Supporting Information

Bistrimethylsilylpropargylic ether: a versatile ambident synthon to access substituted allenynes ethers and α -substituted bispropargylic alcohols

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General Methods: All reagents are commercial grade and were used as received. All reactions were performed under inert atmosphere using anhydrous solvents which were dried and distilled before being used. Thin-layer chromatograms (TLC) and flash chromatography separations were respectively performed on precoated silica gel 60 F 254 plates and on silica gel 60 (230-400 mesh). ^1H NMR spectra were recorded at 400 MHz, shifts are relative to internal TMS. ^{13}C NMR spectra were obtained at 100 MHz. ESITOF mass spectra.. High resolution mass spectra were performed on a Maldi-tof (matrix: dihydroxybenzoic acid). IR spectra were recorded on NaCl plates as thin film.

General procedure for the preparation of bistrimethylsilyl propargylic ethers (3a-3c) : To a stirred solution of bistrimethylsilyl propargylic alcohol **2**¹⁻² (0.44 mmol) in CH_2Cl_2 (7 mL) was added the corresponding silyl chloride (0.48 mmol) and DMAP (0.48 mmol) and the solution was stirred at room temperature until completion. The mixture was concentrated, diluted with Et_2O and washed with brine. The organic layer was separated, dried over magnesium sulfate and concentrated under vacuum. The crude product was purified by flash chromatography on silica gel to yield the corresponding ether.

3-(trimethylsilyloxy)-1,5-bistrimethylsilylpenta-1,4-diyne (3a): From 1,5-bis(trimethylsilyl)penta-1,4-diyne-3-ol **2** (100 mg, 0.44 mmol) and trimethylsilyl chloride (0.17 mL, 0.48 mmol), crude **3a** (104 mg, 80%) was obtained following the general procedure. Purification by flash chromatography (Pentane) afforded **3a** (104 mg, 80%) as a colorless oil. ^1H NMR (CDCl_3) δ 5.17 (s, 1H), 0.23 (s, 9H), 0.19 (s, 18H); ^{13}C NMR (CDCl_3) δ 102.5, 88.8, 53.3, 0.45, -0.48; IR (neat, cm^{-1}) 2962, 2178, 1251, 1058, 845; MS (TOF) m/z ($\text{M}+\text{Na}^+$) = 319.

3-(triethylsilyloxy)-1,5-bistrimethylsilylpenta-1,4-diyne (3b): From alcohol **2** (100 mg, 0.44 mmol) and triethylsilyl chloride (80 μL , 0.48 mmol), ether **3b** (118 mg, 82%) was prepared

following the general procedure. Purification by flash chromatography (Pentane/Et₂O: 98/2) afforded **3b** (118 mg, 82%) as a colourless oil. ¹H NMR (CDCl₃) δ 5.18 (s, 1H), 0.99 (t, *J* = 8.0 Hz, 9H), 0.71 (q, *J* = 8.0 Hz, 6H), 0.18 (s, 18H); ¹³C NMR (CDCl₃) δ 102.6, 88.4, 53.4, 6.6, 4.8, -0.5; IR (neat, cm⁻¹) 2959, 2180, 1288, 1251, 1083, 844; MS (TOF) *m/z* (M+Na⁺) = 361.

(3-(*tert*-butyldimethylsilyloxy)penta-1,4-diyne-1,5-diyl)bistrimethylsilane (3c): From alcohol **2** (100 mg, 0.44 mmol) and *tert*-butyldimethylsilyl chloride (72 mg, 0.48 mmol) **3c** was obtained following the general procedure. Purification by flash chromatography (Pentane/Et₂O: 98/2) afforded **3c** (136 mg, 92%) as a colorless oil. ¹H NMR (CDCl₃) δ 5.19 (s, 1H), 0.93 (s, 9H), 0.20 (s, 24H); ¹³C NMR (CDCl₃) δ 102.7, 88.6, 53.8, 25.6, 18.1, -0.5, -4.4; IR (neat, cm⁻¹) 2960, 2180, 1252, 1082, 843; MS (TOF) *m/z* (M+Na⁺) = 361.

