), 23.3 (q, C$_{14}$).

HRMS (ESI) $m/z$: calcd for C$_{18}$H$_{17}$NO [M + Na]$^+$: 286.12024, found: 286.11947. SFC: OD-H, Pressure = 150 bar, CO$_2$/i-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, tR = 4.4 min and tR = 5.5 min (major).
$N$-(1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-yl)acetamide (3f)

[α]$_D^{20}$ +3.7 ($c$ 0.75, CHCl$_3$, 7% ee). IR (neat): 3296, 3060, 2956, 1648, 1538, 1512, 1370, 1254, 1245, 1179, 1027, 824, 759, 688, 601 cm$^{-1}$. \textbf{1H NMR} (400 MHz, CDCl$_3$) $\delta$ 7.54-7.46 (m, 4H, H$_9$ + H$_{Ph}$), 7.36-7.31 (m, 3H, H$_{Ph}$), 6.91 (d, $J$ = 8.8 Hz, 2H, H$_{10}$), 6.21 (d, $J$ = 8.6 Hz, 1H, H$_1$), 5.99 (d, $J$ = 8.2 Hz, 1H, NH), 3.82 (s, 3H, H$_{12}$), 2.05 (s, 3H, H$_{14}$). \textbf{13C NMR} (100 MHz, CDCl$_3$) $\delta$ 168.8 (s, C$_{13}$), 159.6 (s, C$_{11}$), 131.9 (d, C$_{Ph}$), 131.4 (s, C$_8$), 128.7 (d, C$_{Ph}$), 128.5 (2d, C$_{Ph}$ + C$_9$), 122.6 (s, C$_4$), 114.20 (d, C$_{10}$), 87.4 (s, C$_2$), 84.7 (s, C$_3$), 55.5 (q, C$_{12}$), 44.8 (d, C$_1$), 23.5 (q, C$_{14}$). \textbf{HRMS (ESI)} m/z: calcd for C$_{18}$H$_{17}$NaNO$_2$ [M + Na]$^+$: 302.1151, found: 302.1145. \textbf{SFC}: AD-H, Pressure = 150 bar, CO$_2$/$i$-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, tR = 11.4 min and tR = 14.0 min (major).
$N$-(4-(Naphthalen-1-yl)but-3-yn-2-yl)acetamide (3g)

$[\alpha]^{20}_D -129$ (c 1.1, CHCl$_3$, 72% ee). IR (neat): 3285, 3056, 1635, 1537, 1280, 1138, 774, 715, 604 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.28 (d, $J = 8.4$ Hz, 1H, H$_{12}$), 7.79-7.86 (m, 2H, H$_7$ + H$_9$), 7.65 (dd, $J = 7.1$, 1.0 Hz, 1H, H$_5$ or H$_6$), 7.60-7.54 (m, 1H, H$_{11}$), 7.54-7.49 (m, 1H, H$_{10}$), 7.40 (dd, $J = 8.2$, 7.2 Hz, 1H, H$_5$ or H$_6$), 6.21 (d, $J = 7.0$ Hz, 1H, NH), 5.22 (dq, $J = 13.9$, 6.9 Hz, 1H, H$_1$), 2.04 (s, 3H, H$_{16}$), 1.59 (d, $J = 6.9$ Hz, 3H, H$_{14}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.2 (s, C$_{15}$), 133.4 (s, C$_{13}$ or C$_8$), 133.2 (s, C$_{13}$ or C$_8$), 130.7 (d, C$_5$ or C$_6$), 128.9 (d, C$_7$ or C$_9$), 128.4 (d, C$_7$ or C$_9$), 126.9 (d, C$_{11}$), 126.5 (d, C$_{10}$), 126.1 (d, C$_{12}$), 125.2 (d, C$_5$ or C$_6$), 120.3 (s, C$_4$), 94.4 (s, C$_2$), 80.4 (s, C$_3$), 38.0 (d, C$_1$), 23.4 (q, C$_{16}$), 22.8 (q, C$_{14}$). HRMS (ESI) $m/z$: calcd for C$_{16}$H$_{15}$NaNO [M + Na]$^+$: 260.1046, found: 260.1038. SFC: OD-H, Pressure = 150 bar, CO$_2$/i-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, tR = 9.3 min and tR = 9.9 min (major).
N-(4-(Naphthalen-2-yl)but-3-yn-2-yl)acetamide (3h)

