Electronic Supplementary Material (ESI) for Chemical Communications

Point-to-Helical Chirality Transfer for a Scalable and Resolution-Free Synthesis of a Helicenoidal DMAP Organocatalyst

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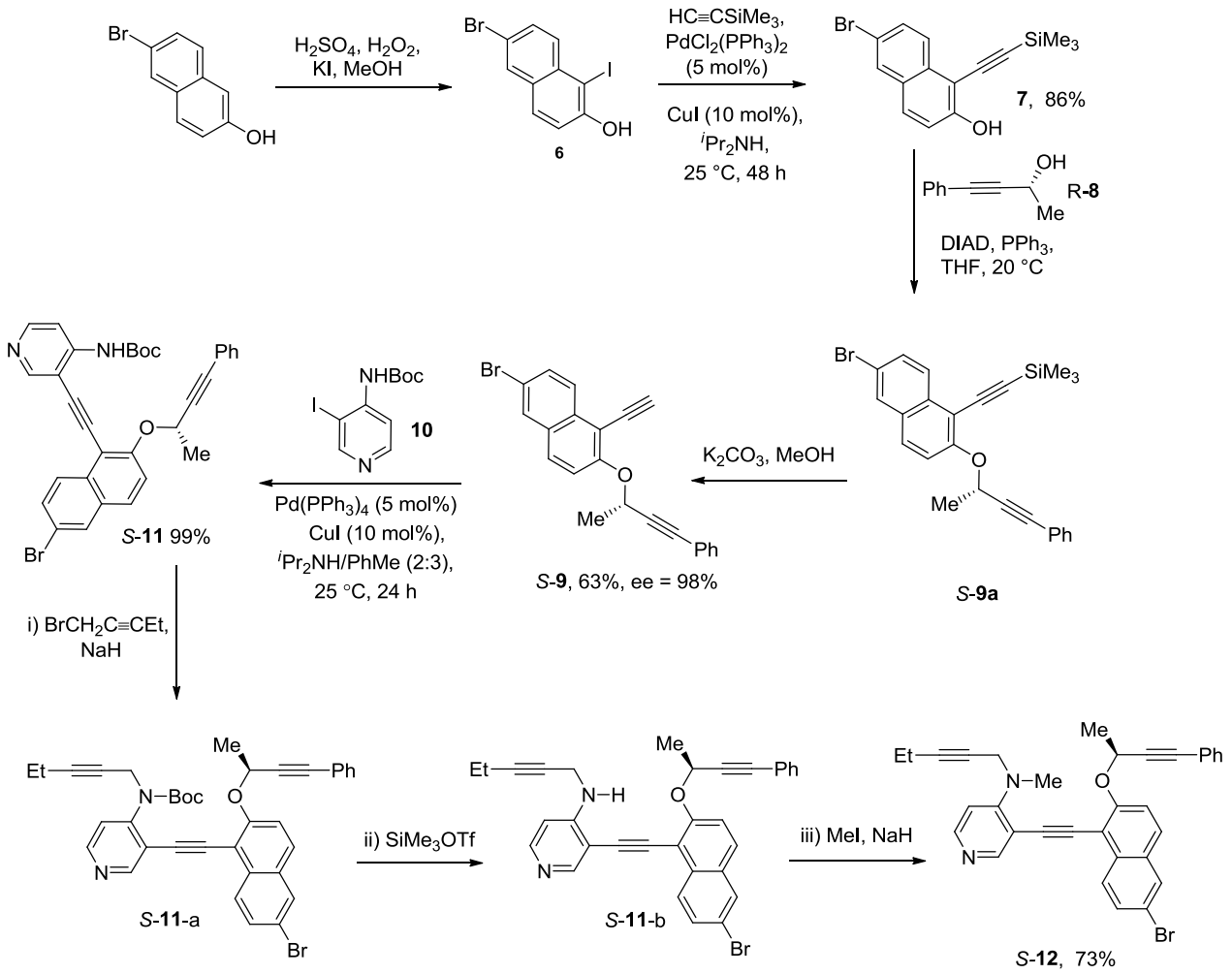
I GENERAL INFORMATION

