

Supporting Information for

Ag(I)-Promoted homo-dimerization of 2-(alkynoyl)alkynylbenzenes *via* a [4+2] cycloaddition of benzopyrylium ions: Access to structurally unique naphthalenes

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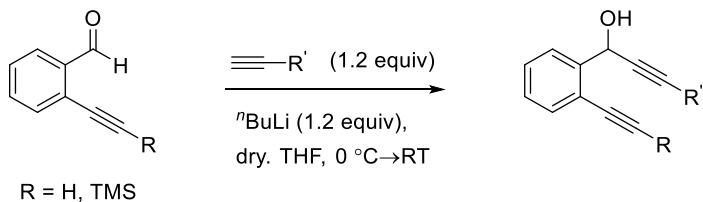
Tamilnadu, INDIA-600036

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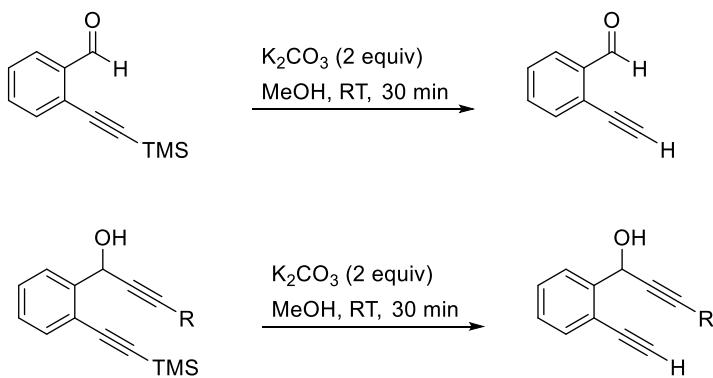
Experimental Section

General procedure A: For the Synthesis of (alkyl) diynol by treating with lithium acetylide



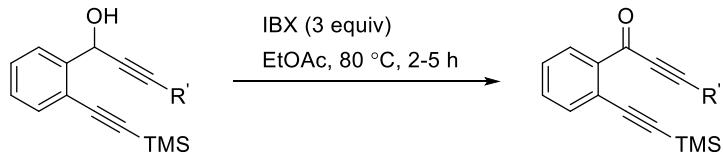
To an ice-cold solution of alkyne (1.2 equiv.) in anhydrous THF under nitrogen atmosphere was added $^n\text{BuLi}$ (1.2 equiv. in THF, 1.6 M in hexanes), after 30 minutes added 2-alkynylbenzaldehyde (1 equiv. in THF) at 0 °C and the reaction mixture was stirred for 1-3 h at 0 °C. Reaction progress was monitored by the thin layer chromatography (TLC) analysis. Reaction mixture was diluted with saturated aq. NH_4Cl solution (10 mL) and extracted with ethyl acetate (2×20 mL). The combined organic layer was washed with brine (10 mL) and dried over Na_2SO_4 . Evaporation of the solvent and purification of the crude mixture by flash column chromatography (9:1, hexane: EtOAc) gave the corresponding secondary alcohols.

General procedure B: For trimethylsilane deprotection



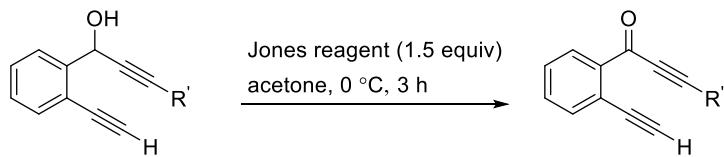
The corresponding TMS alcohol was taken in a round bottom flask equipped with stir bar. Then MeOH and Potassium carbonate (2 equiv.) was added, and stirred the reaction mixture for 30 min at RT. The reaction mixture was diluted with CH_2Cl_2 (10 mL) and water. Aqueous layer was extraxed with CH_2Cl_2 (2×10 mL). The combined organic layers were washed with brine, and dried over Na_2SO_4 . The solvent was removed under reduced pressure. The crude material was purified by flash column chromatography using hexane/ethyl acetate mixture as eluent to yield (89-96%) the corresponding terminal alkyne.

General procedure C: For synthesis of alkynone by employing IBX reagent



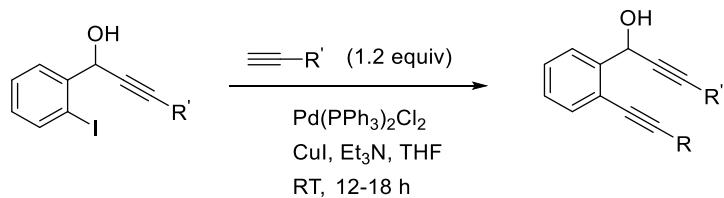
To the solution of alcohol (1 equiv.) dissolved in anhydrous EtOAc under nitrogen atmosphere, was added IBX (3 equiv.) and stirred the reaction mixture at 80 °C for 2-5 h. Reaction progress was monitored by the thin layer chromatography (TLC) analysis. The reaction was filtered through a plug of silica gel, using EtOAc as solvent to remove benzoic acid salts. The crude material was purified by flash column chromatography using hexane/ethyl acetate mixture as eluent to yield (80-90%) the corresponding ketones.

General procedure D: For synthesis of alkynone by employing Jones reagent



To an ice cold solution of alcohol (1 equiv.) dissolved in anhydrous acetone under nitrogen atmosphere, added Jones reagent (3 M solution in water and H₂SO₄), (1.5 equiv.) stirred the reaction mixture for 3 h at same temperature. Reaction progress was monitored by the thin layer chromatography (TLC) analysis. The reaction mixture was diluted with EtOAc (10 mL) and water. The crude material was purified by flash column chromatography using hexane/ethyl acetate mixture as eluent to yield (90-95%) the corresponding ketones.

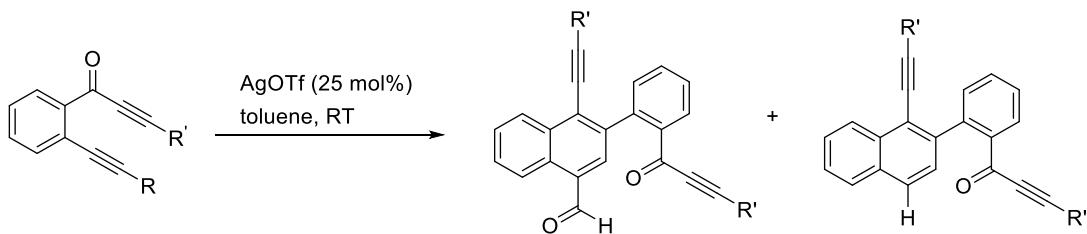
General procedure E: For synthesis of (aryl) diynols by employing Sonogashira cross coupling



The substituted 2-iodo compound (1 equiv.) and alkyne (1.2 equiv.) were taken in to a clean, anhydrous round bottom flask equipped with stir bar. Then anhydrous THF and Et₃N were added under nitrogen atmosphere and cooled the flask to 0 °C. Subsequently, CuI (15 mol%), and Pd(PPh₃)₂Cl₂ (1 mol%) were added to the reaction flask, and stirred the reaction mixture for 30 min at 0 °C. The reaction mixture was then allowed to warm to room temperature (RT) and continued the stirring for 14 h at RT. Reaction progress was monitored by the thin layer

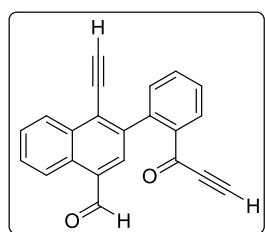
chromatography (TLC) analysis. The reaction was quenched with saturated aq. NH₄Cl solution and extracted with ethyl acetate (EtOAc). The combined organic layers were washed with brine, and dried (Na₂SO₄). The solvent was removed under reduced pressure. The crude material was purified by flash column chromatography using hexane/ethyl acetate mixture as eluent to yield the corresponding coupled product.

General procedure F: Ag-Promoted homo-dimerization of 2-(alkynonyl)alkynylbenzenes via a [4+2]cycloaddition



To a solution of the 2-(alkynonyl)alkynylbenzene in anhydrous toluene (2 mL/0.1 mmol) was added silver triflate (AgOTf) 25 mol% then the reaction mixture was stirred at RT. After completion of the reaction (by TLC analysis), water and EtOAC were added to reaction mixture and extracted with EtOAC. The combined organic layers were washed with the brine, and dried (Na₂SO₄). Evaporation of the solvent under reduced pressure and purification of the crude material by flash column chromatography using hexane-ethyl acetate mixture as eluent yielded the corresponding naphthalene products.

4-[*(Trimethylsilyl)ethynyl*]-3-{2-[3-(*trimethylsilyl*)propioloyl]phenyl}-1-naphthaldehyde (6a**)**

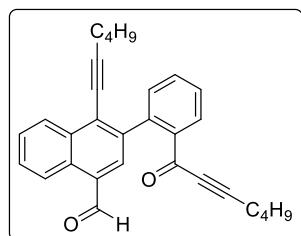


Followed general procedure F: The 2-(alkynonyl)alkynylbenzene¹ **5a** (120 mg, 0.53 mmol), in toluene (10 mL) and AgOTf (34 mg, 0.13 mmol) were stirred for 6 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave inseparable mixture **6a TMS/6a** (81 mg) as a yellow liquid. Inseparable mixture **6a TMS/6a** (81 mg, 0.17 mmol), in anhydrous THF (10 mL) and TBAF (0.5 mL, 0.53 mmol) were stirred for 0.5 h at 0 °C. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **6a** (36 mg, 22%, 0.11 mmol) as a yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 10.4 (1 H, s), 9.28 (1 H, d, *J* = 8.2 Hz), 8.50 (1 H, d, *J* = 8.2 Hz), 8.28 (1 H, d, *J* = 7.7 Hz), 7.89 (1 H, s), 7.76-7.67 (3 H, m), 7.65-7.55 (1 H, m), 7.45 (1 H, d, *J* = 7.5 Hz), 3.54 (1 H, s) and 3.16 (1 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.9,

177.7, 142.0, 140.5, 136.9, 136.1, 134.1, 133.3, 131.9, 131.0, 129.5, 129.4, 128.6, 128.2, 127.1, 125.1, 124.9, 116.0, 89.7, 81.1, 80.8 and 80.1 ppm. **IR** (neat): 2924, 2856, 2091, 1682, 1642, 1234 and 638 cm⁻¹. **HR ESI-MS**: [C₂₂H₁₃O₂]⁺ = [M+H]⁺ requires 309.0916; found 309.0910. **TLC**: R_f = 0.2 (4:1, hexane:EtOAc).

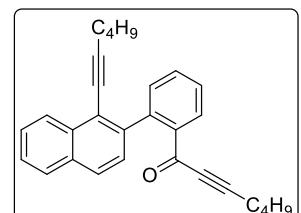
3-[2-(Hept-2-ynoyl)phenyl]-4-(hex-1-yn-1-yl)-1-naphthaldehyde (6b)



Followed general procedure F: The 2-(alkynonyl) alkynylbenzene² **5b** (80 mg, 0.38 mmol), in toluene (5 mL) and AgOTf (25 mg, 0.09 mmol) were stirred for 5 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **6b** (17 mg, 0.08 mmol, 23%) as a yellow liquid along with eluent (9:1 hexane:EtOAc) gave **7b** (52 mg, 0.247 mmol, 65%) as a yellow liquid.

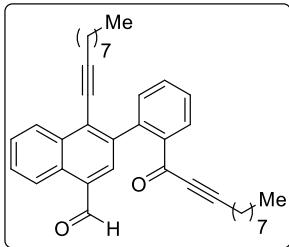
¹H NMR (400 MHz, CDCl₃): δ = 10.3 (1H, s), 9.28 (1 H, d, *J* = 8.2 Hz), 8.48 (1 H, d, *J* = 7.9 Hz), 8.12 (1 H, J = 6.9 Hz), 7.91 (1 H, s), 7.73-7.61 (3 H, m), 7.54 (1 H, t, *J* = 7.4 Hz), 7.42 (1 H, d, *J* = 7.3 Hz), 2.37 (2 H, t, *J* = 6.8 Hz), 1.92 (2 H, m), 1.58 (2 H, m), 1.45-1.37 (2 H, m), 1.16-1.10 (4 H, m), 0.82 (3 H, t, *J* = 7.3 Hz) and 0.79-0.71 (3 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 193.0, 178.8, 141.0, 137.8, 137.7, 134.0, 132.3, 131.6, 130.6, 129.9, 129.7, 129.2, 128.1, 127.9, 127.8, 127.4, 125.0, 104.8, 97.3, 81.2, 78.0, 30.3, 29.6, 22.0, 21.8, 19.7, 18.8, 13.7 and 13.5 ppm. **IR** (neat): 2957, 2928, 2210, 1690, 1644, 1554, 1507, 1255 and 914 cm⁻¹. **HR ESI-MS**: [C₃₀H₂₈O₂]⁺ = [M+H]⁺ requires 420.2089; found 420.2076. **TLC**: R_f = 0.3 (9:1, hexane:EtOAc).

1-[2-[1-(Hex-1-yn-1-yl)naphthalen-2-yl]phenyl}hept-2-yn-1-one (7b)



¹H NMR (400 MHz, CDCl₃): δ = 8.38 (1 H, dd, *J* = 8.2 & 0.69 Hz), 8.02 (1 H, dd, *J* = 7.7 & 1.3 Hz), 7.84 (1 H, d, *J* = 7.4 Hz), 7.80 (1 H, d, *J* = 8.3 Hz), 7.61-7.55 (2 H, m), 7.53 (1 H, dd, *J* = 7.3 & 1.3 Hz), 7.51-7.46 (2 H, m), 7.41 (1 H, m), 2.35 (2 H, t, *J* = 6.9 Hz), 1.87-1.78 (2 H, m), 1.45-1.37 (4 H, m), 1.21 (2 H, t, *J* = 7.1 Hz), 0.83 (4 H, t, *J* = 7.2 Hz), 0.72 and (4 H, t, *J* = 7.0 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.5, 142.4, 141.6, 138.1, 133.6, 132.6, 131.9, 131.7, 129.8, 128.0, 127.6, 127.5, 127.2, 127.0, 126.9, 126.4, 121.1, 100.1, 97.0, 81.3, 77.8, 30.5, 29.5, 22.0, 21.8, 19.5, 18.7, 13.7 and 13.5 ppm. **IR** (neat): 3058, 2958, 2210, 1643, 1265 and 737 cm⁻¹. **HR ESI-MS**: [C₂₉H₂₈O]⁺ = [M+H]⁺ requires 392.2140; found 392.2126. **TLC**: R_f = 0.5 (9:1, hexane:EtOAc).

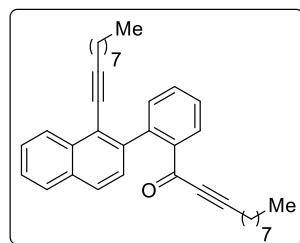
4-(Dec-1-yn-1-yl)-3-[2-(undec-2-ynoyl)phenyl]-1-naphthaldehyde (7c)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **5c** (60 mg, 0.22 mmol), in toluene (5 mL) and AgOTf (14 mg, 0.05 mmol) were stirred for 6 h at RT. Purification by flash column chromatography (19:1 hexane:EtOAc) gave **6c** (14 mg, 0.04 mmol, 25%) as a yellow liquid along with eluent (9:1 hexane:EtOAc) gave **7c** (24 mg, 0.09 mmol, 41 %) as a yellow liquid.

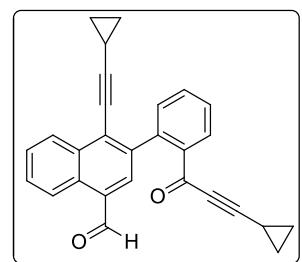
¹H NMR (400 MHz, CDCl₃): δ = 10.3 (1 H, s), 9.28 (1 H, d, *J* = 8.2 Hz), 8.48 (1 H, d, *J* = 7.9 Hz), 8.12 (1 H, d, *J* = 7.6 Hz), 7.91 (1 H, s), 7.75-7.59 (3 H, m), 7.53 (1 H, t, *J* = 7.2 Hz), 7.42 (1 H, d, *J* = 7.1 Hz), 2.37 (2 H, t, *J* = 6.3 Hz), 2.04-1.83 (2 H, m), 1.64-1.53 (2 H, m), 1.48-1.38 (2 H, m), 1.31-1.17 (19 H, m) and 0.93-0.83 (7 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 193.0, 178.8, 140.9, 137.8, 137.6, 133.9, 132.3, 131.5, 130.5, 129.8, 129.6, 129.2, 128.1, 127.7, 127.4, 125.0, 104.9, 97.3, 81.2, 78.0, 31.9, 31.8, 29.3, 29.2, 29.1, 29.0, 28.9, 28.8, 28.2, 27.5, 22.8, 22.7, 20.0, 19.1 and 14.2 ppm. **IR** (neat): 3059, 2928, 2213, 1695, 1639, 1460, 1262 and 739 cm⁻¹. **HR ESI-MS**: [C₃₈H₄₄O₂]⁺ = [M+H]⁺ requires 532.3392; found 532.3378. **TLC**: R_f = 0.2 (9:1, hexane:EtOAc).