$[\alpha]^{20}_{D} = -156 \ (c \ 1.1, \ CHCl_3, \ 96\% \ ee)$. IR (neat): 3292, 3050, 2983, 2931, 1631, 1537, 1367, 1271, 1124, 859, 817, 749, 698, 605 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.94 (s, 1H, H$_{13}$), 7.84-7.74 (m, 3H, H$_6$, H$_8$, H$_{11}$), 7.53-7.43 (m, 3H, H$_5$, H$_9$, H$_{10}$), 6.05 (d, $J = 7.2$ Hz, 1H, NH), 5.11 (dq, $J = 13.9, 6.9$ Hz, 1H, H$_1$), 2.04 (s, 3H, H$_{16}$), 1.53 (d, $J = 6.9$ Hz, 3H, H$_{14}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.1 (s, C$_{15}$), 133.0 (s, C$_7$ or C$_{12}$), 132.9 (s, C$_7$ or C$_{12}$), 131.7 (d, C$_{13}$), 128.5 (d, C$_5$, C$_9$ or C$_{10}$), 128.1 (d, C$_6$, C$_8$ or C$_{11}$), 127.8 (d, C$_6$, C$_8$ or C$_{11}$), 127.8 (d, C$_6$, C$_8$ or C$_{11}$), 126.8 (d, C$_5$, C$_9$ or C$_{10}$), 126.7 (d, C$_5$, C$_9$ or C$_{10}$), 119.9 (s, C$_4$), 89.8 (s, C$_2$), 82.7 (s, C$_3$), 37.9 (d, C$_1$), 23.4 (q, C$_{16}$), 22.7 (q, C$_{14}$). HRMS (ESI) $m/z$: calcd for C$_{16}$H$_{15}$NaNO $[M + Na]^+$: 260.1046, found: 260.1038.

SFC: OD-H, Pressure = 150 bar, CO$_2$/i-PrOH = 90:10, Flow rate = 6 mL/min, UV = 254 nm, $t_R = 6.2$ min (major) and $t_R = 6.9$ min.
$N$-$(4$-$(4$-Methoxyphenyl)$)but$-$3$-yn$-$2$-yl$acetamide (3i)

$[\alpha]^{20}_D$ $-162$ (c 1.0, CHCl$_3$, 93% ee). IR (neat): 3293, 3060, 2993, 2958, 1631, 1538, 1506, 1367, 1289, 1245, 1151, 1029, 835, 704, 635 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.40-7.29 (m, 2H, H$_5$ or H$_6$), 6.86-6.76 (m, 2H, H$_5$ or H$_6$), 5.93 (d, $J$ = 6.6 Hz, 1H, NH), 5.02 (dq, $J$ = 13.8, 6.9 Hz, 1H, H$_1$), 3.80 (s, 3H, H$_8$), 2.00 (s, 3H, H$_{11}$), 1.47 (d, $J$ = 6.8 Hz, 3H, H$_9$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.0 (s, C$_{10}$), 159.7 (s, C$_7$), 133.2 (d, C$_5$ or C$_6$), 114.7 (s, C$_4$), 114.0 (d, C$_5$ or C$_6$), 88.1 (s, C$_2$), 82.2 (s, C$_3$), 55.4 (d, C$_1$), 37.8 (q, C$_8$), 23.4 (q, C$_{11}$), 22.8 (q, C$_9$). HRMS (ESI) $m/z$: calcd for C$_{13}$H$_{15}$NaNO$_2$ [M + Na]$^+$: 240.0995, found: 240.0989. SFC: OD-H, Pressure = 150 bar, CO$_2$/i-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, tR = 3.3 min and tR = 3.7 min (major).
**N-(4-(3-Methoxyphenyl)but-3-yn-2-yl)acetamide (3j)**