2-(3-trimethylsilyl-1-trimethylsilylethynylprop-2-ynoxy)tetrahydropyran (3e): To a stirred solution of alcohol **2** (100 mg, 0.44 mmol) in CH₂Cl₂ (8 mL) at room temperature was added triphenylphosphine hydrobromide (4 mg, 0.01 mmol) and dihydropyran (44 μL, 0.48 mmol). The resulting reaction was stirred until completion of the reaction. The mixture was concentrated, diluted with Et₂O and filtered through celite. The solution was concentrated under vacuum. Purification by flash chromatography (Pentane/Et₂O: 95/5) afforded **3e** as a white solid (104 mg, 75%). ¹H NMR (CDCl₃) δ 5.17 (s, 1H), 4.97 (t, *J* = 6.4 Hz, 1H), 3.90-3.85 (m, 1H), 3.55-3.53 (m, 1H), 1.90-1.56 (m, 6H), 0.19 (s, 18H); ¹³C NMR (CDCl₃) δ 100.1, 99.7, 95.3, 90.2, 89.4, 61.9, 55.4, 29.9, 25.3, 18.6, -

0.4; IR (neat, cm^{-1}) 2962, 2182, 1456, 1378, 1311, 1253, 1017, 852; mp: 47°C; MS (TOF) m/z ($\text{M}+\text{Na}^+$) = 331; HRMS calcd for $\text{C}_{16}\text{H}_{28}\text{O}_2\text{Si}_2\text{Na}$ [$\text{M}+\text{Na}^+$] = 331.1526, found 331.1532.

1,3-dimethoxy-5-(3-trimethylsilyl-1-trimethylsilylprop-2-ynyl)benzene

(3f): To a stirred solution of alcohol **2** (100 mg, 0.44 mmol) in THF (5 mL) at 0°C was added EtMgBr (0.6 mL, 1M in THF, 0.6 mmol), HMPA (0.3 mL) and 3,5-dimethoxybenzyl bromide (122 mg, 0.53 mmol) in solution in THF (1 mL). The mixture was refluxed for 1.5 h and diluted with Et₂O and washed with saturated ammonium chloride. The organic layer was separated, washed with H₂O and brine, and then was dried over magnesium sulfate and was concentrated under vacuum. The crude was purified by flash chromatography (Pentane/Et₂O: 98/2) to afford ether **3f** as a brown oil (67 mg, 40%). ¹H NMR (CDCl₃) δ 6.54 (d, J = 2.2 Hz, 2H), 6.40 (s, 1H), 4.99 (s, 1H), 4.65 (s, 2H), 3.80 (s, 6H), 0.21 (s, 18H); ¹³C NMR (CDCl₃) δ 160.8, 139.3, 105.9, 100.1, 99.5, 90.7, 69.0, 58.5, 55.3, -0.4; IR (neat, cm^{-1}) 2958, 2169, 1599, 1155, 844; MS (TOF) m/z ($\text{M}+\text{H}^+$) = 375.

2-(1-ethynylprop-2-ynyl)tetrahydropyran (4e): To a stirred solution of ether **3e** (77 mg, 0.25 mmol) in MeOH (6 mL) was added K₂CO₃ (35 mg, 0.25 mmol). The reaction mixture was stirred at room temperature for 1 h and then was quenched with saturated ammonium chloride (10 mL) and extracted with Et₂O (3 x 10 mL). The organic layer was separated, washed with H₂O, brine and was dried over anhydrous MgSO₄, filtered and concentrated under vacuum. The crude residue was purified by silica gel flash chromatography (Pentane/Et₂O: 99/1) to yield **4e** (29 mg, 70%) as a

colorless oil. ^1H NMR (CDCl_3) δ 5.20 (dd, $J = 2.4, 2.4$ Hz, 1H), 4.99 (t, $J = 2.8$ Hz, 1H), 3.89-3.84 (m, 1H), 3.59-3.54 (m, 1H), 2.56 (d, $J = 2.4$ Hz, 1H), 2.53 (d, $J = 2.4$ Hz, 1H), 1.90-1.63 (m, 6H); ^{13}C NMR (CDCl_3) δ 95.4, 78.9, 78.4, 73.5, 72.9, 61.9, 54.1, 29.8, 25.1, 18.5; IR (neat, cm^{-1}) 3286, 2946, 2121, 1442, 1119, 1018; MS (TOF) m/z ($\text{M}+\text{K}^+$) = 187.