1-{2-[1-(Dec-1-yn-1-yl)naphthalen-2-yl]phenyl}undec-2-yn-1-one (**7c**)



¹H NMR (400 MHz, CDCl₃): δ = 8.38 (1 H, d, *J* = 8.02 Hz), 8.02 (1 H, d, *J* = 7.4 Hz), 7.86-7.78 (2 H, m), 7.55-7.41 (5 H, m), 7.25 (1 H, s), 2.34 (2 H, t, *J* = 6.8 Hz), 1.30-1.24 (13 H, m), 1.24-1.16 (13 H, m) and 0.88 (6 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.4, 142.4, 141.5, 138.1, 133.5, 132.6, 131.9, 131.7, 129.8, 128.0, 127.6, 127.5, 127.2, 127.0, 126.8, 126.3, 121.0, 100.2, 97.1, 81.3, 77.8, 32.0, 31.9, 29.8, 29.3, 29.2, 29.1, 29.0, 28.9, 28.8, 28.5, 27.5, 22.8, 22.7, 19.8, 19.0 and 14.2 ppm. **IR** (neat): 3060, 2857, 2211, 1647, 1596, 1460, 1281 and 756 cm⁻¹. **HR ESI-MS**: [C₃₇H₄₄O]⁺ = [M+H]⁺ requires 504.3392; found 504.3378. **TLC**: R_f = 0.5 (19:1, hexane:EtOAc).

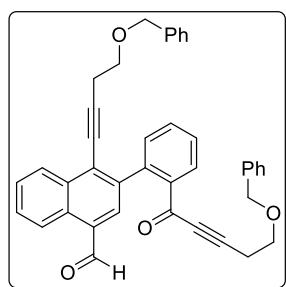
4-(Cyclopropylethynyl)-3-[2-(3-cyclopropylpropioloyl)phenyl]-1-naphthaldehyde (**6d**)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene³ **5d** (200 mg, 1.03 mmol), in toluene (20 mL) and AgOTf (66 mg, 0.25 mmol) were stirred for 3 days at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **6d** (75 mg, 0.38 mmol, 46%) as a yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 10.35 (1 H, s), 9.27 (1 H, d, *J* = 8.1 Hz), 8.44 (1 H, d, *J* = 8.0 Hz), 8.05 (1 H, d, *J* = 7.6 Hz), 7.90 (1 H, s), 7.74-7.58 (3 H, m), 7.52 (1 H, t, *J* = 7.4 Hz), 7.38 (1 H, d, *J* = 7.3 Hz), 1.42-1.34 (1 H, m), 0.97-0.88 (1 H, m), 0.83-0.77 (2 H, m), 0.73-0.49 (4 H, m) and 0.42-0.25 (2 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 193.0, 178.5, 140.7, 137.9, 137.7, 133.8, 132.2, 131.3, 130.1, 129.7, 129.6, 129.2, 128.1, 128.0, 127.7, 127.4, 125.0, 108.2, 101.8, 77.3, 73.0, 9.6, 9.58, 9.22, 9.13, 0.8 and -0.3 ppm. **IR** (neat): 2926, 2855, 2208, 1687, 1640, 1263, 1061, 1036 and 921 cm⁻¹. **HR ESI-MS**: [C₂₈H₂₁O₂]⁺ = [M+H]⁺ requires 389.1542; found 389.1529. **TLC**: R_f = 0.3 (9:1, hexane:EtOAc).

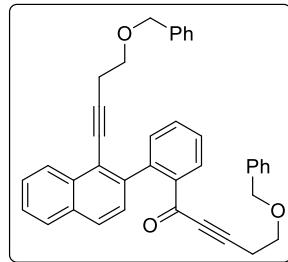
4-[4-(Benzylxy)but-1-yn-1-yl]-3-{2-[5-(benzyloxy)pent-2-ynoyl]phenyl}-1-naphthaldehyde (6e)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **5e** (100 mg, 0.34 mmol), in dry toluene (8 mL) and AgOTf (22 mg, 0.08 mmol) were stirred for 24 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **6e** (20 mg, 0.07 mmol, 21%) as a yellow liquid along with eluent (9:1 hexane:EtOAc) gave **7e** (50 mg, 0.17 mmol, 50 %) as a yellow liquid.

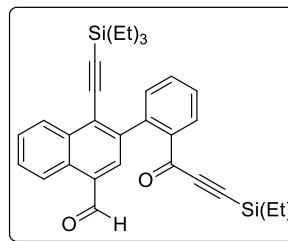
¹H NMR (400 MHz, CDCl₃): δ = 10.3 (1 H, s), 9.26 (1 H, d, *J* = 8.4 Hz), 8.47 (1 H, d, *J* = 8.2 Hz), 8.10 (1 H, d, *J* = 7.34 Hz), 7.88 (1 H, s), 7.69 (1 H, t, *J* = 7.2 Hz), 7.59 (2 H, t, *J* = 7.4 Hz), 7.47 (1 H, t, *J* = 7.0 Hz), 7.40 (1 H, d, *J* = 7.4 Hz), 7.32-7.24 (10 H, m), 4.46 (2 H, s), 4.35 (2 H, s), 3.59-3.38 (2 H, m), 3.26-3.15 (2 H, m), 2.67 (2 H, t, *J* = 6.8 Hz) and 2.26 (2 H, t, *J* = 6.8 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 193.0, 178.5, 140.9, 140.6, 138.0, 137.7, 137.6, 137.4, 133.8, 133.7, 132.5, 131.5, 130.6, 130.1, 130.0, 129.4, 129.3, 128.49, 128.47, 128.1, 127.9, 127.8, 127.75, 127.71, 127.4, 127.2, 124.9, 101.1, 93.4, 81.6, 78.7, 73.0, 72.9, 68.0, 66.9, 21.4 and 20.4 ppm. **IR** (neat): 2923, 2863, 2219, 1688, 1649, 1500, 1249, 1100 and 739 cm⁻¹. **HR ESI-MS**: [C₄₀H₃₂O₄]⁺ = [M+H]⁺ requires 576.2301; found 576.2287. **TLC**: R_f = 0.3 (9:1, hexane:EtOAc).

5-(Benzylxy)-1-{2-[1-(4-(benzyloxy)but-1-yn-1-yl)naphthalen-2-yl}phenyl}pent-2-yn-1-one (7e)



¹H NMR (400 MHz, CDCl₃): δ = 8.37 (1 H, d, *J* = 7.7 Hz), 8.11 (4 H, d, *J* = 7.7 Hz), 7.99 (1 H, d, *J* = 7.6 Hz), 7.83-7.76 (2 H, m), 7.59 (2 H, t, *J* = 7.1 Hz), 7.52-7.44 (6 H, m), 7.41-7.38 (2 H, m), 7.30-7.28 (2 H, m), 4.47 (2 H, s), 4.28 (2 H, s), 3.56-3.44 (2 H, m), 3.12-2.97 (2 H, m), 2.65 (2 H, t, *J* = 7.0 Hz) and 2.14 (2 H, t, *J* = 6.3 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.2, 142.1, 141.5, 138.2, 137.9, 137.8, 133.7, 133.4, 132.4, 132.1, 131.6, 129.8, 128.54, 128.50, 1258.4, 128.0, 127.8, 127.77, 127.71, 127.6, 127.4, 127.0, 126.9, 126.5, 120.7, 96.5, 92.9, 81.7, 78.7, 73.0, 72.8, 68.3, 66.9, 21.2 and 20.3 ppm. **IR** (neat): 3058, 2926, 2862, 1701, 1645, 1595, 1273, 1101 and 705 cm⁻¹. **HR ESI-MS**: [C₃₉H₃₂O₃]⁺ = [M+H]⁺ requires 548.2351; found 548.2338. **TLC**: R_f = 0.6 (9:1, hexane:EtOAc).

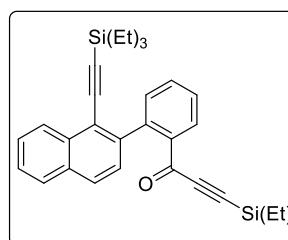
4-[(Triethylsilyl)ethynyl]-3-{2-[3-(triethylsilyl)propioloyl]phenyl}-1-naphthaldehyde (**6f**)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **5f** (145 mg, 0.541 mmol), in toluene (10 mL) and AgOTf (35 mg, 0.25 mmol) were stirred for 1 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **6f** (85 mg, 0.31 mmol, 59%) as yellow liquid along with eluent (4:1 hexane:EtOAc) gave **7f** (38 mg, 0.14 mmol, 27%).

¹H NMR (400 MHz, CDCl₃): δ = 10.35 (1 H, s), 9.30 (1 H, d, *J* = 8.1 Hz), 8.52 (1 H, d, *J* = 8.05 Hz), 8.22 (1 H, d, *J* = 7.55 Hz), 7.87 (1 H, s), 7.74-7.66 (2 H, m), 7.64 (1 H, t, *J* = 7.4 Hz), 7.56 (1 H, t, *J* = 7.5 Hz), 7.45 (1 H, d, *J* = 7.44 Hz), 0.88 (9 H, t, *J* = 7.9 Hz), 0.83 (9 H, t, *J* = 7.8 Hz), 0.56 (6 H, q, *J* = 7.9 Hz) and 0.39 (6 H, q, *J* = 7.9 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.9, 177.9, 141.9, 140.9, 137.5, 136.4, 133.9, 132.6, 131.8, 131.5, 130.3, 129.5, 129.3, 128.3, 128.0, 127.3, 126.4, 125.1, 106.5, 103.2, 102.3, 99.1, 7.50, 7.30, 4.20 and 3.73 ppm. **IR** (neat): 2951, 2885, 2146, 1689, 1645, 1582, 1461, 1235 and 1012 cm⁻¹. **HR ESI-MS**: [C₃₄H₄₁O₂Si₂]⁺ = [M+H]⁺ requires 537.2645; found 537.2654. **TLC**: R_f = 0.4 (4:1, hexane:EtOAc).

3-(Triethylsilyl)-1-{2-{1-[(triethylsilyl)ethynyl]naphthalen-2-yl}phenyl}prop-2-yn-1-one

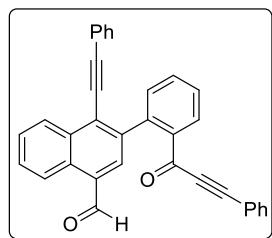


(**7f**)

¹H NMR (400 MHz, CDCl₃): δ = 8.42 (1 H, d, *J* = 8.3 Hz), 8.10 (1 H, d, *J* = 7.7 Hz), 7.87-7.79 (3 H, m), 7.59 (2 H, t, *J* = 7.8 Hz), 7.50-7.46 (2 H, m), 7.36 (1 H, d, *J* = 8.4 Hz), 0.89 (9 H, t, *J* = 7.9 Hz), 0.79 (9 H, t, *J* = 7.9 Hz), 0.55 (6 H, q, *J* = 7.9 Hz) and 0.32 (6 H, q, *J* = 7.8 Hz) ppm. **¹³C NMR**

(100 MHz, CDCl₃): δ = 178.6, 142.6, 142.3, 137.1, 133.5, 132.6, 132.1, 132.0, 130.8, 128.2, 128.1, 127.7, 127.6, 127.2, 126.9, 126.5, 119.9, 103.3, 102.9, 101.5, 98.6, 7.57, 7.31, 4.40 and 3.72 ppm. **IR** (neat): 2955, 2922, 2880, 2148, 1645, 1457, 1271, 1012 and 738 cm⁻¹. **HR ESI-MS**: [C₃₃H₄₁OSi₂]⁺ = [M+H]⁺ requires 509.2696; found 509.2705. **TLC**: R_f = 0.6 (4:1, hexane:EtOAc).

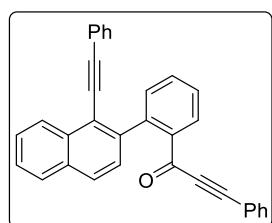
4-(Phenylethynyl)-3-[2-(3-phenylpropioloyl)phenyl]-1-naphthaldehyde (9a)



Followed general procedure F: The 2-(alkynonyl) alkynylbenzene⁴ **8a** (70 mg, 0.23 mmol), in toluene (5 mL) and AgOTf (15 mg, 0.05 mmol) were stirred for 2 h at RT. Purification by flash column chromatography (19:1 hexane:EtOAc) gave **9a** (24 mg, 0.1 mmol, 25%) as a yellow liquid along with eluent (9:1 hexane:EtOAc) gave **10a** (21 mg, 0.08 mmol, 45%) as a yellow liquid.

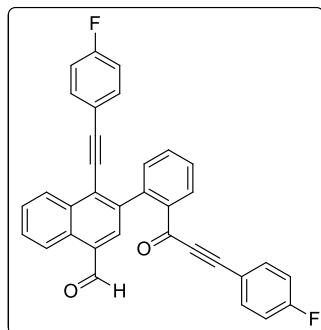
¹H NMR (400 MHz, CDCl₃): δ = 10.3 (1H, s), 9.24 (1 H, d, *J* = 6.7 Hz), 8.57 (1 H, d, *J* = 6.5 Hz), 8.27 (1 H, d, *J* = 7.2 Hz), 8.03 (1 H, s), 7.75-7.61 (4 H, m), 7.53 (1 H, d, *J* = 7.0 Hz), 7.36-7.23 (6 H, m), 7.16 (2 H, t, *J* = 6.9 Hz), 7.05 (2 H, d, *J* = 6.9 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.9, 178.7, 141.1, 140.6, 137.9, 137.3, 133.6, 132.7, 132.6, 131.8, 130.6, 130.5, 130.4, 129.7, 129.4, 129.1, 128.5, 128.4, 128.3, 128.0, 127.3, 126.7, 125.1, 122.6, 119.8, 102.4, 93.6, 88.5 and 86.7 ppm. **IR** (neat): 3059, 2918, 2855, 2198, 1744, 1735, 1692, 1279 and 752 cm⁻¹. **HR ESI-MS**: [C₃₄H₂₀O₂]⁺ = [M+H]⁺ requires 460.1463; found 460.1449. **TLC**: R_f = 0.2 (19:1, hexane:EtOAc). **M.P**: 117-120 °C.

3-Phenyl-1-{2-[1-(phenylethynyl)naphthalen-2-yl]phenyl}prop-2-yn-1-one (10a)



¹H NMR (400 MHz, CDCl₃): δ = 8.48 (1 H, d, *J* = 7.9 Hz), 8.18 (1 H, *J* = 7.5 Hz), 7.87 (1 H, d, *J* = 8.4 Hz), 7.83 (1 H, d, *J* = 8.0 Hz), 7.67 (1 H, t, *J* = 7.3 Hz), 7.61-7.49 (5 H, m), 7.37-7.31 (2 H, m), 7.27-7.22 (4 H, m), 7.12 (2 H, t, *J* = 7.3 Hz) and 6.97 (2 H, d, *J* = 7.3 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.2, 142.1, 141.9, 139.4, 138.2, 133.3, 132.7, 132.3, 131.9, 131.6, 130.15, 130.11, 128.5, 128.3, 128.26, 128.20, 127.9, 127.6, 127.2, 126.8, 126.6, 123.4, 120.1, 120.0, 114.2, 98.6, 93.2, 88.7 and 87.0 ppm. **IR** (neat): 3060, 2926, 2854, 2196, 1637, 1299 and 757 cm⁻¹. **HR ESI-MS**: [C₃₃H₂₁O]⁺ = [M+H]⁺ requires 433.1514; found 433.1502. **TLC**: R_f = 0.5 (19:1, hexane:EtOAc).

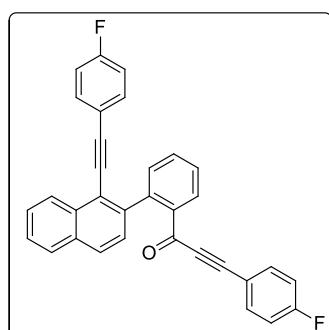
4-[(4-Fluorophenyl)ethynyl]-3-{2-[3-(4-fluorophenyl)propioloyl]phenyl}-1-naphthaldehyde (9b**)**



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene⁵ **8b** (100 mg, 0.31 mmol), in toluene (6 mL) and AgOTf (20 mg, 0.07 mmol) were stirred for 3.5 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **9b** (8 mg, 0.03 mmol, 11%) as a yellow liquid along with eluent (9:1 hexane:EtOAc) gave **10b** (52 mg, 0.20 mmol, 68%) as a yellow liquid.