[α]**D** –116 (c 0.6, CHCl₃, 88% ee). **IR** (neat): 3269, 3059, 2981, 2934, 1649, 1598, 1575, 1542, 1287, 1206, 1176, 1042, 784, 687 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.21 (dd, J = 8.1, 7.7 Hz, 1H, H₉), 7.01 (d, J = 7.6 Hz, 1H, H₉), 6.97-6.92 (m, 1H, H₉), 6.87 (dd, J = 8.3, 2.6 Hz, 1H, H₉), 5.81 (d, J = 6.0 Hz, 1H, NH), 5.05 (dq, J = 14.0, 6.9 Hz, 1H, H₁), 3.80 (s, 3H, H₁₀), 2.02 (s, 3H, H₁₃), 1.49 (d, J = 6.9 Hz, 3H, H₁₁). **¹³C NMR** (100 MHz, CDCl₃) δ 169.0 (s, C₁₂), 159.4 (s, C₆), 129.5 (d, C₉), 124.4 (d, C₈), 123.7 (s, C₄), 116.6 (d, C₇), 115.1 (d, C₉), 89.3 (s, C₂), 82.3 (s, C₃), 55.4 (q, C₁₀), 37.8 (d, C₁), 23.4 (q, C₁₃), 22.7 (q, C₁₁). **HRMS (ESI) m/z**: calcd for C₁₃H₁₅NaNO₂ [M + Na]⁺: 240.0995, found: 240.0989. **SFC**: OD-H, Pressure = 150 bar, CO₂/i-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, tR = 3.2 min and tR = 3.8 min (major).
**N-(4-(2-Methoxyphenyl)but-3-yn-2-yl)acetamide (3k)**

\[ \alpha \]^{20}_{D} -91 (c 1.1, CHCl\textsubscript{3}, 91% ee). **IR** (neat): 3265, 3067, 2979, 2932, 1635, 1546, 1493, 1433, 1369, 1277, 1259, 1112, 1026, 743 cm\textsuperscript{-1}. \(^1\)H NMR (400 MHz, CDCl\textsubscript{3}) \(\delta\) 7.37 (dd, \(J = 7.6, 1.7\) Hz, 1H, H\textsubscript{6} or H\textsubscript{9}), 7.31-7.24 (m, 1H, H\textsubscript{7} or H\textsubscript{8}), 6.89 (d, \(J = 7.5\) Hz, 1H, H\textsubscript{6} or H\textsubscript{9}), 6.85 (d, \(J = 8.4\) Hz, 1H, H\textsubscript{7} or H\textsubscript{8}), 6.06 (s, 1H, NH), 5.08 (dq, \(J = 13.8, 6.9\) Hz, 1H, H\textsubscript{1}), 3.85 (s, 3H, H\textsubscript{10}), 1.99 (s, 3H, H\textsubscript{13}), 1.49 (d, \(J = 6.8\) Hz, 3H, H\textsubscript{11}). \(^{13}\)C NMR (100 MHz, CDCl\textsubscript{3}) \(\delta\) 169.0 (s, C\textsubscript{12}), 160.0 (s, C\textsubscript{5}), 133.9 (d, C\textsubscript{6} or C\textsubscript{9}), 129.9 (d, C\textsubscript{7} or C\textsubscript{8}), 120.5 (d, C\textsubscript{7} or C\textsubscript{8}), 111.7 (s, C\textsubscript{4}), 110.7 (d, C\textsubscript{6} or C\textsubscript{9}), 93.5 (s, C\textsubscript{2}), 78.7 (s, C\textsubscript{3}), 55.8 (q, C\textsubscript{10}), 38.0 (d, C\textsubscript{1}), 23.3 (q, C\textsubscript{13}), 22.8 (q, C\textsubscript{11}). **HRMS (ESI) m/z**: calcd for C\textsubscript{13}H\textsubscript{15}NaNO\textsubscript{2} [M + Na]\textsuperscript{+}: 240.09950, found: 240.09891. **SFC**: OD-H, Pressure = 150 bar, CO\textsubscript{2}/i-PrOH = 90:10, Flow rate = 5 mL/min, UV = 254 nm, t\textsubscript{R} = 4.0 min and t\textsubscript{R} = 4.3 min (major).
N-(4-(p-Tolyl)but-3-yn-2-yl)acetamide (3l)

\[
\begin{align*}
\text{C}_7\text{H}_8\text{N} & \\
\text{O} & \\
\end{align*}
\]  