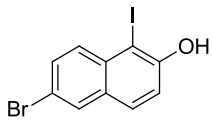
All reagents were purchased from commercial suppliers: Acros Organics, Alfa Aesar or Sigma Aldrich and used without further purification. Reactions were monitored by thin layer chromatography (TLC) using pre-coated MN Alugram Sil G/UV254 silica gel 60 aluminium backed plates. Plates were developed using standard techniques, UV light followed by a chemical dip, $KMnO_4$ or bromocresol green. Flash chromatography was performed on chromatography grade, silica, 60 Å particle size 35-70 micron from Fisher Scientific using the solvent system as stated. ^{1}H and ^{13}C NMR was performed Brüker Avance 400 (^{1}H 400 MHz and ^{13}C 100 MHz) and Brüker Avance 500 (^{1}H 500 MHz and ^{13}C 125 MHz) as stated. Chemical shifts are reported in parts per million (ppm) relative to tetramethylsilane (TMS) (δ = 0.00). Coupling constants are reported in Hertz (Hz) and signal multiplicity is denoted as singlet (s), doublet (d), triplet (t), quartet (q), quintet (quin.), sextet (sex.), septet (sept.), multiplet (m), and broad (br).

II EXPERIMENTAL DATA

The scheme presented in the manuscript for the synthesis of **5**, has been expanded in the scheme below in this supplementary data file to allow explicit numbering of intermediates which were not numbered in the manuscript but presented as multi-step transformation, i.e. **6**, *S*-**9a**, *S*-**11a** and *S*-**11b**.

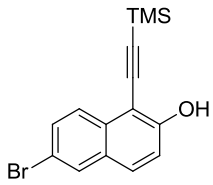


6-Bromo-1-iodonaphthalen-2-ol (6)



To a solution of sulphuric acid (1.80 mL, 33.6 mmol, 1.5 eq.) in methanol (112 mL) was added 6-bromonaphthalen-2-ol (5.00 g, 22.4 mmol, 1.0 eq.). To this was added potassium iodide (4.10 g, 24.6 mmol, 1.1 eq.) followed by hydrogen peroxide (35 wt. %) (1.37 mL, 89.6 mmol, 4.0 eq.) and the reaction left to stir for 4 h. On completion of the reaction the organics were extracted with DCM (2 × 30 mL), combined and washed with Na_2SO_3 (sat.) (50 mL), water (50 mL), and brine (50 mL) and dried over $MgSO_4$. The solvents were removed *in vacuo* and the crude mixture isolated as an off-white solid and used for the next step without further purification (7.43 g, 95%). υ_{max}(film)/cm^{-1}: 3439, 3228, 2923, 1589; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.86 (d, 1H, *J* = 2.2 Hz), 7.75 (d, 1H, *J* = 9.1 Hz), 7.59 (d, 1H, *J* = 8.8 Hz), 7.56 (dd, 1H, *J* = 9.1, 2.2 Hz), 7.23 (d, 1H, *J* = 8.8 Hz), 5.80 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 154.3, 133.7, 132.4, 131.6, 130.7, 130.3, 129.9, 118.3, 117.7, 86.2; (ESI HRMS) *m/z* calcd. for $C_{10}H_5BrIO$ 346.8569 $(M-H)^-$, found 346.8582.

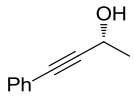
6-Bromo-1-((trimethylsilyl)ethynyl)naphthalen-2-ol (7)



To a solution of naphthol **6** (3.00 g, 8.60 mmol, 1.0 eq.) in diisopropylamine (50 mL) at rt was added bis(triphenylphosphine)palladium(II) dichloride (302 mg, 0.430 mmol, 5 mol %), copper(I) iodide (164 mg, 0.860 mmol, 10 mol %) and trimethylsilylacetylene (3.60 mL, 25.8 mmol, 3.0 eq.) dropwise. The resulting mixture was stirred at rt for 48 h. The solvent was then evaporated to yield the crude product which was purified by flash chromatography (19:1 Pet/EtOAc). The desired product was isolated as a brown oil (2.22 g, 81%); υ_{max} (film)/cm^{-1}: 3487, 2981, 2927, 2232, 1569; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.93 (d, 1H, *J* = 8.8 Hz), 7.90 (d, 1H, *J* = 1.9 Hz), 7.64 (d, 1H, *J* = 9.1 Hz), 7.59 (dd, 1H, *J* = 8.8, 1.9 Hz), 7.20 (d, 1H, *J* = 9.1 Hz), 6.20 (s, 1H), 0.36