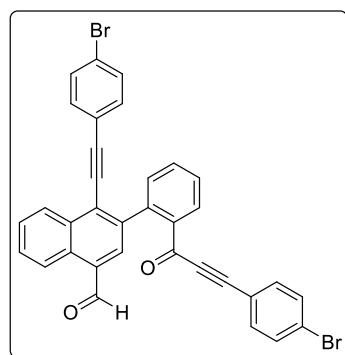
¹H NMR (400 MHz, CDCl₃): δ = 10.3 (1 H, s), 9.22 (1 H, d, *J* = 7.7 Hz), 8.52 (1 H, d, *J* = 7.6 Hz), 8.23 (1 H, d, *J* = 7.6 Hz), 8.03 (1 H, s), 7.73-7.61 (4 H, m), 7.52 (1 H, d, *J* = 7.3 Hz), 7.30 (2 H, t, *J* = 6.3 Hz), 7.00 (2 H, t, *J* = 5.5 Hz), 6.94 (2 H, t, *J* = 8.4 Hz) and 6.85 (2 H, t, *J* = 8.4 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.7, 178.5, 163.6 (d, *J* = 253.66 Hz), 162.9 (d, *J* = 251.3 Hz), 141.0, 140.6, 137.8, 137.1, 134.75 (d, *J* = 8.56 Hz), 133.75 (d, *J* = 8.53 Hz), 133.5, 132.8, 131.8, 130.6, 130.4, 129.7, 129.4, 128.5, 128.1, 127.1, 126.6, 125.1, 118.72 (d, *J* = 3.55 Hz), 115.81 (d, *J* = 22.08), 115.85 (d, *J* = 22.16 Hz), 101, 92.5, 88.4, 86.5 ppm. **IR** (neat): 3058, 2199, 1689, 1641, 1508, 1228, 840 and 738 cm⁻¹. **HR ESI-MS**: [C₃₄H₁₈F₂O₂]⁺ = [M+H]⁺ requires 496.1275; found 496.1261. **TLC**: R_f = 0.2 (9:1, hexane:EtOAc). **M.P**: 122-125 °C.

(4-Fluorophenyl)-1-{2-{1-[(4-fluorophenyl)ethynyl]naphthalen-2-yl}phenyl}prop-2-yn-1-one (10b**)**



¹H NMR (400 MHz, CDCl₃): δ = 8.44 (1 H, d, *J* = 8.1 Hz), 8.14 (1 H, d, *J* = 7.5 Hz), 7.87 (1 H, d, *J* = 8.3 Hz), 7.83 (1 H, d, *J* = 8.0 Hz), 7.68 (1 H, t, *J* = 7.1 Hz), 7.63-7.49 (5 H, m), 7.31 (2 H, t, *J* = 5.9 Hz), 7.02-6.8 (4 H, m) and 6.80 (2 H, t, *J* = 8.3 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.1, 163.58 (d, *J* = 252.50 Hz), 162.63 (d, *J* = 248.65 Hz), 142.0, 141.8, 138.2, 134.88 (d, *J* = 8.85 Hz), 133.48 (d, *J* = 8.38 Hz), 133.2, 132.7, 132.4, 131.9, 129.8, 128.5, 128.0, 127.6, 127.3, 126.75 (d, *J* = 3.72 Hz), 120.1, 119.51 (d, *J* = 3.58 Hz), 116.13 (d, *J* = 3.65 Hz), 115.69 (d, *J* = 22.19 Hz), 115.66 (22.03 Hz), 97.64, 92.2, 88.60, 86.71 ppm. **IR** (neat): 3057, 2927, 2854, 2199, 1643, 1596, 1507, 1265 and 1231 cm⁻¹. **HR ESI-MS**: [C₃₃H₁₈F₂O]⁺ = [M+H]⁺ requires 468.1306; found 468.1326. **TLC**: R_f = 0.6 (9:1, hexane:EtOAc).

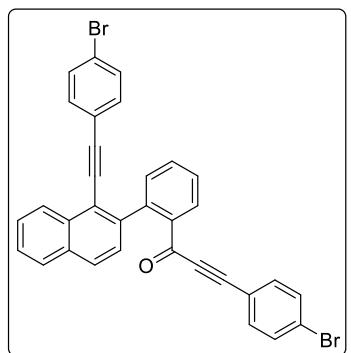
4-[(4-Bromophenyl)ethynyl]-3-{2-[3-(4-bromophenyl)propioloyl]phenyl}-1-naphthaldehyde (9c)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8c** (125 mg, 0.32 mmol), in dry toluene (6 mL) and AgOTf (21 mg, 0.08 mmol) were stirred for 6 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **9c** (59 mg, 0.19 mmol, 60%) as a yellow liquid along with eluent (4:1 hexane:EtOAc) gave **10c** (13 mg, 0.04 mmol, 18%) as a yellow liquid.

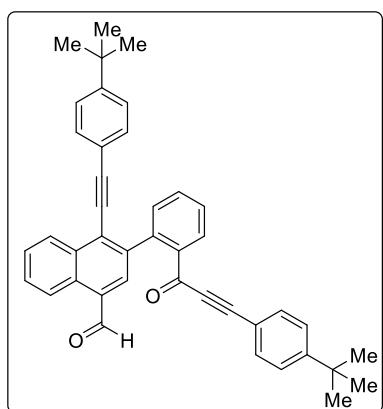
¹H NMR (400 MHz, CDCl₃): δ = 10.4 (1 H, s), 9.23 (1 H, d, *J* = 7.7 Hz), 8.50 (1 H, d, *J* = 7.6 Hz), 8.21 (1 H, d, *J* = 7.4 Hz), 8.03 (1 H, s), 7.74-7.61 (4 H, m), 7.52 (1 H, d, *J* = 7.1 Hz), 7.3 (2 H, d, *J* = 7.7 Hz), 7.30 (2 H, d, *J* = 7.8 Hz), 7.15 (2 H, d, *J* = 7.7 Hz), 6.84 (2 H, d, *J* = 7.7 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.7, 178.4, 141.1, 140.5, 137.8, 136.9, 133.7, 133.5, 133.1, 132.8, 131.8, 131.79, 131.76, 130.8, 130.4, 129.7, 129.5, 128.5, 128.2, 127.1, 126.3, 125.3, 125.1, 123.5, 121.5, 118.5, 101.2, 92.2, 89.2 and 87.8 ppm. **IR** (neat): 3057, 2985, 2927, 2199, 1713, 1644, 1485, 1423, 1264 and 740 cm⁻¹. **HR ESI-MS**: [C₃₄H₁₉Br₂O₂]⁺ = [M+H]⁺ requires 616.9752; found 616.9729. **TLC**: R_f = 0.4 (4:1, hexane:EtOAc). **M.P**: 122-125 °C.

3-(4-Bromophenyl)-1-{2-{1-[(4-bromophenyl)ethynyl]naphthalen-2-yl}phenyl}prop-2-yn-1-one (10c)



¹H NMR (400 MHz, CDCl₃): δ = 8.41 (1 H, d, *J* = 8.0 Hz), 8.12 (1 H, d, *J* = 7.3 Hz), 7.93-7.77 (2 H, m), 7.67 (1 H, t, *J* = 7.0 Hz), 7.63-7.49 (5 H, m), 7.37 (2 H, d, *J* = 7.8 Hz), 7.24 (2 H, d, *J* = 6.8 Hz), 7.17 (2 H, d, *J* = 7.8 Hz) and 6.73 (2 H, d, *J* = 7.8 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.0, 142.0, 141.9, 138.2, 133.8, 133.1, 132.9, 132.7, 132.5, 131.9, 131.64, 131.60, 129.8, 128.8, 128.3, 128.0, 127.5, 127.4, 126.8, 126.7, 124.9, 122.7, 122.3, 119.9, 118.9, 97.6, 91.8, 89.4 and 88.2 ppm. **IR** (neat): 3056, 2925, 2859, 1642, 1471, 1268 and 740 cm⁻¹. **HR ESI-MS**: [C₃₃H₁₉Br₂O]⁺ = [M+H]⁺ requires 588.9803; found 588.9779. **TLC**: R_f = 0.6 (4:1, hexane:EtOAc). **M.P**: 208-210 °C.

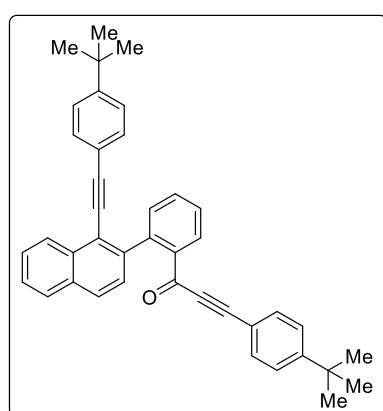
4-{{[4-(tert-Butyl)phenyl]ethynyl}-3-{2-[3-[4-(tert-butyl)phenyl]propioloyl]phenyl}-1-naphthaldehyde (9d)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8d** (67 mg, 0.18 mmol), in toluene (3 mL) and AgOTf (11 mg, 0.04 mmol) were stirred for 7 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **9d** (22 mg, 0.07 mmol, 44%) as a yellow liquid, **10d** (5 mg, 0.01 mmol, 5%).

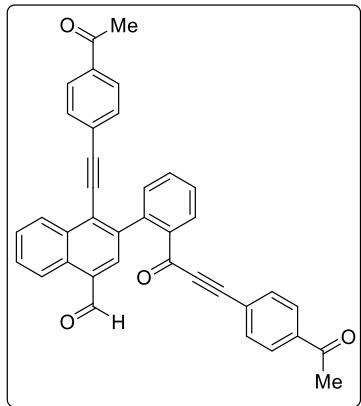
¹H NMR (400 MHz, CDCl₃): δ = 10.38 (1 H, s), 9.22 (1 H, d, *J* = 5.7 Hz), 8.56 (1 H, d, *J* = 5.2 Hz), 8.26 (1 H, d, *J* = 6.9 Hz), 8.02 (1 H, s), 7.72-7.61 (4 H, m), 7.54-7.50 (1 H, m), 7.33-7.24 (4 H, m), 7.18 (2 H, d, *J* = 7.6 Hz), 7.02 (2 H, d, *J* = 7.8 Hz) and 1.26 (18 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.9, 178.8, 154.0, 152.5, 141.0, 140.7, 138.1, 137.5, 133.7, 132.5, 131.9, 131.6, 130.6, 130.4, 129.8, 129.3, 128.4, 127.9, 127.3, 127.0, 125.4, 125.1, 119.7, 116.8, 102.9, 94.3, 88.4, 86.3, 35.0, 34.9 31.2 and 31.1 ppm. **IR** (neat): 2963, 2861, 2196, 1688, 1509, 1300, 1268, 1013 and 749 cm⁻¹. **HR ESI-MS**: [C₄₁H₃₆O]⁺ = [M+H]⁺ requires 544.2766; found 544.2752. **TLC**: R_f = 0.3 (4:1, hexane:EtOAc).

3-[4-(tert-Butyl)phenyl]-1-{2-[1-[[4-(tert-butyl)phenyl]ethynyl]naphthalen-2-yl}phenyl}-prop-2-yn-1-one (10d**)**



¹H NMR (400 MHz, CDCl₃): δ = 8.39 (1 H, d, *J* = 8.0 Hz), 8.09 (1 H, d, *J* = 7.6 Hz), 7.76 (2 H, t, *J* = 8.8 Hz), 7.56 (1 H, t, *J* = 7.2 Hz), 7.51-7.38 (5 H, m), 7.24-7.14 (4 H, m), 7.06 (2 H, d, *J* = 7.8 Hz), 6.88 (2 H, d, *J* = 7.8 Hz) and 1.19 ,1.16 (18 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.3, 153.7, 151.5, 142.1, 141.8, 138.3, 133.4, 132.6, 132.1, 132.0, 131.3, 128.25, 128.22, 127.8, 127.6, 127.1, 126.9, 126.5, 125.3, 125.2, 120.5, 120.1, 117.1, 114.2, 98.8, 93.9, 88.6, 86.5, 35.0, 34.8, 31.2 and 31.1 ppm. **IR** (neat): 3057, 2964, 2858, 2361, 2195, 1643 and 1267 cm⁻¹. **HR ESI-MS**: [C₄₁H₃₈O]⁺ = [M+H]⁺ requires 545.2844; found 545.2843. **TLC**: R_f = 0.5 (4:1, hexane:EtOAc).

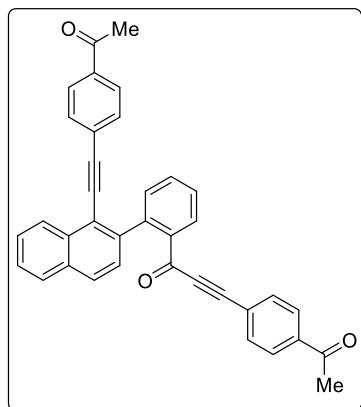
4-[(4-Acetylphenyl)ethynyl]-3-{2-[3-(4-acetylphenyl)propioloyl]phenyl}-1-naphth-aldehyde (9e**)**



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8e** (120 mg, 0.34 mmol), in dry toluene (6 mL) and AgOTf (22 mg, 0.08 mmol) were stirred for 13 h at RT. Purification by flash column chromatography (2:1 hexane:EtOAc) gave **9e** (60 mg, 0.22 mmol, 65%) as yellow liquid along with eluent (2:1 hexane:EtOAc) gave **10e** (22 mg, 0.08 mmol, 25%) as a yellow liquid.

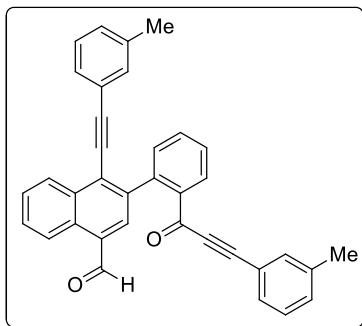
¹H NMR (400 MHz, CDCl₃): δ = 10.4 (1 H, s), 9.21 (1 H, s), 8.53 (1 H, s), 8.35-8.15 (1 H, m), 8.09-7.97 (1 H, m), 7.88-7.61 (8 H, m), 7.58-7.48 (1 H, m), 7.46-7.31 (2 H, m), 7.16-6.95 (2 H, m), 2.55 (3 H, s) and 2.53 (3 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 197.1, 197.0, 192.6, 178.3, 141.5, 140.5, 137.7, 137.6, 136.8, 136.7, 133.6, 133.0, 132.5, 131.8, 131.1, 130.5, 129.7, 129.6, 128.7, 128.3, 128.0, 127.2, 127.1, 126.0, 125.1, 124.2, 101.2, 91.8, 90.3, 89.7, 26.7 and 26.6 ppm. **IR** (neat): 2728, 2307, 2199, 1684, 1646, 1600, 1264, 1008 and 740 cm⁻¹. **HR ESI-MS**: [C₃₈H₂₄NaO₄]⁺ = [M+Na]⁺ requires 567.1572; found 567.1556. **TLC**: R_f = 0.4 (2:1, hexane:EtOAc).

4-[(4-Acetylphenyl)ethynyl]-3-{2-[3-(4-acetylphenyl)propioloyl]phenyl}-1-naphthaldehyde (10e**)**



¹H NMR (400 MHz, CDCl₃): δ = 8.62 (1 H, d, J = 7.5 Hz), 8.51 (1 H, d, J = 8.0 Hz), 8.14 (2 H, d, J = 7.6 Hz), 8.08 (2 H, d, J = 7.7 Hz), 7.84 (2 H, d, J = 7.8 Hz), 7.73-7.58 (4 H, m), 7.55 (2 H, d, J = 8.1 Hz), 7.50-7.41 (3 H, m), 7.39 (1 H, t, J = 7.1 Hz), 2.71 (3 H, s) and 2.68 (3 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 197.9, 197.3, 191.4, 143.9, 140.6, 140.3, 139.9, 137.0, 136.9, 136.5, 136.3, 135.1, 133.0, 132.5, 131.9, 131.6, 130.07, 130.03, 129.9, 129.1, 128.7, 128.4, 127.7, 126.9, 124.3, 123.8, 113.7, 99.3, 88.6 and 26.8 ppm. **HR ESI-MS**: [C₃₇H₂₄NaO₃]⁺ = [M+Na]⁺ requires 539.1623; found 539.1606. **TLC**: R_f = 0.6 (2:1, hexane:EtOAc). **M.P**: 267-270 °C.