\[
\begin{align*}
[a]_D^{20} & \quad -157 \ (c \ 0.47, \ \text{CHCl}_3, \ 91\% \ ee) \ . \ \text{IR} \ (\text{neat}): \ 3297, \ 2988, \ 2933, \ 1635, \ 1537, \ 1440, \ 1372, \ 1270, \ 1133, \ 821, \ 692, \ 600 \ \text{cm}^{-1} \ . \\
\text{H NMR} \ (400 \ \text{MHz, CDCl}_3) \ & \ \delta \ 7.30 \ (d, \ J = 8.1 \ \text{Hz, 2H, H}_5 \ or \ H_6), \ 7.11 \ (d, \ J = 7.9 \ \text{Hz, 2H, H}_5 \ or \ H_6), \ 5.79 \ (d, \ J = 5.4 \ \text{Hz, 1H, NH}), \ 5.09-4.98 \ (m, \ 1H, H_1), \ 2.35 \ (s, \ 3H, H_8), \ 2.01 \ (s, \ 3H, H_{11}), \ 1.49 \ (d, \ J = 6.8 \ \text{Hz, 3H, H}_9) \ . \\
\text{C NMR} \ (100 \ \text{MHz, CDCl}_3) \ & \ \delta \ 169.0 \ (s, \ C_{10}), \ 138.6 \ (s, \ C_7), \ 131.7 \ (d, \ C_5 \ or \ C_6), \ 129.2 \ (d, \ C_5 \ or \ C_6), \ 119.6 \ (s, \ C_4), \ 88.8 \ (s, \ C_2), \ 82.6 \ (s, \ C_3), \ 37.9 \ (d, \ C_1), \ 23.5 \ (q, \ C_{11}), \ 22.8 \ (q, \ C_9), \ 21.6 \ (q, \ C_8) \ . \\
\text{HRMS (ESI)} \ m/z: \ \text{calcd for C}_{13}\text{H}_{15}\text{NaNO} \ [\text{M + Na}]^+: \ 224.10459, \ \text{found}: \ 224.10450 \ . \\
\text{SFC: OD-H, Pressure} \ = \ 150 \ \text{bar, CO}_2/i-\text{PrOH} = 90:10, \ \text{Flow rate} = 5 \ \text{mL/min, UV} = 254 \ \text{nm, tR} = 3.2 \ \text{min and tR} = 3.8 \ \text{min (major).} 
\]
N-(4-(2,4-Difluorophenyl)but-3-yn-2-yl)acetamide (3m)

\[
\begin{align*}
\text{IR (neat): } & 3278, 3069, 2983, 2931, 1633, 1541, 1504, 1263, 1143, 1101, 968, 854, 732, 604 \text{ cm}^{-1}. \\
\text{HRMS (ESI) m/z: calcd for } & C_{12}H_{11}F_2NaNO [M + Na]^+ : 246.0701, \text{ found: } 246.0670. \\
\text{SFC: OD-H, Pressure } & = 150 \text{ bar, CO}_2/i-\text{PrOH} = 97:3, \text{ Flow rate } = 5 \text{ mL/min, UV } = 254 \text{ nm, t}R = 6.5 \text{ min and } tR = 7.0 \text{ min (major).}
\end{align*}
\]
\( N-(4-(3-(\text{Trifluoromethyl})\text{phenyl})\text{but-3-yn-2-yl})\text{acetamide} \) (3n)

\[
\begin{align*}
\text{C} & \quad 169.3 (\text{s, C}_12), 134.9 (\text{d, C}_9), 130.9 (\text{s, J} = 32.0 \text{ Hz, C}_6), 128.9 (\text{d, C}_8), 128.6 (\text{d, J} = 4.0 \text{ Hz, C}_5), 125.0 (\text{d, J} = 4.0 \text{ Hz, C}_7), 123.8 (\text{s, J} = 271.0 \text{ Hz, C}_10), 123.7 (\text{s, C}_4), 91.2 (\text{s, C}_2), 80.8 (\text{s, C}_3), 37.6 (\text{d, C}_1), 23.2 (\text{q, C}_13), 22.3 (\text{q, C}_11). \\
\text{HRMS (ESI)} \quad m/z: \text{calcd for } \text{C}_{13}\text{H}_{12}\text{F}_3\text{NaNO} [\text{M + Na}]^+: 278.07632, \text{found: 278.07628. SFC: OD-H, Pressure = 150 bar, } \text{CO}_2/\text{i-PrOH} = 96:4, \text{Flow rate = 5 mL/min, UV = 254 nm, tR = 4.6 min and tR = 5.0 min (major).}
\end{align*}
\]
N-(4-(Pyridin-2-yl)but-3-yn-2-yl)acetamide (3o)