1,3-dimethoxy-5-(1-ethynyl-prop-2-ynyloxymethyl)benzene (4f): To a stirred solution of ether **3f** (94 mg, 0.25 mmol) in MeOH (6 mL) was added K_2CO_3 (35 mg, 0.25 mmol). The reaction mixture was stirred at room temperature for 1 h and then was quenched with saturated ammonium chloride (10 mL) and extracted with Et_2O (3 x 10 mL). The organic layer was separated, washed with H_2O , brine and was dried over anhydrous MgSO_4 , filtered and concentrated under vacuum. The crude residue was purified by silica gel flash chromatography (Pentane/ Et_2O : 98/2) to afford **4f** (37 mg, 65%) as a colorless oil. ^1H NMR (CDCl_3) δ 6.55 (d, $J = 2.0$ Hz, 2H), 6.41 (t, $J = 2.4$ Hz, 1H), 5.03 (t, $J = 2.4$ Hz, 1H), 4.67 (s, 2H), 3.80 (s, 6H), 2.60 (d, $J = 2.4$ Hz, 2H); ^{13}C NMR (CDCl_3) δ 160.8, 138.9, 105.8, 100.1, 78.3, 73.9, 69.2, 57.3, 55.3; IR (neat, cm^{-1}) 3284, 2918, 2119, 1598, 1461, 1153, 1054, 665; MS (TOF) m/z ($\text{M}+\text{Na}^+$) = 253.

(3-(tert-butyldiphenylsilanyloxy)penta-1,4-diyne-1,5-diyl)trimethylsilane (5d): To a stirred solution of ether **4d** (250 mg, 0.78 mmol) in THF (25 mL) at -78°C was added dropwise $n\text{-BuLi}$ (0.5 mL, 1.6 M in hexane, 0.78 mmol). After 10 min of stirring, a solution of TMSCl (0.1 mL, 0.78 mmol) in THF (15 mL) was added. The mixture was warmed to RT for 2.5 h, quenched with saturated ammonium chloride, and extracted with Et_2O . The organic layer was separated, washed

with H₂O, brine and then was dried over magnesium sulfate, and was concentrated under vacuum. Purification of the crude by flash chromatography (Pentane/CH₂Cl₂: 9/1) afforded **5d** (183 mg, 43%) as a colorless oil, as well as **3d** (11%) and starting product **4d** (24%). **5d**: ¹H NMR (CDCl₃) δ 7.78-7.74 (m, 4H), 7.45-7.27 (m, 6H), 5.07 (d, *J* = 2.4 Hz, 1H), 2.49 (d, *J* = 2.4 Hz, 1H), 1.09 (s, 9H), 0.13 (s, 9H); ¹³C NMR (CDCl₃) δ 135.8, 132.6, 129.8, 127.5, 101.9, 89.3, 81.1, 71.9, 54.1, 26.6, 19.2, -0.5; IR (neat, cm⁻¹) 3290, 3072, 2933, 111, 845, 701; MS (TOF) *m/z* (M+Na⁺) = 413.

2-(1-ethynylpropa-1,2-dienyloxy)tetrahydropyran (8e): To a stirred solution of **3e** (200 mg, 0.64 mmol) in THF (12 mL) was added Bu₄NF (0.64 mL, 1M in THF, 0.64 mmol). The resulting brown solution was stirred at room temperature for 10 min. The reaction was quenched with saturated ammonium chloride and extracted with Et₂O. The organic layer was separated, washed with H₂O, brine and then dried over magnesium sulfate and concentrated under vacuum. The crude was purified by flash chromatography on silica gel (Pentane/Et₂O: 99/1) to afford **8e** (61 mg, 58%) as an unstable yellow oil. ¹H NMR (CDCl₃) δ 5.60 (dd, *J* = 1.2, 11.4 Hz, 1H), 5.53 (dd, *J* = 1.2, 11.4 Hz, 1H), 5.11 (t, *J* = 2.8 Hz, 1H), 3.91-3.82 (m, 1H), 3.60-3.56 (m, 1H), 3.35 (s, 1H), 1.90-1.43 (m, 6H); ¹³C NMR (CDCl₃) δ 206.8, 113.9, 96.1, 89.7, 83.4, 75.2, 65.7, 29.6, 25.4, 18.3; IR (neat, cm⁻¹) 3271, 2945, 2109, 1942, 1441, 1035; MS (TOF) *m/z* (M+Na⁺) = 187.