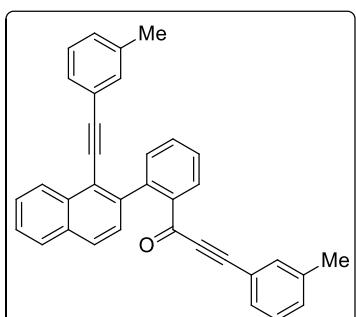
3-{2-[3-(*m*-Tolyl)propioloyl]phenyl}-4-(*m*-tolylethynyl)-1-naphthaldehyde (9f**)**



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene⁶ **8f** (100 mg, 0.31 mmol), in toluene (6 mL) and AgOTf (21 mg, 0.07 mmol) were stirred for 1.5 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **9f** (11mg, 0.04 mmol, 8%) as a yellow liquid along with eluent (9:1 hexane:EtOAc) gave **10f** (50 mg, 0.20 mmol, 67%) as a yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 10.35 (1 H, s), 9.23 (1 H, d, *J* = 6.3 Hz), 8.56 (1 H, d, *J* = 6.0 Hz), 8.24 (1 H, d, *J* = 7.1 Hz), 8.00 (1 H, s), 7.72-7.56 (4 H, m), 7.50 (1 H, d, *J* = 6.9 Hz), 7.19-7.00 (6 H, m), 6.90 (1 H, d, *J* = 6.6 Hz), 6.73 (1 H, s), 2.22 (3 H, s) and 2.16 (3 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.8, 178.6, 141.0, 140.5, 138.0, 137.9, 137.4, 133.6, 132.9, 132.6, 132.2, 131.7, 131.3, 130.48, 130.45, 129.9, 129.7, 129.6, 129.3, 128.9, 128.3, 128.2, 128.1, 127.9, 127.2, 126.8, 125.0, 122.3, 119.5, 102.8, 94.0, 88.2, 86.4, 21.2 and 21.0 ppm. **IR** (neat): 3057, 2926, 2731, 2193, 1730, 1697, 1580, 905 and 720 cm⁻¹. **HR ESI-MS**: [C₃₆H₂₄O₂]⁺ = [M+Na]⁺ requires 488.1776; found 488.1767. **TLC**: R_f = 0.2 (9:1, hexane:EtOAc).

3-(*m*-Tolyl)-1-{2-[1-(*m*-tolylethynyl)naphthalen-2-yl]phenyl}prop-2-yn-1-one (**10f**)

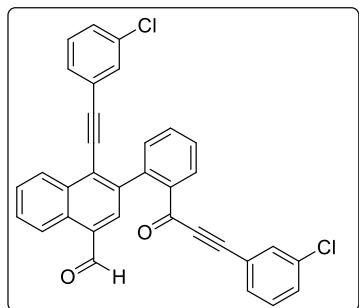


¹H NMR (400 MHz, CDCl₃): δ = 8.51 (1 H, d, *J* = 8.0 Hz), 8.18 (1 H, d, *J* = 7.5 Hz), 7.86 (2 H, t, *J* = 8.8 Hz), 7.70-7.48 (6 H, m), 7.20-7.11 (3 H, m), 7.10-7.00 (3 H, m), 6.86 (1 H, d, *J* = 6.9 Hz), 6.69 (1 H, s), 2.26 (3 H, s) and 2.17 (3 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.3, 142.1, 141.8, 138.3, 137.9, 137.8, 133.4, 133.2, 132.7, 132.2, 132.1, 132.0, 131.1, 130.0, 129.8, 129.2, 128.7, 128.3, 128.24, 128.22, 128.0, 127.8, 127.7, 127.2, 126.8, 126.6, 123.2, 120.1, 119.9, 98.9, 93.7, 88.5, 86.7, 21.3 and 21.1 ppm. **IR** (neat): 3055, 2923, 2855, 2192, 1641, 1594, 1485, 1446, 1022 and 778 cm⁻¹. **HR ESI-MS**: [C₃₅H₂₄O]⁺ = [M+H]⁺ requires 460.1827; found 460.1811. **TLC**: R_f = 0.4 (9:1, hexane:EtOAc).

4-[(3-Chlorophenyl)ethynyl]-3-{2-[3-(3-chlorophenyl)propioloyl]phenyl}-1-naphth-aldehyde (**9g**)

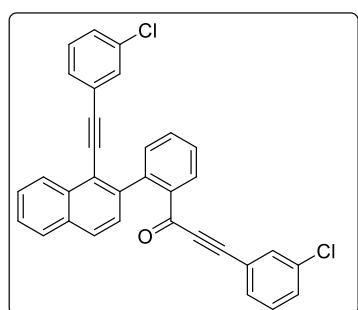
Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8g** (320 mg, 0.94 mmol), in toluene (18 mL) and AgOTf (60 mg, 0.23 mmol) were stirred for 3.5 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **9g** (130 mg, 0.49 mmol, 52%) as

yellow liquid along with eluent (4:1 hexane:EtOAc) gave **10g** (46 mg, 0.18 mmol, 19%) as a yellow liquid.



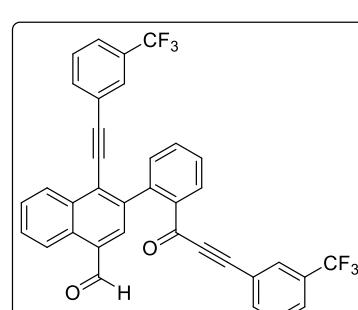
¹H NMR (400 MHz, CDCl₃): δ = 10.41 (1 H, s), 9.22 (1 H, d, *J* = 7.4 Hz), 8.51 (1 H, d, *J* = 7.2 Hz), 8.23 (1 H, d, *J* = 7.49 Hz), 8.03 (1 H, s), 7.76-7.65 (4 H, m), 7.52 (1 H, d, *J* = 7.4 Hz), 7.30-7.23 (3 H, m), 7.23-7.16 (2 H, m), 7.11 (1 H, t, *J* = 7.7 Hz) and 7.01-6.89 (2 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.7, 178.3, 141.3, 140.5, 137.8, 136.9, 134.34, 134.31, 133.6, 132.9, 132.1, 131.8, 131.5, 130.9, 130.7, 130.6, 130.5, 129.9, 129.7, 129.68, 129.64, 129.3, 128.6, 128.3, 127.6, 127.1, 126.1, 125.1, 124.2, 121.4, 100.7, 91.5, 88.8 and 87.8 ppm. **IR** (neat): 3059, 2983, 2926, 2201, 1696, 1642, 1472, 1264 and 742 cm⁻¹. **HR ESI-MS**: [C₃₄H₁₉Cl₂O₂]⁺ = [M+H]⁺ requires 529.0762; found 529.0749. **TLC**: R_f = 0.4 (4:1, hexane:EtOAc).

3-(3-Chlorophenyl)-1-{2-[1-[(3-chlorophenyl)ethynyl]naphthalen-2-yl}phenyl}prop-2-yn-1-one (**10g**)



¹H NMR (400 MHz, CDCl₃): δ = 8.58 (1 H, d, *J* = 7.7 Hz), 8.47 (1 H, d, *J* = 8.2 Hz), 7.76-7.70 (1 H, m), 7.70-7.56 (6 H, m), 7.53-7.44 (4 H, m), 7.44-7.33 (4 H, m) and 7.27-7.23 (1 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 191.3, 143.8, 139.7, 137.2, 136.5, 136.3, 135.1, 134.7, 134.2, 133.1, 131.6, 130.1, 129.96, 129.93, 129.8, 129.7, 129.58, 129.53, 129.1, 128.56, 128.50, 127.9, 127.7, 126.9, 124.6, 124.2, 123.8, 113.7, 98.7 and 86.6 ppm. **IR** (neat): 3050, 2010, 1654, 1567, 1212 and 750 cm⁻¹. **TLC**: R_f = 0.6 (4:1, hexane:EtOAc).

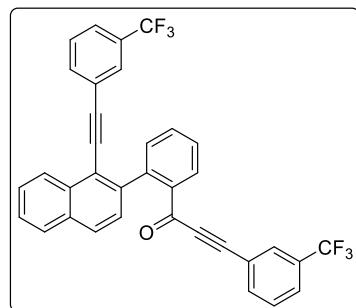
4-{{[3-(Trifluoromethyl)phenyl]ethynyl}-3-{2-[3-(trifluoromethyl)phenyl]propioloyl}-phenyl}-1-naphthaldehyde (**9h**)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8h** (160 mg, 0.43 mmol), in toluene (8 mL) and AgOTf (28 mg, 0.10 mmol) were stirred for 12 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **9h** (81 mg, 0.27 mmol, 63%) as yellow liquid along with eluent (4:1 hexane:EtOAc) gave **10h** (35 mg, 0.12 mmol, 28%) as a yellow liquid.

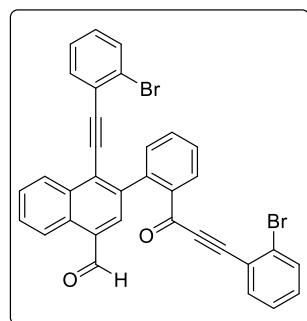
¹H NMR (400 MHz, CDCl₃): δ = 10.4 (1 H, s), 9.32-9.16 (1 H, m), 8.58-8.47 (1 H, m), 8.25 (1 H, d, *J* = 7.6 Hz), 8.0 (1 H, s), 7.75-7.66 (4 H, m), 7.59-7.47 (6 H, m), 7.40 (1 H, d, *J* = 7.5 Hz), 7.30 (1 H, t, *J* = 7.8 Hz) and 7.21 (1 H, d, *J* = 7.6 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.6, 178.3, 141.5, 140.5, 137.7, 136.8, 136.7, 135.5, 134.7, 133.6, 133.5, 133.1, 131.9, 131.1, 130.5, 129.7, 129.08, 129.01 (C, q, *J* = 3.6 Hz), 128.7, 128.6 (C, q, *J* = 4.2 Hz), 128.4, 127.08, 127.02 (C, q, *J* = 3.9 Hz), 126.7, 125.9, 125.6 (C, q, *J* = 3.4 Hz), 125.4, 125.1, 123.5, 120.7, 100.5, 91.1, 89.0 and 88.2 ppm. **¹⁹F NMR** (282 MHz, CDCl₃): δ = -63.00 (s, 3 F, CF₃) and -63.05 (s, 3 F, CF₃) ppm. **IR** (neat): 3062, 2926, 2856, 2202, 1692, 1644, 1594, 1333 and 903 cm⁻¹. **HR ESI-MS**: [C₃₆H₁₉F₆O₂]⁺ = [M+H]⁺ requires 597.1289; found 597.1275. **TLC**: R_f = 0.5 (4:1, hexane:EtOAc).

3-[3-(Trifluoromethyl)phenyl]-1-{2-{1-[[3-(trifluoromethyl)phenyl]ethynyl}naphthalen-2-yl}phenyl}prop-2-yn-1-one (10h)



¹H NMR (400 MHz, CDCl₃): δ = 8.61 (1 H, d, *J* = 7.6 Hz), 8.52 (1 H, d, *J* = 8.3 Hz), 8.01 (1 H, s), 7.94 (1 H, d, *J* = 7.6 Hz), 7.80 (1 H, d, *J* = 7.7 Hz), 7.75-7.63 (8 H, m), 7.56 (2 H, t, *J* = 8.3 Hz), 7.48 (1 H, t, *J* = 7.6 Hz), 7.40 (1 H, t, *J* = 7.5 Hz) and 7.26 (1 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 191.3, 143.8, 139.9, 139.7, 136.6, 136.3, 136.1, 135.2, 134.9, 133.2, 133.1, 131.4, 130.0, 129.9, 129.5, 128.9, 128.8, 128.5 (q, C, *J* = 3.3 Hz), 127.8, 126.9, 126.63 (q, C, *J* = 3.2 Hz), 125.8 (q, C, *J* = 3.2 Hz), 125.2 (q, C, *J* = 3.2 Hz), 124.3, 123.8, 113.7, 98.5, 86.9 and 76.7 ppm. **¹⁹F NMR** (282 MHz, CDCl₃): δ = -62.3 (s, 3 F, CF₃) and -62.8 (s, 3 F, CF₃) ppm. **IR** (neat): 3057, 2926, 2856, 1727, 1331, 1265, 1131 and 741 cm⁻¹. **HR ESI-MS**: [C₃₅H₁₉F₆O]⁺ = [M+H]⁺ requires 569.1340; found 569.1320. **TLC**: R_f = 0.6 (4:1, hexane:EtOAc). **M.P.**: 187-190 °C.

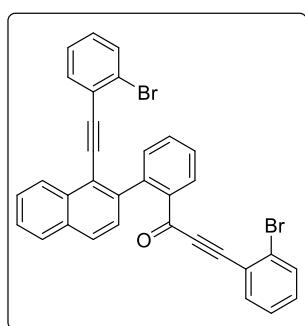
4-[(2-Bromophenyl)ethynyl]-3-{2-[3-(2-bromophenyl)propioloyl]phenyl}-1-naphthaldehyde (9i)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8i** (280 mg, 0.73 mmol), in dry toluene (14 mL) and AgOTf (47 mg, 0.18 mmol) were stirred for 12 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **9i** (100 mg, 0.32 mmol, 45%) as yellow liquid along with eluent (4:1 hexane:EtOAc) gave **10i** (56 mg, 0.19 mmol, 26%) as a yellow liquid.

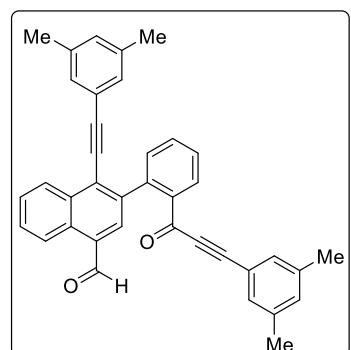
¹H NMR (400 MHz, CDCl₃): δ = 10.3 (1 H, s), 9.28-9.16 (1 H, m), 8.81-8.64 (1 H, m), 8.46 (1 H, d, *J* = 7.5 Hz), 7.99 (1 H, s), 7.73-7.63 (4 H, m), 7.53-7.45 (3 H, m), 7.24 (1 H, d, *J* = 7.7 Hz), 7.19-7.10 (4 H, m) and 7.05 (1 H, t, *J* = 7.3 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.9, 178.2, 141.5, 140.6, 137.2, 137.1, 134.7, 133.9, 133.8, 133.0, 132.5, 132.4, 131.9, 131.7, 131.5, 130.7, 130.0, 129.6, 129.3, 128.5, 128.1, 127.4, 127.1, 127.0, 126.2, 125.8, 125.3, 125.0, 124.9, 122.4, 100.3, 91.5, 90.99 and 90.95 ppm. **IR** (neat): 3059, 2200, 1693, 1642, 1587, 1262 and 733 cm⁻¹. **HR ESI-MS**: [C₃₄H₁₉Br₂O₂]⁺ = [M+H]⁺ requires 616.9752; found 616.9729. **TLC**: R_f = 0.3 (4:1, hexane:EtOAc).

3-(2-Bromophenyl)-1-{2-[1-[(2-bromophenyl)ethynyl]naphthalen-2-yl]phenyl}prop-2-yn-1-one (10i)



¹H NMR (400 MHz, CDCl₃): δ = 8.81 (1 H, d, *J* = 7.6 Hz), 8.70 (1 H, d, *J* = 8.2 Hz), 7.79 (2 H, d, *J* = 7.7 Hz), 7.74 (1 H, d, *J* = 8.0 Hz), 7.71-7.56 (4 H, m), 7.51-7.37 (7 H, m) and 7.33-7.27 (2 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 191.3, 144.1, 139.7, 139.6, 136.8, 136.3, 135.0, 133.8, 133.0, 132.8, 132.7, 130.8, 130.3, 129.9, 129.87, 129.85, 129.0, 128.7, 127.7, 127.5, 127.46, 127.41, 125.5, 125.3, 124.2, 124.1, 123.5, 114.0, 98.6 and 89.9 ppm. **IR** (neat): 3058, 2926, 2861, 2199, 1724, 1646, 1473 and 722 cm⁻¹. **HR ESI-MS**: [C₃₃H₁₉Br₂O]⁺ = [M+H]⁺ requires 588.9803; found 588.9779. **TLC**: R_f = 0.6 (4:1, hexane:EtOAc).

4-[(3,5-Dimethylphenyl)ethynyl]-3-{2-[3-(3,5-dimethylphenyl)propioloyl]phenyl}-1-naphthaldehyde (9j)

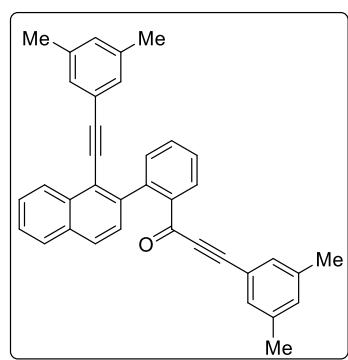


Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8j** (328 mg, 0.97 mmol), in toluene (18 mL) and AgOTf (62 mg, 0.24 mmol) were stirred for 13 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **9j** (150 mg, 0.58 mmol, 60%) as yellow liquid along with eluent (4:1 hexane:EtOAc) gave **10j** (35 mg, 0.14 mmol, 15%) as a yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 10.34 (1 H, s), 9.24 (1 H, d, *J* = 7.4 Hz), 8.56 (1 H, d, *J* = 7.5 Hz), 8.23 (1 H, d, *J* = 7.5 Hz), 7.99 (1 H, s), 7.70-7.56 (4 H, m), 7.49 (1 H, d, *J* = 7.3 Hz), 6.94 (2 H, s), 6.87 (2 H, s), 6.60 (2 H, s), 2.1 (6 H, s) and 2.1 (6 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.8, 178.6, 140.9, 140.6, 138.0, 137.8, 137.5, 133.6, 132.5, 132.3, 131.7,

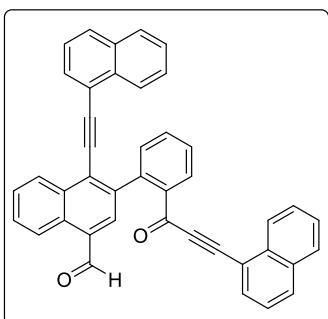
130.9, 130.3, 130.2, 129.6, 129.4, 129.2, 128.2, 127.8, 127.2, 126.9, 125.0, 122.2, 119.3, 103.1, 94.4, 88.0, 86.1, 21.0 and 20.9 ppm. **IR** (neat): 2955, 2924, 2726, 2192, 1736, 1690, 1636, 1240, 1060 and 751 cm⁻¹. **HR ESI-MS**: [C₃₈H₂₉O₂]⁺ = [M+H]⁺ requires 517.2168; found 517.2157. **TLC**: R_f = 0.4 (4:1, hexane:EtOAc).