$[\alpha]^{20}_{D} = -105$ (c 1.2, CHCl$_3$, 91% ee). IR (neat): 3257, 3051, 2982, 2932, 2237, 1649, 1583, 1541, 1464, 1428, 1371, 1267, 1147, 973, 779, 741 cm$^{-1}$. $^1$H NMR (400 MHz, C$_6$D$_6$) $\delta$ 8.32 (ddd, $J = 4.9$, 1.8, 0.9 Hz, 1H, H$_8$), 7.06 (dt, $J = 7.8$, 1.1 Hz, 1H, H$_6$), 6.84 (td, $J = 7.7$, 1.8 Hz, 1H, H$_5$), 6.48 (ddd, $J = 7.6$, 4.9, 1.2 Hz, 1H, H$_7$), 6.03 (d, $J = 7.4$ Hz, 1H, NH), 5.24 (dq, $J = 13.9$, 6.9 Hz, 1H, H$_1$), 1.60 (s, 3H, H$_{11}$), 1.25 (d, $J = 6.9$ Hz, 3H, H$_9$). $^{13}$C NMR (100 MHz, C$_6$D$_6$) $\delta$ 168.4 (s, C$_{10}$), 150.2 (d, C$_8$), 143.8 (s, C$_4$), 135.8 (d, C$_5$), 127.2 (d, C$_6$), 122.7 (d, C$_7$), 90.7 (s, C$_2$), 82.2 (s, C$_3$), 37.4 (d, C$_1$), 22.8 (q, C$_{11}$), 22.1 (q, C$_9$). HRMS (ESI) $m/z$: calcd for C$_{11}$H$_{13}$N$_2$O [M]$^+$: 189.1022, found: 189.1027. SFC: OD-H, Pressure = 150 bar, CO$_2$/i-PrOH = 91:9, Flow rate = 5 mL/min, UV = 254 nm, tR = 4.5 min (major) and tR = 5.1 min.
**[(E)-N-(Oct-5-en-3-yn-2-yl)acetamide (3p)](attachment:PDF)***

\[
\begin{align*}
\text{O} & \\
\text{NH} & \\
\end{align*}
\]

[α]^{20}_D –126 (c 0.4, CHCl₃, 88% ee). **IR** (neat): 3270, 2965, 2932, 2363, 1650, 1544, 1452, 1372, 1277, 1183, 1120, 957 cm⁻¹. **¹H NMR** (400 MHz, C₆D₆) \(\delta\) 6.11 (dt, \(J = 15.8, 6.6\) Hz, 1H, H₅), 5.45 (dq, \(J = 15.9, 1.7\) Hz, 1H, H₄), 5.21-5.11 (m, 1H, H₁), 4.95 (s br, 1H, NH), 1.77 (pd, \(J = 7.5, 1.7\) Hz, 2H, H₆), 1.42 (s, 3H, H₁₀), 1.21 (d, \(J = 6.8\) Hz, 3H, H₈), 0.73 (t, \(J = 7.4\) Hz, 3H, H₇). **¹³C NMR** (100 MHz, C₆D₆) \(\delta\) 167.7 (s, C₉), 146.3 (d, C₅), 109.0 (d, C₄), 89.2 (s, C₂), 81.2 (s, C₃), 37.6 (d, C₁), 26.3 (t, C₆), 22.7 (2q, C₈ + C₁₀), 13.0 (q, C₇). **HRMS (ESI)** \(m/z\): calcld for C₁₀H₁₅NaNO \([M + Na]^+\): 188.1046, found: 188.1046. **SFC:** OD-H, Pressure = 150 bar, CO₂/i-PrOH = 93:7, Flow rate = 5 mL/min, UV = 254 nm, tR = 1.8 min and tR = 2.0 min (major).
N-(8-((tert-Butyldiphenylsilyl)oxy)oct-5-yn-4-yl)acetamide (3r)