1,3-dimethoxy-5-(1-ethynylpropa-1,2-dienyloxymethyl)benzene (8f): To a stirred solution of **3f** (200 mg, 0.53 mmol) in THF (6 mL) was added *t*-BuOK (65 mg, 0.53 mmol). The resulting brown solution was stirred at room temperature for 10 min. The reaction was quenched with saturated ammonium chloride and extracted with Et₂O. The organic layer was separated, washed with H₂O,

brine and dried over magnesium sulfate, filtered and concentrated under vacuum. The crude was purified by flash chromatography (Pentane/CH₂Cl₂: 98/2) to afford **8f** as a yellow oil (72 mg, 59%). ¹H NMR (CDCl₃) δ 6.52 (d, *J* = 2.0 Hz, 2H), 6.40 (t, *J* = 2.4 Hz, 1H), 5.65 (d, *J* = 0.8 Hz, 2H), 4.59 (s, 2H), 3.80 (s, 6H), 3.35 (s, 1H); ¹³C NMR (CDCl₃) δ 205.5, 160.8, 138.8, 117.5, 105.5, 99.9, 92.2, 83.3, 75.4, 70.6, 55.3; IR (neat, cm⁻¹) 3269, 2937, 2117, 1723, 1599, 1462, 1205, 1155, 1066, 687; MS (TOF) *m/z* (M+Na⁺) = 253.

4-(tert-butylphenylsilyloxy)-6-trimethylsilyl-4-trimethylsilylhex-1-en-5-yne

(9b): To a stirred solution of **3d** (100 mg, 0.22 mmol) in THF (5 mL) at -78°C was added dropwise *n*-BuLi (0.22 mL, 1.6 M in hexane, 0.22 mmol) and a solution of allyliodide (20 μL, 0.24 mmol) in THF (1 mL). After stirring for 10 min at -78°C, the reaction was quenched with saturated ammonium chloride, warmed to RT and extracted with Et₂O. The organic layer was separated, washed with H₂O, brine and dried over magnesium sulfate, filtered and concentrated under vacuum. The crude was purified by flash chromatography with pentane to afford **9b** (49 mg, 45%) as yellow oil. ¹H NMR (CDCl₃) δ 7.77 (d, *J* = 7.1 Hz, 4H), 7.42-7.31 (m, 6H), 6.05-5.96 (m, 1H), 5.20-5.15 (m, 2H), 2.72 (d, *J* = 7.2 Hz, 2H), 1.04 (s, 9H), -0.03 (s, 18H); ¹³C NMR (CDCl₃) δ 136.4, 134.3, 132.7, 129.1, 126.9, 118.7, 104.9, 88.9, 65.2, 50.6, 26.8, 19.3, -0.6; IR (neat, cm⁻¹) 3074, 3050, 2960, 2174, 1428, 1250, 110, 843, 700; MS (TOF) *m/z* (M+Na⁺) = 525; HRMS calcd for C₃₀H₄₂OSi₃Na [M+Na⁺] = 525.2441, found 525.2439.

2-(1,3-bistrimethylsilyl-1-trimethylsilylprop-2-ynyl)oxytetrahydropyran (9c): To a stirred solution of **3e** (60 mg, 0.19 mmol) in THF (5 mL) at -78°C was added dropwise *n*-BuLi (0.12

mL, 1.6 M in hexane, 0.19 mmol) and then a solution of TMSCl (25 μ L, 0.19 mmol) in THF (1 mL). After stirring for 10 min at -78°C , the reaction was quenched with saturated ammonium chloride, warmed to RT and extracted with Et₂O. The organic layer was separated, washed with H₂O, brine and dried over magnesium sulfate, filtered and concentrated under vacuum. The crude was purified by flash chromatography (Pentane/Et₂O: 99/1) to afford **9c** as a yellow oil (58 mg, 79%). ¹H NMR (CDCl₃) δ 5.3 (s, 1H), 4.02-3.96 (m, 1H), 3.54-3.51 (m, 1H), 1.89-1.54 (m, 6H), 0.20 (s, 9H), 0.16 (s, 18H); ¹³C NMR (CDCl₃) δ 103.9, 103.1, 95.4, 93.7, 90.5, 62.6, 61.4, 30.7, 25.6, 18.7, -0.1, -4.8; IR (neat, cm⁻¹) 2959, 2152, 1249, 844, 758; MS (TOF) m/z (M+Na⁺) = 403.