3-(3,5-Dimethylphenyl)-1-{2-[1-[(3,5-dimethylphenyl)ethynyl]naphthalen-2-yl}phenyl}-prop-2-yn-1-one (10j)



¹H NMR (400 MHz, CDCl₃): δ = 8.53 (1 H, d, *J* = 8.2 Hz), 8.19 (1 H, d, *J* = 7.5 Hz), 7.86 (2 H, t, *J* = 5.9 Hz), 7.69-7.50 (6 H, m), 6.99 (2 H, s), 6.89 (2 H, s), 6.59 (2 H, s), 2.24 (6 H, s) and 2.15 (6 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 179.3, 142.1, 141.8, 138.3, 137.79, 137.73, 133.4, 132.7, 132.16, 132.12, 132.0, 130.4, 130.2, 130.0, 129.2, 128.2, 127.7, 127.1, 126.9, 126.5, 123.1, 120.1, 119.7, 99.1, 94.1, 88.2, 86.4, 21.1 and 20.9 ppm. **IR** (neat): 3051, 2926, 2191, 1637, 1599, 1263 and 742 cm⁻¹. **HR ESI-MS**: [C₃₇H₂₉O]⁺ = [M+H]⁺ requires 489.2218; found 489.2229. **TLC**: R_f = 0.6 (4:1, hexane:EtOAc).

3-{2-[3-(Naphthalen-1-yl)propioloyl]phenyl}-4-(naphthalen-1-ylethyynyl)-1-naphthaldehyde (9k)

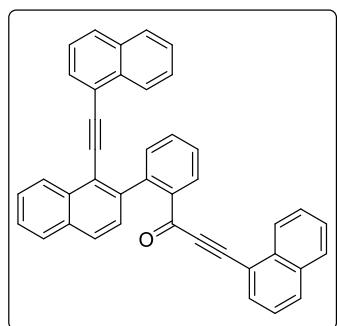


Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8k** (333 mg, 0.94 mmol), in dry toluene (18 mL) and AgOTf (60 mg, 0.23 mmol) were stirred for 10 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **9k** (130 mg, 0.46 mmol, 50%) as yellow liquid along with eluent (4:1 hexane:EtOAc) gave **10k** (77 mg, 0.28 mmol, 30%) as a yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 10.32 (1 H, s), 9.24-9.11 (1 H, m), 8.67-8.59 (1 H, m), 8.36 (1 H, d, *J* = 7.5 Hz), 8.02 (1 H, s), 7.87 (1 H, d, *J* = 8.3 Hz), 7.80-7.69 (5 H, m), 7.65-7.59 (5 H, m), 7.54 (1 H, d, *J* = 8.1 Hz), 7.41-7.37 (2 H, m), 7.34-7.29 (2 H, m), 7.23-7.18 (1 H, m), 7.15 (1 H, t, *J* = 7.8 Hz) and 7.09 (1 H, d, *J* = 7.0 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.8, 178.6, 141.1, 140.9, 137.8, 137.3, 133.7, 133.1, 133.0, 132.9, 132.7, 132.5, 131.8, 131.1, 130.4, 129.7, 129.5, 129.2, 128.5, 128.3, 128.2, 128.0, 127.4, 127.1, 126.9, 126.7, 126.5, 126.4, 126.0, 125.6, 125.1, 125.0, 124.8, 120.3, 117.2, 100.9, 93.0, 92.2 and 91.5 ppm. **IR** (neat):

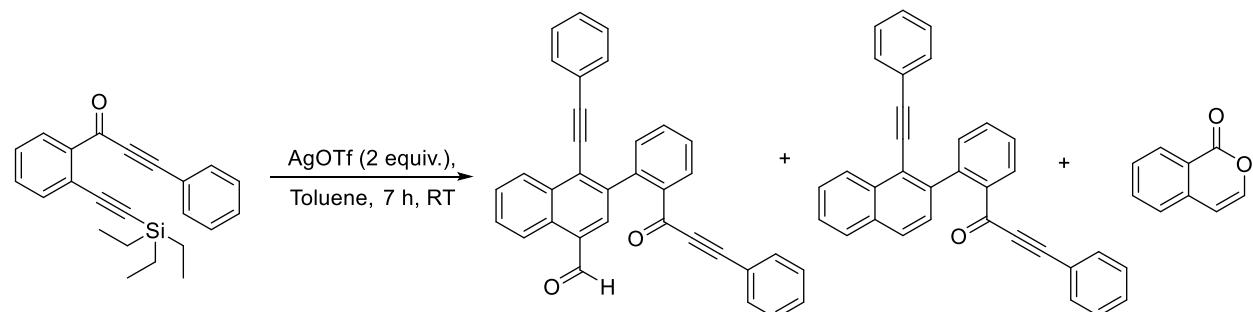
3057, 2923, 2859, 2188, 1688, 1637, 1586, 1292 and 743 cm⁻¹. **HR ESI-MS:** [C₄₂H₂₅O₂]⁺ = [M+H]⁺ requires 561.1855; found 561.1800. **TLC:** R_f = 0.4 (4:1, hexane:EtOAc).

3-(Naphthalen-1-yl)-1-{2-[1-(naphthalen-1-ylethynyl)naphthalen-2-yl]phenyl}prop-2-yn-1-one (10k)



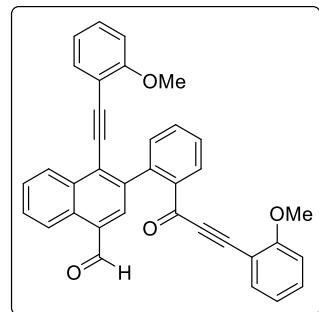
¹H NMR (400 MHz, CDCl₃): δ = 8.78 (1 H, d, *J* = 7.6 Hz), 8.69 (1 H, d, *J* = 8.5 Hz), 8.66 (1 H, d, *J* = 8.4 Hz), 8.04-7.94 (4 H, m), 7.71 (1 H, t, *J* = 7.7 Hz), 7.68-7.62 (4 H, m), 7.62-7.55 (4 H, m), 7.53 (1 H, t, *J* = 7.7 Hz), 7.47-7.43 (2 H, m) and 7.36-7.21 (5 H, m) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 191.3, 144.1, 139.7, 136.7, 136.4, 134.9, 134.1, 133.6, 133.5, 133.4, 132.5, 131.1, 129.8, 129.77, 129.71, 129.6, 128.7, 128.6, 128.5, 127.5, 127.4, 127.1, 127.0, 126.8, 126.2, 126.0, 125.6, 125.5, 125.4, 124.1, 123.9, 120.6, 114.3, 107.1, 98.5, 91.9 and 90.2 ppm. **IR** (neat): 3056, 2929, 2861, 2187, 1729, 1638, cm⁻¹. **HR ESI-MS:** [C₄₁H₂₅O]⁺ = [M+H]⁺ requires 533.1905; found 533.1883. **TLC:** R_f = 0.5 (4:1, hexane:EtOAc).

1*H*-isochromen-1-one (4a):



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8l** (60 mg, 0.174 mmol), in dry toluene (4 mL) and AgOTf (89 mg, 0.34 mmol) were stirred for 7 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) gave **9a** (20 mg, 0.08 mmol, 50%) as yellow liquid along with eluent (4:1 hexane:EtOAc) gave **10a** (9 mg, 0.03 mmol, 23%) as a yellow liquid, and isocoumarin **4a** (3 mg, 0.02 mmol, 14%).

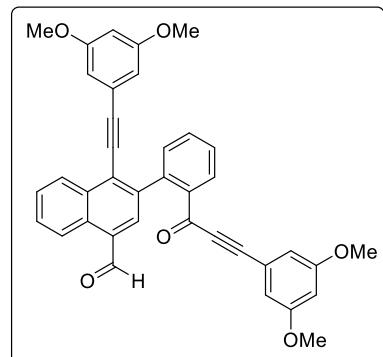
4-[(2-Methoxyphenyl)ethynyl]-3-{2-[3-(2-methoxyphenyl)propioloyl]phenyl}-1-naphthaldehyde (9m)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene⁴ **8m** (180 mg, 0.54 mmol), in toluene (10 mL) and AgOTf (35 mg, 0.13 mmol) were stirred for 5 h at RT. Purification by flash column chromatography (3:1 hexane:EtOAc) gave **9m** (45 mg, 0.17 mmol, 39%) as yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 10.33 (1 H, s), 9.32-9.09 (1 H, m), 8.86-8.67 (1 H, m), 8.41 (1 H, d, *J* = 7.6 Hz), 7.96 (1 H, s), 7.72-7.58 (4 H, m), 7.53 (1 H, d, *J* = 7.4 Hz), 7.31-7.24 (1 H, m), 7.23-7.17 (2 H, m), 7.01 (1 H, d, *J* = 7.5 Hz), 6.82-6.70 (4 H, m), 3.82 (3 H, s) and 3.77 (3 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 193.0, 178.6, 161.3, 160.4, 140.7, 137.8, 137.6, 134.5, 133.8, 133.5, 132.4, 132.2, 132.0, 131.4, 130.5, 130.1, 129.1, 128.2, 127.7, 127.6, 126.9, 124.9, 120.4, 112.1, 110.6, 109.2, 99.2, 92.6, 90.9, 90.7 and 55.2 ppm. **IR (neat):** 3070, 2927, 2846, 2194, 1685, 1639, 1492, 1276 and 740 cm⁻¹. **TLC:** R_f = 0.2 (3:1, hexane:EtOAc).

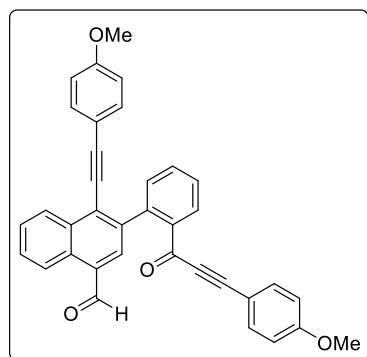
4-[(3,5-Dimethoxyphenyl)ethynyl]-3-{2-[3-(3,5-dimethoxyphenyl)propioloyl]phenyl}-1-naphthaldehyde (9n)



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **8n** (137 mg, 0.37 mmol), in toluene (7 mL) and AgOTf (24 mg, 0.09 mmol) were stirred for 8 h at RT. Purification by flash column chromatography (3:1 hexane:EtOAc) gave **9n** (48 mg, 0.16 mmol, 45%) as yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 10.31 (1 H, s), 9.14 (1 H, d, *J* = 8.1 Hz), 8.45 (1 H, d, *J* = 8.0 Hz), 8.16 (1 H, d, *J* = 7.7 Hz), 7.94 (1 H, s), 7.65-7.57 (3 H, m), 7.53 (1 H, t, *J* = 7.5 Hz), 7.43 (1 H, d, *J* = 7.4 Hz), 6.38 (2 H, s), 6.34-6.27 (2 H, m), 6.17 (2 H, s), 3.65 (6 H, s), 3.58 (6 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.8, 178.6, 160.5, 160.4, 141.3, 140.6, 138.0, 137.1, 133.6, 132.7, 131.9, 130.6, 130.5, 129.7, 129.4, 128.4, 128.0, 127.2, 126.5, 125.1, 123.8, 121.0, 110.1, 109.6, 104.1, 102.5, 102.4, 93.7, 87.8, 86.3, 55.5 and 55.4 ppm. **IR (neat):** 2985, 2356, 1735, 1670, 1373, 1234 and 1045 cm⁻¹. **TLC:** R_f = 0.4 (3:1, hexane:EtOAc).

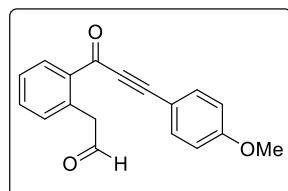
4-[(4-Methoxyphenyl)ethynyl]-3-{2-[3-(4-methoxyphenyl)propioloyl]phenyl}-1-naphthaldehyde (9o**)**



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene⁵ **8o** (100 mg, 0.30 mmol), in toluene (6 mL) and AgOTf (39 mg, 0.15 mmol) were stirred for 24 h at RT. Purification by flash column chromatography (5:1 hexane:EtOAc) gave **9o** (9 mg, 0.03 mmol, 10%) as a yellow liquid and **11** (49 mg, 0.17 mmol, 60%) as pale yellow liquid.

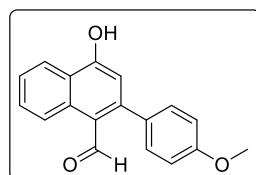
¹H NMR (400 MHz, CDCl₃): δ = 10.3 (1 H, s), 9.17 (1 H, d, *J* = 7.3 Hz), 8.49 (1 H, d, *J* = 6.8 Hz), 8.16 (1 H, d, *J* = 7.4 Hz), 7.9 (1 H, s), 7.67-7.57 (3 H, m), 7.57-7.51 (1 H, m), 7.45 (1 H, d, *J* = 7.1 Hz), 7.19 (2 H, d, *J* = 8.8 Hz), 6.94 (2 H, d, *J* = 8.4 Hz), 6.69 (2 H, d, *J* = 8.4 Hz), 6.59 (2 H, d, *J* = 8.4 Hz) and 3.69 (6 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 192.9, 178.8, 161.4, 160.3, 140.8, 140.7, 139.4, 138.2, 137.6, 134.8, 133.6, 133.4, 132.4, 131.9, 130.4, 130.2, 129.8, 129.34, 128.37, 127.9, 127.4, 127.2, 125.1, 114.8, 114.1, 111.7, 103.1, 94.8, 88.7, 85.9, 55.48 and 55.40 ppm. **IR (neat)**: 3063, 2978, 2883, 2243, 1765, 1735, 1692, 1289 and 710 cm⁻¹. **HR ESI-MS**: [C₃₆H₂₅O₄]⁺ = [M+H]⁺ requires 521.1753; found 521.1740. **TLC**: R_f = 0.3 (5:1, hexane:EtOAc).

2-{2-[3-(4-Methoxyphenyl)propioloyl]phenyl}acetaldehyde (11**):**



¹H NMR (400 MHz, CDCl₃): δ = 9.81 (1 H, s), 8.42 (1 H, d, *J* = 7.5 Hz), 7.61 (2 H, d, *J* = 8.2 Hz), 7.57-7.49 (2 H, m), 7.27-7.24 (1 H, m), 6.92 (2 H, d, *J* = 8.1 Hz), 4.10 (2 H, s) and 3.84 (3 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 198.8, 179.4, 161.9, 135.6, 135.2, 134.6, 133.9, 133.5, 133.1, 127.8, 114.5, 111.8, 94.4, 87.9, 55.5 and 49.3 ppm. **IR (neat)**: 2981, 2190, 1735, 1600, 1257 and 755 cm⁻¹. **TLC**: R_f = 0.4 (5:1, hexane:EtOAc).

4-Hydroxy-2-(4-methoxyphenyl)-1-naphthaldehyde (14**)**

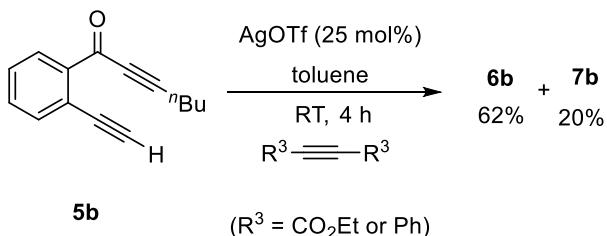


Followed general procedure F: The 1,5-diketone **11** (15 mg, 0.05 mmol), in toluene (1 mL) and AgOTf (4 mg, 0.01 mmol) were stirred for 1 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **14** (13 mg, 0.04 mmol, 87%) as yellow semi solid.