$\left[\alpha\right]_{D}^{20} -38$ (c 1.28, CHCl$_3$, 91% ee). IR (neat): 3233, 2930, 1631, 1555, 1430, 1375, 1107, 1095, 915, 745, 705, 684, 609 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.74-7.63 (m, 4H, H$_2$Ph), 7.50-7.35 (m, 6H, H$_3$Ph), 5.65 (d, $J$ = 8.2 Hz, 1H, NH), 4.73-4.67 (m, 1H, H$_1$), 3.76 (t, $J$ = 6.8 Hz, 2H, H$_3$), 2.45 (td, $J$ = 6.9, 2.0 Hz, 2H, H$_4$), 1.94 (s, 3H, H$_{16}$), 1.67-1.51 (m, 2H, H$_{12}$), 1.49-1.36 (m, 2H, H$_{13}$), 1.07 (s, 9H, H$_{11}$), 0.92 (t, $J$ = 7.3 Hz, 3H, H$_{14}$). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 169.0 (s, C$_{15}$), 135.7 (d, C$_{Ph}$), 133.7 (s, C$_{6}$), 129.8 (d, C$_{Ph}$), 127.8 (d, C$_{Ph}$), 80.6 (s, C$_{2}$), 80.5 (s, C$_{3}$), 62.5 (t, C$_{5}$), 41.7 (d, C$_{1}$), 38.4 (t, C$_{12}$), 26.7 (q, C$_{11}$), 23.4 (q, C$_{16}$), 22.9 (t, C$_{4}$), 19.3 (s, C$_{10}$), 19.1 (t, C$_{13}$), 13.8 (q, C$_{14}$). HRMS (ESI) $m/z$: calcd for C$_{26}$H$_{35}$NaNO$_2$Si [M + Na]$^+$: 444.2329, found: 444.2332. SFC: OD-H, Pressure = 150 bar, CO$_2$/i-PrOH = 95:5, Flow rate = 5 mL/min, UV = 254 nm, tR = 7.2 min and tR = 7.7 min (major).
\[ \alpha \] $^2_{\text{D}}$ $-$63 (c 0.35, CHCl$_3$, 94% ee). IR (neat): 3279, 2957, 2874, 1645, 1543, 1367, 1283, 1106, 1031, 1002, 738, 695, 603 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.39-7.27 (m, 5H, H$_{\text{Ph}}$), 5.60 (d, $J = 8.1$ Hz, 1H, NH), 4.75-4.67 (m, 1H, H$_1$), 4.55 (s, 2H, H$_6$), 3.57 (t, $J = 7.0$ Hz, 2H, H$_4$), 2.50 (td, $J = 7.0$, 2.2 Hz, 2H, H$_4$), 1.97 (s, 3H, H$_{15}$), 1.70-1.52 (m, 2H, H$_{11}$), 1.50-1.37 (m, 2H, H$_{12}$), 0.93 (t, $J = 7.3$ Hz, 3H, H$_{13}$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 169.0 (s, C$_{14}$), 138.2 (s, C$_7$), 128.6 (d, C$_{\text{Ph}}$), 127.9 (d, C$_{\text{Ph}}$), 127.8 (d, C$_{\text{Ph}}$), 80.5 (s, C$_2$), 80.2 (s, C$_3$), 73.1 (t, C$_6$), 68.5 (t, C$_5$), 41.7 (d, C$_1$), 38.4 (t, C$_{11}$), 23.4 (q, C$_{15}$), 20.2 (t, C$_4$), 19.1 (t, C$_{12}$), 13.8 (q, C$_{13}$). HRMS (ESI) $m/z$: calcld for C$_{17}$H$_{23}$NaNO$_2$ [M + Na]$^+$: 296.1621, found: 296.1623. SFC: OD-H, Pressure = 150 bar, CO$_2$/i-PrOH = 92:8, Flow rate = 5 mL/min, UV = 254 nm, tR = 5.1 min and tR = 5.4 min (major).
(But-3-yn-1-yloxy)(tert-butyl)diphenylsilane

![Chemical Structure](image)

To a solution of but-3-yn-1-ol (1.2 mL, 16 mmol) in CH₂Cl₂ (40 mL) was added DMAP (0.5 g, 4 mmol) followed by Et₃N (19 mmol, 3.6 mL) and TBDPSCI (5.2 mL, 20 mmol). The reaction mixture was then stirred for 2 h at room temperature before a saturated aqueous solution of NaHCO₃ (20 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with CH₂Cl₂ (2 x 20 mL). The combined organic layers were eventually washed with brine (50 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was purified by flash column chromatography (PE/AcOEt = 98:2) to afford the corresponding protected alcohol (3.90 g, quant.).