2-(1,1-bis(trimethylsilyl)ethynylbut-3-enyloxy)tetrahydropyran (9d): To a stirred solution of **3e** (200 mg, 0.64 mmol) in THF (5 mL) at -78°C was added dropwise *n*-BuLi (0.4 mL, 1.6 M in hexane, 0.64 mmol) and then a solution of allyliodide (60 μ L, 0.64 mmol) in THF (1 mL). After stirring for 10 min at -78°C , the reaction was quenched with saturated ammonium chloride, warmed to RT and extracted with Et₂O. The organic layer was separated, washed with H₂O, brine and dried over magnesium sulfate, filtered and concentrated under vacuum. The crude was purified by flash chromatography with pentane to afford **9d** as a yellow oil (177 mg, 81%). ¹H NMR (CDCl₃) δ 5.99-5.89 (m, 1H), 5.29 (t, J = 3.2 Hz, 1H), 5.18-5.14 (m, 2H), 4.04-3.98 (m, 1H), 3.54-3.48 (m, 1H), 2.68 (d, J = 8.0 Hz, 2H), 1.86-1.56 (m, 6H), 0.17 (s, 18H); ¹³C NMR (CDCl₃) δ 132.3, 118.6, 103.8, 102.2, 95.7, 90.5, 88.6, 68.2, 62.4, 48.3, 30.9, 25.4, 19.3, -0.2; IR (neat, cm⁻¹) 2958, 2173, 1251, 844, 760; MS (TOF) m/z (M+Na⁺) = 371; HRMS calcd for C₁₉H₃₂O₂Na [M+Na⁺] = 371.1839, found 371.1822.

2-(1-methyl-3-trimethylsilyl-1-trimethylsilylprop-2-yn-1-yloxy)tetrahydro-pyran

(9e): To a stirred solution of **3e** (100 mg, 0.32 mmol) in THF (5 mL) at -78°C was added dropwise *n*-BuLi (0.2 mL, 1.6 M in hexane, 0.32 mmol) and then a solution of methyl iodide (20 μL , 0.32 mmol) in THF (1 mL). After stirring for 10 min at -78°C , the reaction was quenched with saturated ammonium chloride, warmed to RT and extracted with Et_2O . The organic layer was separated, washed with H_2O , brine and then dried over magnesium sulfate and concentrated under vacuum. The crude was purified by flash chromatography with pentane to afford **9e** as a yellow oil (55 mg, 60%). ^1H NMR (CDCl_3) δ 5.23 (t, $J = 2.8$ Hz, 1H), 4.03-3.98 (m, 1H), 3.54-3.50 (m, 1H), 1.86-1.54 (m, 12H), 0.18 (s, 18H); ^{13}C NMR (CDCl_3) δ 104.8, 103.8, 96.1, 88.6, 87.4, 65.1, 62.8, 31.9, 31.2, 25.3, 19.6, -0.2; IR (neat, cm^{-1}) 2958, 2175, 1442, 1365, 1251, 844, 760; MS (TOF) m/z ($\text{M}+\text{Na}^+$) = 345; HRMS calcd for $\text{C}_{17}\text{H}_{30}\text{O}_2\text{Si}_2\text{Na}$ [$\text{M}+\text{Na}^+$] = 345.1682, found 345.1682.

1,3-dimethoxy-5-(1-(3,5-dimethoxybenzyloxy)-3-trimethylsilyl-1-trimethylsilylprop-2-yn-1-yloxy)methylbenzene (9f): To a stirred solution of **3f** (150 mg, 0.67 mmol) in THF (5 mL) at -78°C was added dropwise *n*-BuLi (0.6 mL, 1.6 M in hexane, 0.98 mmol), HMPA (0.05 mL) and a solution of 3,5-dimethoxybenzyl bromide (271 mg, 1.7 mmol) in THF (1 mL). The reaction was warmed to RT for 2 h and was quenched with saturated ammonium chloride and diluted with Et_2O . The organic layer was separated, washed with H_2O , brine and then dried over magnesium sulfate, and was concentrated under vacuum. The crude was purified by flash chromatography (Pentane/ Et_2O : 98/2) to afford **9f** as a brown oil (140 mg, 40%). ^1H NMR (CDCl_3) δ 6.58 (s, 2H),

6.51 (s, 2H), 6.38 (s, 2H), 4.71 (s, 2H), 3.77 (s, 12H) ; 3.21 (s, 2H), 0.17 (s, 18H); ^{13}C NMR (CDCl_3)
 δ 160.6, 159.9, 140.9, 137.4, 109.5, 105.4, 102.6, 99.6, 99.1, 90.9, 70.5, 67.7, 55.2, 55.1, 49.0, -0.3;
IR (neat, cm^{-1}) 2960, 2164, 1598, 1468, 1202, 1161, 1051, 832; MS (TOF) m/z ($\text{M}+\text{Na}^+$) = 547.

5.301

209.058

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76.938
76.621

65.163
62.798

31.905
31.765
31.222
25.337
19.647

-0.309
-0.366

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