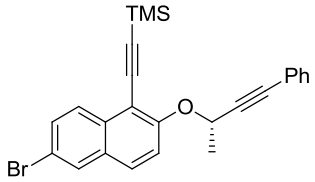
(s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 157.2, 132.4, 131.0, 130.5, 130.1, 129.8, 127.0, 118.1, 117.7, 108.3, 103.5, 97.1, 0.44; (ESI HRMS) *m/z* calcd. for $C_{15}H_{14}BrOSi$ 316.9997 $(M-H)^-$, found 317.0006.

(*R*)-4-Phenylbut-3-yn-2-ol (*R*-8)[1]



To a solution of iodobenzene (5.27 mL, 47.1 mmol, 1.1 eq.) in diisopropylamine (57 mL) was added bis(triphenylphosphine)palladium(II) dichloride (210 mg, 0.30 mmol, 0.7 mol %) and copper(I) iodide (195 mg, 1.03 mmol, 1.4 mol %). To the resultant mixture was added (*R*)-but-3-yn-2-ol (3.37 mL, 42.8 mmol, 1.0 eq.) dropwise at 0 ^oC. The mixture was allowed to warm to rt and the reaction stirred for 2 h. Upon completion of the reaction, solvents were removed *in vacuo* prior to purification by flash chromatography (100% DCM), yielding the desired product as an orange oil (6.26 g, 100%). $[\alpha]_D^{25}$ = +35 (*c* 1.0, DCM, *ee* = 98%); lit.[1] $[\alpha]_D^{25}$= +37 (*c* 0.8, $CHCl_3$, *ee* = 98%). Chiral HPLC analysis performed using CHIRALCEL OB, 19:1 Hex/IPA, 254 nm, 1.0 mL/min, t_R = 17.3 (major), t_R = 19.6 (minor). All other data as previously stated.

(*S*)-((6-Bromo-2-((4-phenylbut-3-yn-2-yl)oxy)naphthalen-1-yl)ethynyl)trimethylsilane (*S*-9a)

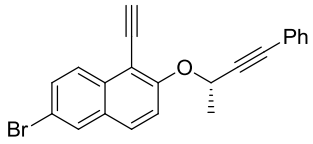


To a solution of **7** (1.05 g, 3.29 mmol, 1.1 eq.) in THF (9 mL) was added triphenylphosphine (0.940 g, 3.59 mol, 1.2 eq.) followed by (*R*)-4-phenylbut-3-yn-2-ol *R*-**8** (3.71 mL, 2.99 mmol, 1.0 eq.). The resulting mixture was cooled to 0 ^oC and then diisopropyl azodicarboxylate (638 µL, 3.29 mmol, 1.1 eq.) was added dropwise. The reaction was warmed to rt and left to stir under argon for 12 h. Removal of the solvent *in vacuo* followed by purification by flash chromatography (19:1 Pet /THF) gave the desired product as an orange oil (100 mg, 63%). $[\alpha]_D^{25}$ = -11 (*c* 0.8,

[1] A. Kawachi, H. Maeda, H.Nakamura, N. Doi, and K. Tamao *J. Am. Chem. Soc.* 2001, **123**, 3143.

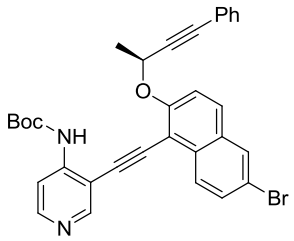
DCM, *ee* = 98%). Chiral HPLC analysis performed using CHIRALPAK AD, 99:1 Hex/IPA, 254 nm, 0.25 mL/min, t_R = 21.67 (minor), t_R = 23.12 (major); All other data as previously stated.