**IR** (neat): 3305, 2931, 2857, 1427, 1105, 822, 737, 700, 613 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.76-7.70 (m, 4H, Hₗ₈), 7.50-7.39 (m, 6H, H₉), 3.83 (t, J = 7.1 Hz, 2H, H₄), 2.49 (td, J = 7.1, 2.7 Hz, 2H, H₃), 1.98 (t, J = 2.7 Hz, 1H, H₁), 1.10 (s, 9H, H₁₀). **¹³C NMR** (100 MHz, CDCl₃) δ 135.7 (d, Cₗ₈), 133.7 (s, C₅), 129.8 (d, C₉), 127.8 (d, C₉), 81.6 (s, C₂), 69.5 (s, C₁), 62.4 (t, C₄), 26.9 (q, C₁₀), 22.7 (t, C₃), 19.3 (s, C₉).

[(But-3-yn-1-yloxy)methyl]benzene

![Chemical Structure](image)

To a suspension of NaH (60 % wt, 1.8 g, 45 mmol) in DMF (35 mL) at 0 °C was added but-3-yn-1-ol (2.4 mL, 31 mmol) drop-wise. The reaction mixture was then stirred at 0 °C for 1.5 h, time after which a solution of BnBr (5.7 mL, 48 mmol) in DMF (5 mL) was added and stirring was continued for 2 h at room temperature. The reaction was quenched with a saturated aqueous solution of NH₄Cl and the aqueous phase was extracted twice with EtOAc (2 x 20 mL). The combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel (PE/AcOEt = 98:2) to afford the corresponding protected alcohol (92%, 4.57 g).

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IR (neat): 3293, 2864, 1454, 1362, 1098, 1028, 737, 697, 638 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.40-7.29 (m, 5H, H$_{\text{Ph}}$), 4.59 (s, 2H, H$_2$), 3.63 (t, $J = 6.9$ Hz, 2H, H$_4$), 2.53 (td, $J = 6.9$, 2.7 Hz, 2H, H$_3$), 2.02 (t, $J = 2.7$ Hz, 1H, H$_1$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 138.1 (s, C$_6$), 128.5 (d, C$_{\text{Ph}}$), 127.8 (2d, 2C$_{\text{Ph}}$), 81.4 (s, C$_1$), 73.1 (t, C$_5$), 69.4 (s, C$_2$), 68.2 (t, C$_4$), 20.0 (t, C$_3$).

$N$-(8-Hydroxyoctan-4-yl)acetamide (8)

To a degased solution of acetamide 7 (290 mg, 1.06 mmol) in EtOH (3 mL) was added Pd/C (10 wt%, 33 mg). After degasing the mixture under vacuum several times, the solution was then stirred overnight under positive pressure of H$_2$. The catalyst was eventually filtered through a plug of Celite$^\circledR$ and the solvent was evaporated under reduced pressure. The crude residue was engaged in the next step without further purification (100%, 198 mg).

$[\alpha]_{D}^{20} -6.6$ (c 1.2, CHCl$_3$). IR (neat): 3272, 2935, 2859, 1642, 1556, 1459, 1368, 1309, 1052, 749, 616 cm$^{-1}$. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.56 (d, $J = 8.1$ Hz, 1H, NH), 3.94 (s br, 1H, H$_1$), 3.63 (t, $J = 6.2$ Hz, 2H, H$_3$), 2.36 (s br, 1H, OH), 2.00 (s, 3H, H$_{10}$), 1.61-1.28 (m, 10H, H$_2$ + H$_3$ + H$_4$ + H$_6$ + H$_7$), 0.91 (t, $J = 7.1$ Hz, 3H, H$_8$). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 170.3 (s, C$_9$), 62.6 (t, C$_5$), 49.3 (d, C$_1$), 37.6 (t, C$_2$ or C$_3$ or C$_4$ or C$_6$ or C$_7$), 35.1 (t, C$_2$ or C$_3$ or C$_4$ or C$_6$ or C$_7$), 32.6 (t, C$_2$ or C$_3$ or C$_4$ or C$_6$ or C$_7$), 23.5 (q, C$_{10}$), 22.1 (t, C$_2$ or C$_3$ or C$_4$ or C$_6$ or C$_7$), 19.3 (t, C$_2$ or C$_3$ or C$_4$ or C$_6$ or C$_7$), 14.1 (q, C$_8$). HRMS (ESI) $m/z$: calcd for C$_{10}$H$_{21}$NaNO$_2$ [M + Na]$^+$: 210.1464, found: 210.1462.