¹H NMR (400 MHz, CDCl₃): δ = 13.9 (1 H, s), 8.51 (1 H, d, *J* = 8.2 Hz), 7.76 (3 H, t, *J* = 7.7 Hz), 7.68-7.59 (2 H, m), 7.55 (1 H, t, *J* = 7.2 Hz), 7.27-7.21 (1 H, m), 7.02 (2 H, d, *J* = 8.1 Hz)

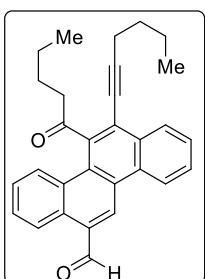
and 3.90 (3 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 200.1, 163.6, 162.8, 137.2, 131.8, 130.8, 130.2, 127.5, 126.0, 125.5, 124.5, 117.8, 113.8, 112.8 and 55.6 ppm. **IR** (neat): 3050, 2923, 2850, 1596, 1465, 1261, 1172 and 736 cm⁻¹. **TLC**: R_f = 0.2 (4:1, hexane:EtOAc).

failure of the cross [4+2] cycloaddition of pyrylium ion with alkyne:



Followed general procedure F: The 2-(alkynonyl)alkynylbenzene **5b** (50 mg, 0.23 mmol), and diphenylacetylene or diethylacetylene dicarboxylate (external alkyne-1.2 equiv.) (in case of diethyl but-2-yndioate, 46 mg, 0.27 mmol and in case of diphenyl acetylene 49 mg, 0.27 mmol) in toluene (5 mL) and AgOTf (14 mg, 0.05 mmol) were stirred for 4 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave **6b** (30 mg, 0.14 mmol, 62%) as a yellow liquid and **7b** (9 mg, 0.04 mmol, 20%) as pale yellow liquid.

12-(Hex-1-yn-1-yl)-11-pentanoylchrysene-6-carbaldehyde (22)

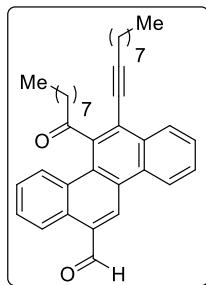


The carbonylated naphthalene **6b** (30 mg, 0.07 mmol), in 1, 2-DCB (3 mL) were stirred for 21 h at 150 °C. Purification by flash column chromatography (4:1 hexane:EtOAc) gave the chrysene derivative **22** (19 mg, 0.04 mmol, 65%) as a red solid.

Chemical Structure: A chemical structure of 2-hydroxypropanal is shown, consisting of a carbonyl group (C=O) attached to a methyl group (CH₃) which is further attached to a formyl group (-CHO).

1H NMR (400 MHz, CDCl₃): δ = 10.5 (1 H, s), 9.39 (1 H, d, *J* = 7.5 Hz), 9.1 (1 H, s), 8.77 (1 H, d, *J* = 7.4 Hz), 8.58 (1 H, d, *J* = 7.4 Hz), 8.43 (1 H, d, *J* = 7.4 Hz), 7.86-7.70 (3 H, m), 7.65 (1 H, t, *J* = 6.5 Hz), 2.76-2.57 (4 H, m), 1.80-1.69 (4 H, m), 1.63-1.56 (2 H, m), 1.38-1.31 (2 H, m), 1.02 (3 H, t, *J* = 6.1 Hz) and 0.86 (3 H, t, *J* = 6.5 Hz) ppm. **13C NMR** (100 MHz, CDCl₃): δ = 209.6, 193.2, 141.6, 133.7, 131.4, 130.7, 130.16, 131.10, 129.5, 128.8, 128.6, 128.5, 128.1, 128.0, 127.6, 127.2, 126.6, 125.6, 122.9, 122.1, 104.2, 77.0, 44.3, 30.8, 26.1, 22.29, 22.23, 19.9, 14.0 and 13.8 ppm. **IR** (neat): 3060, 2929, 2864, 2216, 1696, 1577, 1263 and 742 cm⁻¹. **HR ESI-MS:** [C₃₀H₂₈O₂]⁺ = [M+H]⁺ requires 420.2089; found 420.2071. **TLC:** R_f = 0.5 (4:1, hexane:EtOAc). **M.P:** 93-95 °C.

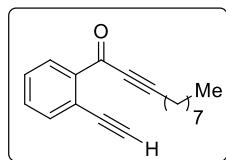
12-(Dec-1-yn-1-yl)-11-nonanoylchrysene-6-carbaldehyde (23)



The carbonylated naphthalene **6c** (30 mg, 0.05 mmol), in 1,2-DCB (3 mL) were stirred for 28 h at 150 °C. Purification by flash column chromatography (4:1 hexane:EtOAc) gave the chrysene derivative **23** (12 mg, 0.02 mmol, 45%) as a red solid.

1H NMR (400 MHz, CDCl₃): δ = 10.5 (1 H, s), 9.41 (1 H, d, *J* = 8.1 Hz), 9.18 (1 H, s), 8.81 (1 H, d, *J* = 8.3 Hz), 8.61 (1 H, d, *J* = 8.0 Hz), 8.45 (1 H, d, *J* = 8.5 Hz), 7.83 (1 H, t, *J* = 7.7 Hz), 7.77 (2 H, t, *J* = 8.0 Hz), 7.66 (1 H, t, *J* = 7.4 Hz), 2.69 (2 H, t, *J* = 7.3 Hz), 2.63 (2 H, t, *J* = 7.0 Hz), 1.80-1.71 (4 H, m), 1.62-1.53 (5 H, m), 1.33-1.28 (9 H, m) and 0.91-0.84 (12 H, m) ppm. **13C NMR** (100 MHz, CDCl₃): δ = 209.6, 193.3, 141.6, 133.8, 131.4, 130.7, 130.18, 130.13, 129.5, 128.8, 128.6, 128.5, 128.1, 128.0, 127.6, 125.6, 123.0, 122.1, 114.2, 104.3, 44.6, 32.0, 31.9, 29.54, 29.50, 29.41, 29.3, 29.29, 29.26, 29.1, 28.7, 24.1, 22.8, 20.2 and 14.2 ppm. **IR** (neat): 3061, 2926, 2858, 2219, 1740, 1698, 1462, 1265 and 756 cm⁻¹. **HR ESI-MS**: [C₃₈H₄₆O₂]⁺ = [M+H]⁺ requires 533.3420; found 533.3395. **TLC**: R_f = 0.5 (4:1, hexane:EtOAc).

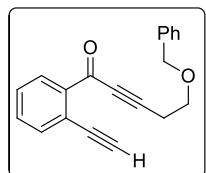
1-(2-Ethynylphenyl)undec-2-yn-1-one (**5c**)



Followed general procedure D: Alcohol **S1** (400 mg, 1.49 mmol), Jones reagent (3 M solution in water and H₂SO₄) (0.6 mL, 1.79 mmol), anhydrous acetone (4 mL) was stirred for 2 h at 0 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the 2-(alkynonyl)alkynylbenzene **5c** (350 mg, 1.3 mmol, 88%) as a red oil.

1H NMR (400 MHz, CDCl₃): δ = 8.10 (1 H, d, *J* = 7.8 Hz), 7.61 (1 H, d, *J* = 7.5 Hz), 7.53-7.40 (2 H, m), 3.39 (1 H, s), 2.45 (2 H, t, *J* = 7.2 Hz), 1.67-1.60 (2 H, m), 1.50-1.37 (2 H, m), 1.31-1.24 (8 H, m) and 0.87 (3 H, t, *J* = 7.0 Hz) ppm. **13C NMR** (100 MHz, CDCl₃): δ = 177.4, 139.1, 135.5, 132.3, 131.8, 128.5, 121.6, 97.4, 82.8, 82.0, 80.7, 31.9, 29.2, 29.1, 29.0, 27.7, 22.7, 19.4 and 14.2 ppm. **IR** (neat): 3298, 2934, 2858, 2212, 1647, 1464, 1257 and 737 cm⁻¹. **HR ESI-MS**: [C₁₉H₂₃O]⁺ = [M+H]⁺ requires 267.1749; found 267.1735. **TLC**: R_f = 0.6 (19:1, hexane:EtOAc).

5-(Benzylxylo)-1-(2-ethynylphenyl)pent-2-yn-1-one (**5e**)

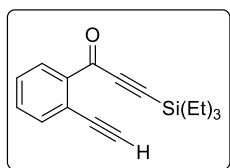


Followed general procedure D: Alcohol **S2** (320 mg, 1.1 mmol), Jones reagent (3 M solution in water and H₂SO₄) (0.4 mL, 1.32 mmol), anhydrous acetone (4 mL) was stirred for 2 h at 0 °C. Purification by flash column

chromatography (19:1 hexane:EtOAc) gave the 2-(alkynonyl)alkynyl-benzene **5e** (181 mg, 1.11 mmol, 57%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.12 (1 H, t, *J* = 8.2 Hz), 7.61 (1 H, d, *J* = 7.4 Hz), 7.51-7.47 (1 H, m), 7.41-7.26 (6 H, m), 4.58 (2 H, s), 3.71 (2 H, t, *J* = 6.5 Hz), 3.38 (1 H, s) and 2.78 (2 H, t, *J* = 6.5 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.7, 138.7, 137.8, 135.5, 132.4, 132.1, 130.2, 128.6, 127.9, 127.8, 121.6, 93.7, 83.0, 82.0, 81.1, 73.2, 67.3 and 20.9 ppm. **IR** (neat): 3300, 2930, 2858, 2207, 1648, 1261 and 880 cm⁻¹. **HR ESI-MS**: [C₂₀H₁₇O₂]⁺ = [M+H]⁺ requires 289.1307; found 289.1306. **TLC**: R_f = 0.5 (19:1, hexane:EtOAc).

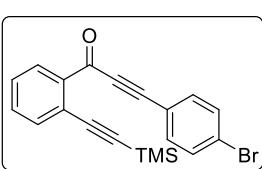
1-(2-Ethynylphenyl)-3-(triethylsilyl)prop-2-yn-1-one (**5f**)



Followed general procedure D: Alcohol **S3** (200 mg, 0.7 mmol), Jones reagent (3 M solution in water and H₂SO₄) (0.3 mL, 0.88 mmol), anhydrous acetone (4 mL) was stirred for 2 h at 0 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the 2-(alkynonyl)-alkynylbenzene **5f** (180 mg, 0.6 mmol, 91%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.15 (1 H, d, *J* = 7.5 Hz), 7.61 (1 H, d, *J* = 7.4 Hz), 7.50 (1 H, t, *J* = 7.5 Hz), 7.45 (1 H, t, *J* = 7.5 Hz), 3.38 (1 H, s), 1.04 (9 H, t, *J* = 7.9 Hz) and 0.72 (6 H, q, *J* = 7.9 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 176.7, 138.5, 135.5, 132.6, 132.1, 128.5, 121.7, 102.7, 99.1, 83.0, 81.9, 7.48 and 3.98 ppm. **IR** (neat): 3292, 2953, 2885, 2149, 1651, 1467, 1234, 1012 and 743 cm⁻¹. **HR ESI-MS**: [C₁₇H₂₁OSi]⁺ = [M+H]⁺ requires 269.1362; found 269.1354. **TLC**: R_f = 0.6 (19:1, hexane:EtOAc).

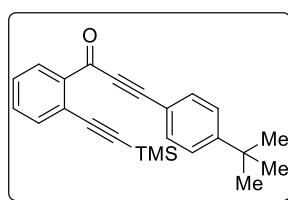
3-(4-Bromophenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (**8c**)



Followed general procedure C: Alcohol **S4** (415 mg, 1.08 mmol), IBX (393 mg, 1.40 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (9:1 hexane:EtOAc) gave the ketone **8c** (350 mg, 0.91 mmol, 84%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.06 (1 H, d, *J* = 7.7 Hz), 7.62 (1 H, d, *J* = 7.5 Hz), 7.57-7.48 (5 H, m), 7.47-7.37 (1 H, m) and 0.21 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.5, 138.9, 135.2, 134.5, 132.5, 132.1, 131.2, 128.3, 125.7, 122.8, 119.3, 102.9, 101.7, 92.0, 88.9 and 0.00 ppm. **IR** (neat): 2959, 2926, 2199, 1647, 1586, 1481, 1275 and 846 cm⁻¹. **HR ESI-MS**: [C₂₀H₁₈BrOSi]⁺ = [M+H]⁺ requires 381.0310; found 381.0305. **TLC**: R_f = 0.2 (9:1, hexane:EtOAc). **M. P.:** 73-75 °C.

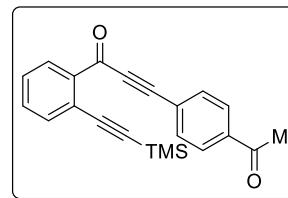
3-[4-(*tert*-Butyl)phenyl]-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (8d**)**



Followed general procedure C: Alcohol **S5** (85 mg, 0.22 mmol), IBX (185 mg, 0.66 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the ketone **8d** (71 mg, 0.18 mmol, 82%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.11 (1 H, d, *J* = 7.7 Hz), 7.60 (3 H, t, *J* = 8.8 Hz), 7.52-7.39 (4 H, m), 1.32 (9 H, s) and 0.21 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.7, 154.6, 139.2, 135.1, 133.2, 132.2, 131.3, 128.3, 125.8, 122.7, 117.3, 103.1, 101.4, 94.1, 88.0, 35.2, 31.1 and -0.11 ppm. **IR** (neat): 3060, 2963, 2197, 2155, 1646, 1305 and 745 cm⁻¹. **HR ESI-MS**: [C₂₄H₂₇OSi]⁺ = [M+H]⁺ requires 359.1831; found 359.1832. **TLC**: R_f = 0.5 (19:1, hexane:EtOAc).

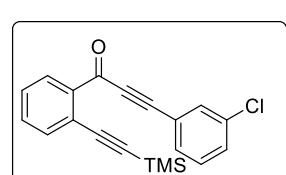
3-(4-Acetylphenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (8e**)**



Followed general procedure C: Alcohol **S6** (300 mg, 0.86 mmol), IBX (315 mg, 1.12 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (9:1 hexane:EtOAc) gave the ketone **8e** (240 mg, 0.70 mmol, 81%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.09 (1 H, d, *J* = 7.6 Hz), 7.98 (2 H, d, *J* = 7.9 Hz), 7.73 (2 H, d, *J* = 7.9), 7.63 (1 H, d, *J* = 7.5 Hz), 7.52 (1 H, t, *J* = 7.5 Hz), 7.45 (1 H, t, *J* = 7.5 Hz), 2.63 (3 H, s) and 0.21 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 197.2, 177.3, 138.7, 138.1, 135.2, 133.3, 132.6, 131.3, 128.48, 128.42, 125.0, 122.9, 102.8, 101.9, 91.4, 89.9, 26.8 and 0.10 ppm. **IR** (neat): 2961, 2200, 2156, 1687, 1648, 1601, 1298, 1259 and 859 cm⁻¹. **HR ESI-MS**: [C₂₂H₂₁O₂Si]⁺ = [M+H]⁺ requires 345.1311; found 345.1306. **TLC**: R_f = 0.4 (9:1, hexane:EtOAc). **M.P.**: 65-68 °C.

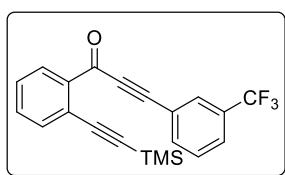
3-(3-Chlorophenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (8g**)**



Followed general procedure C: Alcohol **S7** (370 mg, 1.0 mmol), IBX (396 mg, 1.41 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the ketone **8g** (340 mg, 0.91 mmol, 93%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.07 (1 H, d, *J* = 7.6 Hz), 7.67-7.57 (2 H, m), 7.56-7.48 (2 H, m), 7.45 (2 H, t, *J* = 7.1 Hz), 7.34 (1 H, t, *J* = 7.8 Hz) and 0.021 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.3, 138.8, 135.2, 134.7, 132.9, 132.5, 131.3, 131.2, 131.1, 130.0, 128.4, 122.9, 122.1, 102.9, 101.8, 91.2, 88.6 and 0.10 ppm. **IR** (neat): 2961, 2900, 2203, 2157, 1646, 1591, 1562, 1475, 1266, 1012 and 874 cm⁻¹. **HR ESI-MS**: [C₂₀H₁₈ClOSi]⁺ = [M+H]⁺ requires 337.0815; found 337.0806. **TLC**: R_f = 0.2 (19:1, hexane:EtOAc).

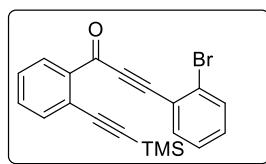
3-[3-(Trifluoromethyl)phenyl]-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (8h)



Followed general procedure C: Alcohol **S8** (480 mg, 1.29 mmol), IBX (469 mg, 1.67 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the ketone **8h** (356 mg, 0.96 mmol, 75%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.08 (1 H, d, *J* = 7.6 Hz), 7.89 (1 H, s), 7.82 (1 H, d, *J* = 7.7 Hz), 7.71 (1 H, d, *J* = 7.8 Hz), 7.63 (1 H, d, *J* = 7.6 Hz), 7.58-7.49 (2 H, m), 7.46 (1 H, t, *J* = 7.6 Hz) and 0.20 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.3, 138.6, 136.2, 135.2, 132.6, 131.3, 129.8 (C, q, *J* = 3.6 Hz), 129.4, 128.4, 127.3 (C, q, *J* = 3.6 Hz), 122.9, 121.4, 102.8, 101.9, 90.8, 88.7 and 0.14 ppm. **¹⁹F NMR** (282 MHz, CDCl₃): δ = -63.0 (s, 3 F, CF₃) ppm. **IR** (neat): 2961, 2204, 2158, 1650, 1480, 1436, 1334, 1010 and 873 cm⁻¹. **HR ESI-MS**: [C₂₁H₁₈F₃OSi]⁺ = [M+H]⁺ requires 371.1079; found 371.1077. **TLC**: R_f = 0.2 (19:1, hexane:EtOAc). **M. P.:** 187-190 °C.