5-Acetamidoctyl-4-methylbenzenesulfonate

To a solution of alcohol 8 (55 mg, 0.29 mmol) in CH$_2$Cl$_2$ (2 mL), was added DMAP (7 mg, 0.06 mmol) followed by Et$_3$N (50 µL, 0.35 mmol) and TsCl (67 mg, 0.35 mmol). The reaction mixture was then stirred for 4 h at room temperature before a saturated aqueous solution of NaHCO$_3$ (5 mL) was added. The organic layer was then separated and the aqueous phase was extracted twice with CH$_2$Cl$_2$ (2 x 10 mL). The combined organic layers were eventually washed.
with brine (15 mL), dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel (PE/Et₂O = 80:20) to afford 5-acetamidoctyl-4-methylbenzenesulfonate as colorless oil (79 mg, 80%).

\[ [\alpha]^{20}_D \approx -6.5 \, (c \ 0.87, CHCl_3) \]

**IR** (neat): \(3280, 2955, 1642, 1543, 1355, 1174, 924, 814, 662 \text{ cm}^{-1}\).

**H NMR** (400 MHz, CDCl₃) \(\delta\): 7.76 (d, \(J = 8.3 \text{ Hz}, 2\text{H}, H_{Ph}\)), 7.34 (d, \(J = 8.1 \text{ Hz}, 2\text{H}, H_{Ph}\)), 5.42 (d, \(J = 8.6 \text{ Hz}, 1\text{H}, NH\)), 4.05-3.93 (m, 2H, H₅), 3.85 (s, 1H, H₁), 2.44 (s, 3H, H₁₀), 1.95 (s, 3H, H₁₅), 1.72-1.56 (m, 2H, H₄), 1.43-1.24 (m, 8H, H₂+H₃+H₁₁+H₁₂), 0.87 (t, \(J = 6.9 \text{ Hz}, 3\text{H}, H_{13}\)).

**C NMR** (100 MHz, CDCl₃) \(\delta\): 169.9 (s, C₁₄), 144.9 (s, C₆), 133.1 (s, C₉), 130.0 (d, C₆H₅), 127.9 (d, C₆H₅), 70.5 (t, C₃), 48.8 (s, C₁), 37.4 (t, C₂ or C₃ or C₁₁ or C₁₂), 34.5 (t, C₂ or C₃ or C₁₁ or C₁₂), 28.7 (t, C₄), 23.5 (q, C₁₅), 21.8 (t, C₂ or C₃ or C₁₁ or C₁₂), 21.7 (q, C₁₀), 19.2 (t, C₂ or C₃ or C₁₁ or C₁₂), 14.1 (q, C₁₃).

**HRMS** (ESI) \(m/z\): calcd for C₁₇H₂₇NaNO₄S [M + Na]^+: 364.1553, found: 364.1554.

**N-acetyl (S)-coniine (9)**

To a solution of 5-acetamidoctyl-4-methylbenzenesulfonate (40 mg, 0.12 mmol) in THF (4 mL) was NaH (60 % wt, 8 mg, 0.18 mmol) and the resulting mixture was stirred at reflux for 2 h, time after which we lowered the temperature to room temperature and slowly added water (4 mL). The aqueous phase was extracted twice with EtOAc (2 x 10 mL) and the combined organic layers were dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure. The crude residue was finally purified by flash column chromatography over silica gel (PE/EtOAc = 70:30) to afford \(N\)-acetyl (S)-coniine 9 as colorless oil (15 mg, 74%). The spectroscopic and physical data of 9 were identical with those reported in the literature for \(N\)-acetyl (S)-coniine \{[\alpha]^{20}_D +46.0 \, (c \ 0.65, CHCl₃); \text{lit.} \,[\alpha]^{22}_D +46.9 \, (c \ 0.4, CHCl₃)\}.⁹

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