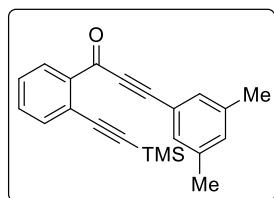
3-(2-Bromophenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (8i)



Followed general procedure C: Alcohol **S9** (311 mg, 0.80 mmol), IBX (294 mg, 1.0 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the ketone **8i** (280 mg, 0.73 mmol, 91%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.34 (1 H, d, *J* = 7.4 Hz), 7.72-7.57 (3 H, m), 7.57-7.41 (2 H, m), 7.41-7.27 (2 H, m) and 0.25 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.0, 138.5, 135.4, 135.2, 132.9, 132.6, 132.3, 131.9, 128.3, 127.4, 126.9, 122.9, 122.8, 103.2, 101.4, 91.2, 90.6 and 0.07 ppm. **IR** (neat): 3026, 2920, 1997, 1653, 1664, 1365, 1170 and 846 cm⁻¹. **HR ESI-MS**: [C₂₀H₁₈BrOSi]⁺ = [M+H]⁺ requires 381.0310; found 381.0305. **TLC**: R_f = 0.2 (19:1, hexane:EtOAc).

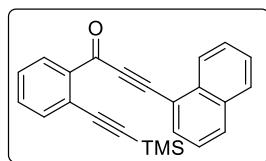
3-(3,5-Dimethylphenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (8j)



Followed general procedure C: Alcohol **S10** (400 mg, 1.20 mmol), IBX (438 mg, 1.56 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the ketone **8j** (360 mg, 1.09 mmol, 91%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.96 (1 H, d, *J* = 7.5 Hz), 7.48 (1 H, d, *J* = 7.4 Hz), 7.38-7.28 (2 H, m), 7.16-7.10 (2 H, m), 6.96 (1 H, s), 2.19 (6 H, s) and 0.08 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.7, 139.2, 138.4, 135.1, 132.9, 132.2, 131.3, 131.0, 128.3, 122.7, 120.0, 103.0, 101.4, 94.2, 87.7, 21.1 and 0.10 ppm. **IR** (neat): 2960, 2922, 2192, 2159, 1645, 1596, 1473, 1261, 1053 and 875 cm⁻¹. **HR ESI-MS:** [C₂₂H₂₃OSi]⁺ = [M+H]⁺ requires 331.1518; found 331.1409. **TLC:** R_f = 0.4 (19:1, hexane:EtOAc). **M.P.:** 60-63 °C.

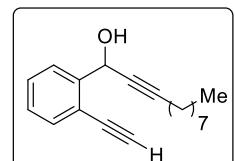
3-(Naphthalen-1-yl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-one (8k)



Followed general procedure C: Alcohol **S11** (400 mg, 1.29 mmol), IBX (411 mg, 1.46 mmol), dry EtOAc (4 mL) were stirred for 3 h at 80 °C. Purification by flash column chromatography (19:1 hexane:EtOAc) gave the ketone **8k** (370 mg, 1.0 mmol, 81%) as a red oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.42 (1 H, d, *J* = 8.1 Hz), 8.19 (1 H, d, *J* = 7.4 Hz), 8.02-7.82 (3 H, m), 7.71-7.43 (6 H, m) and 0.15 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 177.7, 139.4, 135.2, 133.8, 133.5, 133.2, 132.3, 131.6, 131.2, 128.6, 128.4, 127.8, 127.0, 126.1, 125.3, 122.8, 118.0, 103.0, 101.8, 92.9, 91.9 and 0.17 ppm. **IR** (neat): 3058, 2961, 2901, 2190, 1642, 1300 and 864 cm⁻¹. **HR ESI-MS:** [C₂₄H₂₁OSi]⁺ = [M+H]⁺ requires 353.1362; found 353.1356. **TLC:** R_f = 0.5 (19:1, hexane:EtOAc). **M. P.:** 82-85 °C.

1-(2-Ethynylphenyl)undec-2-yn-1-ol (S1)

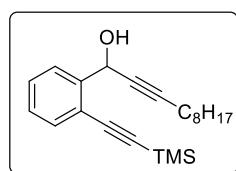


Followed procedure B: The TMS Protected alkyne **S1-a** (200 mg, 0.58 mmol), in MeOH (5 mL) and Potassium carbonate (162 mg, 1.17 mmol) were stirred for 15 min RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave the Terminal alkyne **S1** (140 mg, 0.52 mmol, 89%) as a yellow liquid.

¹H NMR (400 MHz, CDCl₃): δ = 7.72 (1 H, d, *J* = 7.6 Hz), 7.50 (1 H, d, *J* = 7.7 Hz), 7.38 (1 H, t, *J* = 7.7 Hz), 7.27 (1 H, t, *J* = 7.5 Hz), 5.87 (1 H, s), 3.36 (1 H, s), 2.63 (1 H, br s), 2.25 (2

H, td, $J = 7.1$ & 1.6 Hz), 1.52 (2 H, pent, $J = 7.4$ Hz), 1.43-1.33 (2 H, m), 1.26 (8 H, m) and 0.87 (3 H, t, $J = 6.9$ Hz) ppm. ^{13}C NMR (100 MHz, CDCl_3): $\delta = 143.6, 133.1, 129.4, 128.1, 126.8, 120.4, 87.9, 82.5, 81.2, 79.2, 63.1, 31.9, 29.2, 29.1, 28.9, 28.6, 22.7, 18.9$ and 14.2 ppm. IR (neat): 3298, 3064, 2952, 2912, 2858, 2226, 1458, 999 and 763 cm^{-1} . HR ESI-MS: $[\text{C}_{19}\text{H}_{25}\text{O}]^+ = [\text{M}+\text{H}]^+$ requires 269.1905; found 269.1907. TLC: $R_f = 0.2$ (9:1, hexane:EtOAc).

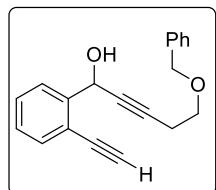
1-[2-[(Trimethylsilyl)ethynyl]phenyl]undec-2-yn-1-ol (S1-a)



Followed general procedure A: To an ice-cold solution of dec-1-yne (0.44 mL, 2.47 mmol), in anhydrous THF stirred for 15 min at 0 °C. Later added $^\circ\text{BuLi}$ (1.6 M in hexane) (1.5 mL, 2.47 mmol), stirred for 15 min. added 2-[(trimethylsilyl)ethynyl]benzaldehyde⁴ (200 mg, 1.23 mmol) drop wise. Then, warmed to rt for 3 h. Reaction mixture was diluted with aq. NH_4Cl solution (10 mL), extracted with ethyl acetate, dried (MgSO_4), concentrated, purification of the crude reaction mixture by flash column chromatography (9:1 Hexane:EtOAc) gave the aryl alcohol **S1-a** (350 mg, 1.02 mmol, 84%) as a yellow oil.

^1H NMR (400 MHz, CDCl_3): $\delta = 7.67$ (1 H, d, $J = 7.5$ Hz), 7.46 (1 H, d, $J = 7.4$ Hz), 7.34 (1 H, t, $J = 7.5$ Hz), 7.28-7.20 (1 H, m), 5.83 (1 H, s), 2.92-2.70 (1 H, m), 2.35-2.20 (2 H, m), 1.60-1.48 (2 H, m), 1.41-1.34 (2 H, m), 1.31-1.23 (8 H, m), 0.91-0.84 (3 H, m) and 0.27 (9 H, s) ppm. ^{13}C NMR (100 MHz, CDCl_3): $\delta = 143.6, 132.7, 129.1, 128.0, 126.6, 121.2, 102.5, 100.4, 87.7, 79.1, 63.5, 31.9, 29.2, 29.1, 29.0, 28.6, 22.7, 18.9, 14.2$ and -0.02 ppm. IR (neat): 3414, 2927, 2859, 2155, 1010 and 846 cm^{-1} . HR ESI-MS: $[\text{C}_{22}\text{H}_{33}\text{OSi}]^+ = [\text{M}+\text{H}]^+$ requires 341.2301; found 341.2290. TLC: $R_f = 0.3$ (9:1, hexane:EtOAc).

5-(Benzylxylo)-1-(2-ethynylphenyl)pent-2-yn-1-ol (S2)

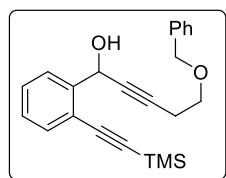


Followed general procedure B: The TMS Protected alkyne **S2-a** (250 mg, 0.69 mmol), in MeOH (5 mL) and Potassium carbonate (190 mg, 1.38 mmol) were stirred for 15 min RT. Purification by flash column chromatography (9:1 hexane:EtOAc) gave the Terminal alkyne **S2** (192 mg, 0.66 mmol, 96%) as a yellow liquid.

^1H NMR (400 MHz, CDCl_3): $\delta = 7.71$ (1 H, d, $J = 7.6$ Hz), 7.49 (1 H, d, $J = 7.4$ Hz), 7.36-7.26 (7 H, m), 5.86 (1 H, s), 4.53 (2 H, s), 3.61 (2 H, t, $J = 6.9$ Hz), 3.34 (1 H, s), 2.57 (2 H, t, $J = 6.6$ Hz) and 2.15 (1 H, s) ppm. ^{13}C NMR (100 MHz, CDCl_3): $\delta = 143.3, 138.1, 133.1, 129.5, 128.5, 128.2, 127.8, 126.8, 120.4, 84.3, 82.6, 81.1, 80.5, 73.0, 68.3, 63.0$ and 20.4 ppm. IR

(neat): 3350, 2826, 2687, 2115, 150, 1220 and 840 cm⁻¹. **HR ESI-MS:** [C₂₀H₁₉O₂]⁺ = [M+H]⁺ requires 291.1307; found 291.1309. **TLC:** R_f = 0.2 (9:1, hexane:EtOAc).

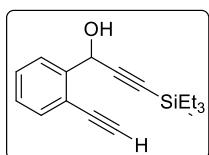
5-(BenzylOxy)-1-{2-[{(trimethylsilyl)ethynyl]phenyl}pent-2-yn-1-ol (**S2-a**)}



Followed general procedure A: To an ice-cold solution of dec-1-yne The [(but-3-yn-1-yloxy)methyl]benzene (395 mg, 2.45 mmol), in dry THF stirred for 15 min at 0 °C. Later added ⁷BuLi (1.6 M in hexane) (1.5 mL, 2.47 mmol), stirred for 15 min. added 2-[{(trimethylsilyl)ethynyl]benzaldehyde⁴ (250 mg, 1.23 mmol) drop wise. Then, warmed to rt for 2 h. Reaction mixture was diluted with aq. NH₄Cl solution (10 mL), extracted with ethyl acetate, dried (MgSO₄), concentrated, purification of the crude reaction mixture by flash column chromatography (9:1 Hexane:EtOAc) gave the aryl alcohol **S2-a** (420 mg, 1.02 mmol, 94%) as a yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.66 (1 H, d, J = 7.4 Hz), 7.46 (1 H, d, J = 7.2 Hz), 7.35-7.26 (7 H, m), 5.83 (1 H, s), 4.53 (2 H, s), 3.61 (2 H, t, J = 6.8 Hz), 2.58 (2 H, t, J = 5.5 Hz) and 0.26 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 143.3, 138.1, 132.7, 129.1, 128.6, 128.5, 128.0, 127.8, 126.7, 121.2, 102.4, 100.5, 84.0, 80.4, 73.0, 68.3, 63.3, 20.4 and -0.01 ppm. **IR** (neat): 3449, 2922, 2857, 1726, 1455, 1266 and 735 cm⁻¹. **HR ESI-MS:** [C₂₃H₂₆O₂NaSi]⁺ = [M+Na]⁺ requires 385.1600; found 385.1586. **TLC:** R_f = 0.4 (9:1, hexane:EtOAc).

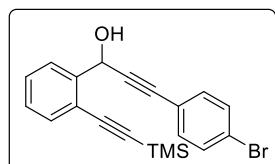
1-(2-Ethynylphenyl)-3-(triethylsilyl)prop-2-yn-1-ol (**S3**)



Followed general procedure A: To an ice-cold solution of triethylsilylacetylene (0.6 mL, 3.36 mmol), in dry THF stirred for 15 min at 0 °C. Later added ⁷BuLi (1.6 M in hexanes 2 mL, 3.36 mmol) stirred for 15 min. added 2-ethynylbenzaldehyde (366 mg, 2.8 mmol) drop wise. Then, warmed to rt for 3 h. Reaction mixture was diluted with aq. NH₄Cl solution (10 mL), extracted with ethyl acetate, dried (MgSO₄), concentrated, purification of the crude reaction mixture by flash column chromatography (9:1 Hexane:EtOAc) gave the (2-ethynylphenyl)-3-(triethylsilyl)propargyl alcohol **S3** (400 mg, 1.48 mmol, 53%) as a dark yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.74 (1 H, d, J = 7.6 Hz), 7.51 (1 H, d, J = 7.4 Hz), 7.39 (1 H, t, J = 7.4 Hz), 7.29 (1 H, t, J = 7.2 Hz), 5.89 (1 H, d, J = 4.8 Hz), 3.36 (1 H, s), 2.63 (1 H, br s), 0.99 (9 H, t, J = 7.8 Hz) and 0.62 (6 H, q, J = 7.8 Hz) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 142.8, 133.2, 129.4, 128.3, 126.9, 120.7, 105.5, 89.3, 82.7, 81.1, 63.5, 7.5 and 4.3 ppm. **IR** (neat): 3400, 2951, 2884, 2172, 1465, 1238, 1034 and 726 cm⁻¹. **HR ESI-MS:** [C₁₇H₂₃OSi]⁺ = [M+H]⁺ requires 271.1518; found 271.1514. **TLC:** R_f = 0.2 (9:1, hexane:EtOAc).

3-(4-Bromophenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-ol (S4)

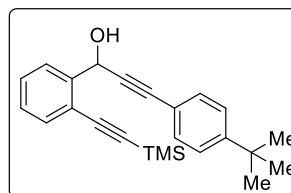


Followed procedure E: The [2-(trimethylsilylethynyl)phenyl]-propargyl alcohol⁷ (300 mg, 1.31 mmol), 1-bromo-4-iodobenzene (554 mg, 1.97 mmol), dry THF (7 mL), Et₃N (1 mL), CuI (37 mg, 0.19 mmol) and PdCl₂(PPh₃)₂ (9 mg, 0.01 mmol) were stirred for 12 h at RT.

Purification by flash column chromatography (9:1 hexane:EtOAc) mixture as eluent to yield coupled product [2-(alkynyl)phenyl]propargyl alcohol **S4** (450 mg, 1.17 mmol, 89%) as a pale yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.68 (1 H, d, *J* = 7.7 Hz), 7.50 (1 H, d, *J* = 7.5 Hz), 7.44 (2 H, d, *J* = 8.2 Hz), 7.38 (1 H, t, *J* = 7.41 Hz), 7.34-7.26 (3 H, m), 6.01 (1 H, d, *J* = 2.3 Hz), 2.98 (1 H, br s), and 0.26 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 142.7, 133.3, 133.0, 131.6, 129.3, 128.3, 126.7, 122.9, 121.6, 121.3, 102.3, 101.0, 89.3, 85.5, 63.9 and 0.00 ppm. **IR** (neat): 3432, 3056, 2927, 2854, 2155, 1484, 1446, 1263 and 753 cm⁻¹. **HR ESI-MS**: [C₂₀H₁₈ClOSi]⁺ = [M+H]⁺ requires 337.0815; found 337.0806. **TLC**: R_f = 0.3 (9:1, hexane:EtOAc). **M. P.**: 1107-110 °C.

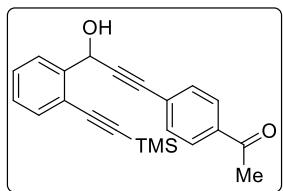
3-[4-(*tert*-Butyl)phenyl]-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-ol (S5)



Followed general procedure A: To an ice-cold solution of 1-(*tert*-butyl)-4-ethynylbenzene (0.44 mL, 2.47 mmol), in anhydrous THF stirred for 15 min at 0 °C. Later added ⁷BuLi (1.6 M in hexane) (1.5 mL, 2.47 mmol), stirred for 15 min. added 2-[(trimethylsilyl)ethynyl]benzaldehyde⁴ (200 mg, 1.23 mmol) drop wise. Then, warmed to rt for 3 h. Reaction mixture was diluted with aq. NH₄Cl solution (10 mL), extracted with ethyl acetate, dried (MgSO₄), concentrated, purification of the crude reaction mixture by flash column chromatography (9:1 Hexane:EtOAc) gave the aryl alcohol **S5** (240 mg, 0.62 mmol, 64%) as a yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.77 (1 H, d, *J* = 7.4 Hz), 7.53 (1 H, d, *J* = 7.2 Hz), 7.47-7.25 (6 H, m), 6.10 (1 H, s), 3.05 (1 H, br s), 1.34 (9 H, s) and 0.32 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 151.8, 143.1, 132.8, 131.6, 129.2, 128.1, 126.8, 125.3, 121.3, 119.6, 102.5, 100.6, 87.5, 86.7, 63.8, 34.8, 31.2 and 0.00 ppm. **HR ESI-MS**: [C₂₄H₂₈NaOSi]⁺ = [M+Na]⁺ requires 383.1807; found 383.1801. **TLC**: R_f = 0.2 (9:1, hexane:EtOAc).

1-{4-[3-Hydroxy-3-{2-[(trimethylsilyl)ethynyl]phenyl}prop-1-yn-1-yl}phenyl}ethan-1-one (S6)

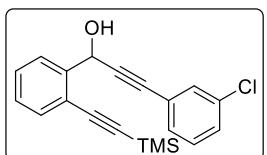


Followed procedure E: The [2-(trimethylsilyl)ethynyl]phenyl]-propargyl alcohol⁷ (300 mg, 1.31 mmol), 4-iodoacetophenone (485 mg, 1.9 mmol), dry THF (7 mL), Et₃N (1 mL), CuI (40 mg, 0.19 mmol) and PdCl₂(PPh₃)₂ (10 mg, 0.01 mmol) were stirred for 12 h at RT.

Purification by flash column chromatography (4:1 hexane:EtOAc) mixture as eluent to yield coupled product [2-(alkynyl)phenyl]propargyl alcohol **S6** (450 mg, 1.30 mmol, 99%) as a pale yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.88 (2 H, d, *J* = 8.0 Hz), 7.70 (1 H, d, *J* = 7.7 Hz), 7.57-7.46 (3 H, m), 7.38 (1 H, t, *J* = 7.5 Hz), 7.29 (1 H, t, *J* = 7.5 Hz), 6.06 (1 H, s), 3.26 (1 H, br s), 2.57 (3 H, s), 0.26 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 197.5, 142.6, 136.4, 132.9, 131.9, 129.2, 128.3, 128.2, 127.5, 126.6, 121.2, 102.3, 101.0, 91.7, 85.4, 63.7, 26.6 and 0.03 ppm. **IR** (neat): 3435, 2925, 2858, 2154, 1683, 1602, 1262 and 858 cm⁻¹. **HR ESI-MS**: [C₂₂H₂₃O₂Si]⁺ = [M+H]⁺ requires 347.1467; found 347.1512. **TLC**: R_f = 0.3 (4:1, hexane:EtOAc).

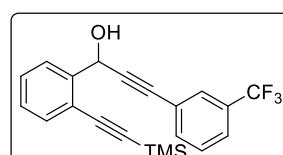
3-(3-Chlorophenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-ol (**S7**)



Followed procedure E: The [2-(trimethylsilyl)ethynyl]phenyl]-propargyl alcohol⁷ (360 mg, 1.57 mmol), 1-chloro-3-iodobenzene (567 mg, 2.36 mmol), dry THF (7 mL), Et₃N (1 mL), CuI (44 mg, 0.23 mmol) and PdCl₂(PPh₃)₂ (11 mg, 0.01 mmol) were stirred for 12 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) mixture as eluent to yield coupled product [2-(alkynyl)phenyl]propargyl alcohol **S7** (400 mg, 1.18 mmol, 75%) as a pale yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.68 (1 H, d, *J* = 7.6 Hz), 7.51 (1 H, d, *J* = 7.5 Hz), 7.44 (1 H, s), 7.41-7.26 (4 H, m), 7.26-7.19 (1 H, m), 6.02 (1 H, d, *J* = 5.3 Hz), 2.99 (1 H, s), 0.27 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 142.6, 134.2, 133.0, 131.8, 130.0, 129.6, 129.3, 128.9, 128.4, 126.7, 124.3, 121.3, 102.3, 101.0, 89.4, 85.1, 63.9 and 0.00 ppm. **HR ESI-MS**: [C₂₀H₁₉ClNaOSi]⁺ = [M+Na]⁺ requires 361.0791; found 361.0780. **TLC**: R_f = 0.3 (9:1, hexane:EtOAc).

3-[3-(Trifluoromethyl)phenyl]-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-ol (**S8**)

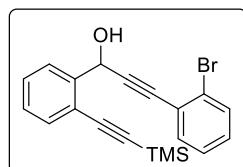


Followed general procedure E: The [2-(trimethylsilyl)ethynyl]-phenyl]propargyl alcohol⁷ (360 mg, 1.57 mmol), 1-iodo-3-(trifluoromethyl)benzene (644 mg, 2.36 mmol), dry THF (7 mL), Et₃N (1 mL), CuI (45 mg, 0.23 mmol) and PdCl₂(PPh₃)₂ (11 mg, 0.01 mmol)

were stirred for 12 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc) mixture as eluent to yield coupled product [2-(alkynyl)phenyl]propargyl alcohol **S8** (513 mg, 1.37 mmol, 88%) as a pale yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.72 (1 H, s), 7.69 (1 H, d, *J* = 7.7 Hz), 7.62 (1 H, d, *J* = 7.7 Hz), 7.56 (1 H, d, *J* = 7.8 Hz), 7.52 (1 H, d, *J* = 7.5 Hz), 7.44 (1 H, d, *J* = 7.8 Hz), 7.39 (1 H, t, *J* = 7.5 Hz), 7.30 (1 H, t, *J* = 7.4 Hz), 6.04 (1 H, d, *J* = 5.7 Hz), 3.21-2.99 (1 H, m) and 0.27 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 142.6, 134.9, 133.0, 129.3, 128.9, 128.7 (C, q, *J* = 3.9 Hz), 128.4, 126.7, 125.2 (C, q, *J* = 3.7 Hz), 123.6, 121.2, 102.3, 101.1, 89.9, 84.9, 63.8 and 0.05 ppm. **¹⁹F NMR** (282 MHz, CDCl₃): δ = -62.96 (s, 3 F, CF₃) ppm. **IR** (neat): 3373, 2962, 2155, 1481, 1333, 1133 and 871 cm⁻¹. **TLC**: R_f = 0.2 (4:1, hexane:EtOAc).

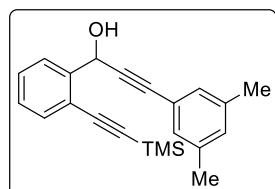
3-(2-Bromophenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-ol (**S9**)



Followed procedure E: The [2-(trimethylsilyl)ethynyl]phenyl]propargyl alcohol⁷ (360 mg, 1.57 mmol), 1-bromo-4-iodobenzene (674 mg, 2.36 mmol), dry THF (7 mL), Et₃N (1 mL), CuI (44 mg, 0.23 mmol) and PdCl₂(PPh₃)₂ (11 mg, 0.01 mmol) were stirred for 12 h at RT. Purification by flash column chromatography (9:1 hexane:EtOAc) mixture as eluent to yield coupled product [2-(alkynyl)phenyl]propargyl alcohol **S9** (340 mg, 0.89 mmol, 57%) as a pale yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.83 (1 H, d, *J* = 7.6 Hz), 7.57 (1 H, d, *J* = 7.9 Hz), 7.54-7.46 (2 H, m), 7.38 (1 H, t, *J* = 7.5 Hz), 7.31-7.22 (2 H, m), 7.16 (1 H, td, *J* = 1.2 & 7.7 Hz), 6.11 (1 H, d, *J* = 5.6 Hz), 3.06-2.82 (1 H, m) and 0.27 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 142.6, 133.8, 132.9, 132.5, 129.8, 129.2, 128.3, 127.1, 127.0, 125.7, 124.8, 121.4, 102.4, 100.8, 92.7, 85.2, 63.9 and 0.01 ppm. **IR** (neat): 3425, 3056, 2959, 2926, 2154, 1470, 1434 and 740 cm⁻¹. **HR ESI-MS**: [C₂₀H₁₈ClOSi]⁺ = [M+H]⁺ requires 337.0815; found 337.0806. **TLC**: R_f = 0.2 (9:1, hexane:EtOAc).

3-(3,5-Dimethylphenyl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-ol (**S10**)

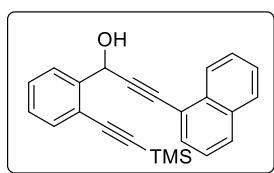


Followed procedure E: The [2-(trimethylsilyl)ethynyl]phenyl]propargyl alcohol⁷ (360 mg, 1.57 mmol), 1-iodobenzene-3,5-dimethylbenzene (551 mg, 2.36 mmol), dry THF (7 mL), Et₃N (1 mL), CuI (44 mg, 0.23 mmol) and PdCl₂(PPh₃)₂ (11 mg, 0.01 mmol) were stirred for 12 h at RT. Purification by flash column chromatography (4:1 hexane:EtOAc)

mixture as eluent to yield coupled product [2-(alkynyl)phenyl]propargyl alcohol **S10** (450 mg, 1.35 mmol, 86%) as a pale yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 7.71 (1 H, d, *J* = 7.7 Hz), 7.50 (1 H, d, *J* = 7.5 Hz), 7.37 (1 H, t, *J* = 7.5 Hz), 7.27 (1 H, t, *J* = 9.5 Hz), 7.10 (2 H, s), 6.95 (1 H, s), 6.03 (1 H, d, *J* = 5.7 Hz), 2.95-2.87 (1 H, m), 2.27 (6 H, s) and 0.27 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 143.1, 137.9, 132.9, 130.5, 129.6, 129.2, 128.2, 126.8, 122.2, 121.3, 102.5, 100.8, 87.4, 87.0, 63.9, 21.1 and 0.00 ppm. **IR** (neat): 3573, 3453, 2962, 2154, 1598, 1261 and 853 cm⁻¹. **HR ESI-MS**: [C₂₂H₂₅OSi]⁺ = [M+H]⁺ requires 333.1675; found 333.1626. **TLC**: R_f = 0.3 (4:1, hexane:EtOAc).

3-(Naphthalen-1-yl)-1-{2-[(trimethylsilyl)ethynyl]phenyl}prop-2-yn-1-ol (**S11**)

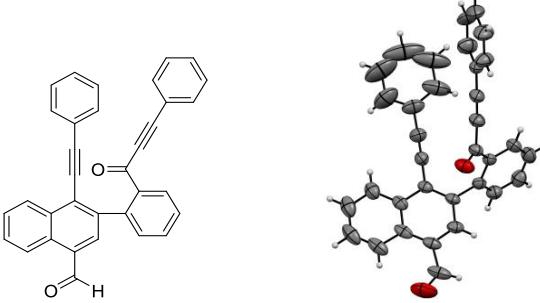


Followed procedure E: The [2-(trimethylsilylethynyl)phenyl]-propargyl alcohol⁷ (360 mg, 1.57 mmol), 1-iodonaphthalene (603 mg, 2.36 mmol), dry THF (7 mL), Et₃N (1 mL), CuI (45 mg, 0.23 mmol) and PdCl₂(PPh₃)₂ (11 mg, 0.01 mmol) were stirred for 12 h at RT.

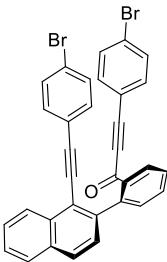
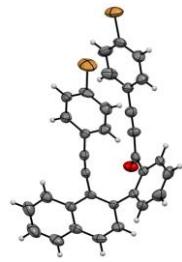
Purification by flash column chromatography (9:1 hexane:EtOAc) mixture as eluent to yield coupled product [2-(alkynyl)phenyl]propargyl alcohol **S11** (440 mg, 1.24 mmol, 79%) as a pale yellow oil.

¹H NMR (400 MHz, CDCl₃): δ = 8.33 (1 H, d, *J* = 8.0 Hz), 7.87-7.78 (3 H, m), 7.71 (1 H, d, *J* = 7.1 Hz), 7.59-7.47 (3 H, m), 7.45-7.37 (2 H, m), 7.31 (1 H, t, *J* = 7.4 Hz), 6.21 (1 H, d, *J* = 5.8 Hz), 3.06 (1 H, d, *J* = 5.5 Hz) and 0.27 (9 H, s) ppm. **¹³C NMR** (100 MHz, CDCl₃): δ = 143.1, 133.4, 133.2, 133.0, 130.8, 129.3, 129.1, 128.3, 126.9, 126.8, 126.5, 126.3, 125.2, 121.3, 120.3, 102.5, 101.0, 93.1, 84.8, 64.2 and 0.00 ppm. **IR** (neat): 3420, 2959, 2154, 1396, 1254, 1020 and 861 cm⁻¹. **HR ESI-MS**: [C₂₄H₂₃OSi]⁺ = [M+H]⁺ requires 355.1518; found 355.1495. **TLC**: R_f = 0.3 (9:1, hexane:EtOAc).

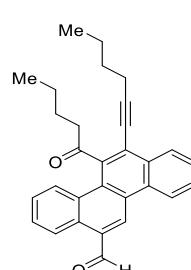
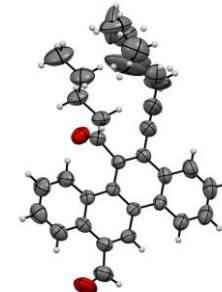
Crystallographic Data and Structure Refinements Summary for Compound **9a**

Molecular Structure (ORTEP Diagram)	
CCDC number	CCDC 1878982
Formula	C ₃₄ H ₂₀ O ₂
Formula weight	460.50
Color	Light Yellow
Temperature/K	296(2) K
Radiation	MoK\alpha
Wavelength/Å	0.71073 Å
Crystal system	Monoclinic
Space group	C2/c
a (Å)	31.9258 (15)
b (Å)	18.7977 (9)
c (Å)	8.2450(4) Å
α (°)	90
β (°)	96.472 (2)
γ (°)	90
Volume(Å ³)	4916.6 (4)
Z	8
Density (g/cm ³)	1.244
μ (1/mm)	0.076
F (000)	1920.0
θ (min, max)	3.16, 26.63
No. of unique reflns	4306 [R(int) = 0.0414]
No. of parameters	325
R _{obs} , wR ₂ _obs	0.0644, 0.1611
GooF	1.083

Crystallographic data and structure refinements summary for compound **10c**

Molecular Structure (ORTEP Diagram)	 
CCDC number	CCDC 1965357
Formula	C ₃₃ H ₁₈ Br ₂ O
Formula weight	590.29
Color	Light Yellow
Temperature/K	296(2) K
Radiation	MoK\alpha
Wavelength/Å	0.71073 Å
Crystal system	Monoclinic
Space group	C 1 c 1
a (Å)	13.5539(7)
b (Å)	17.8625(8)
c (Å)	12.4649(9)
α (°)	90
β (°)	122.657(2)
γ (°)	90
Volume(Å ³)	2540.8(3)
Z	4
Density (g/cm ³)	1.543
μ (1/mm)	3.215
F (000)	1176
θ (min, max)	2.12 to 24.83
No. of unique reflns	3604
No. of parameters	325
R _{obs} , wR ₂ _obs	R1 = 0.0635, wR2 = 0.0.835
Δρ _{min} , Δρ _{max} (eÅ ⁻³)	0.322 and -0.359
GooF	0.954

Crystallographic Data and Structure Refinements Summary for Compound 22

Molecular Structure (ORTEP Diagram)	 
CCDC number	CCDC 1878983
Formula	C ₃₀ H ₂₈ O ₂
Formula weight	420.20
Color	clear light brown
Temperature/K	296(2) K
Radiation	MoK\alpha
Wavelength/\AA	0.71073 \AA
Crystal system	triclinic unit
a (\AA)	8.9341(4) \AA
b (\AA)	10.7311(5)
c (\AA)	12.8284(5)
\alpha (°)	73.101(2)°
\beta (°)	82.359(2)°
\gamma (°)	83.312(2)°
Volume(\AA ³)	1162.40(9) \AA ³
Density (g/cm ³)	1.201 g/cm ³
\mu (1/mm)	3.215
F (000)	448 e ⁻
\theta (min, max)	of 25.00° (0.84 \AA resolution),
No. of unique reflns	4096
No. of parameters	325
R _{obs} , wR _{2_obs}	5.46%, 18.33%
\Delta\rho_{min}, \Delta\rho_{max} (e\AA ⁻³)	0.9820 and 0.9930
GooF	1.016.

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