(*S*)-6-Bromo-1-ethynyl-2-((4-phenylbut-3-yn-2-yl)oxy)naphthalene (*S*-9)



To a solution of *S*-**9a** (220 mg, 0.490 mmol, 1.0 eq.) in MeOH (15 mL) was added potassium carbonate (170 mg, 1.23 mmol, 2.5 eq.) and the reaction left to stir at rt for 1 h. Following completion of the reaction the mixture was filtered and the solvent removed *in vacuo*. The crude mixture was taken up in EtOAc (15 mL), washed with water (10 mL), brine (10 mL) and then dried over $MgSO_4$. Following filtration, the solvent was removed under reduced pressure to give the crude product which was purified by flash chromatography (19:1 Pet/THF) to give the desired product as an off white amorphous solid (183 mg, 99%). $[\alpha]_D^{25}$ = -15 (*c*, 1.0, DCM); υ_{max} (film)/cm^{-1}: 3293, 2231, 2163; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 8.14 (d, 1H, *J* = 8.8 Hz), 7.91 (d, 1H, *J* = 1.9 Hz), 7.70 (d, 1H, *J* = 9.1 Hz), 7.57 (dd, 1H, *J* = 8.8, 1.9 Hz), 7.49 (d, 1H, *J* = 9.1 Hz), 7.37–7.32 (m, 2H), 7.28–7.22 (m, 3H), 5.27 (q, 1H, *J* = 6.6 Hz), 3.70 (s, 1H), 1.83 (d, 3H, *J* = 6.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 158.8, 133.6, 132.0, 130.9, 130.4, 130.3, 129.4, 128.9, 128.6, 127.6, 122.6, 118.8, 118.2, 108.1, 88.3, 87.5, 86.8, 67.1, 30.7, 22.8; (ESI HRMS) *m/z* calcd. for $C_{22}H_{15}BrONa$ 397.0204 $(M+Na)^+$, found 397.0214.

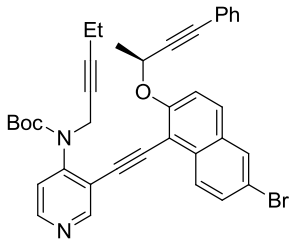
(*S*)-*tert*-Butyl (3-((6-bromo-2-((4-phenylbut-3-yn-2-yl)oxy)naphthalen-1-yl)ethynyl)pyridin-4-yl)carbamate (*S*-11)



To a solution of carbamate **10** (0.680 g, 2.11 mmol, 1.1 eq.) in diisopropylamine (12 mL) was added tetrakis(triphenylphosphine)palladium(0) (111 mg, 96.0 μmol, 5 mol %) and copper(I)

iodide (37.0 mg, 0.190 mmol, 10 mol %). The mixture was degassed by passing nitrogen through the solution and stirred for 15 min at rt. To this was added a degassed solution of *S*-**9** (0.720 g, 1.92 mmol, 1.0 eq.) in PhMe (29 mL) *via* syringe pump over a period of 30 min. The resultant mixture was left to stir at rt for 24 h. Evaporation of the solvents and subsequent purification of the residue by flash chromatography (4:1 Pet/EtOAc) afforded the title product as a yellow oil (1.08 g, 99%). $[\alpha]_D^{25}$ = +171 (*c* 0.8, DCM); υ_{max} (film)/cm^{-1}: 3380, 2983, 2220, 1736; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 8.78 (br s, 1H), 8.49 (br s, 1H), 8.22 (br s, 1H), 8.09 (d, 1H, *J* = 8.8 Hz), 7.91 (d, 1H, *J* = 1.6 Hz), 7.77 (s, 1H), 7.72 (d, 1H, *J* = 9.1 Hz), 7.60 (dd, 1H, *J* = 8.8, 1.6 Hz), 7.57 (d, 1H, *J* = 9.1 Hz), 7.43– 7.37 (m, 2H), 7.37–7.27 (m, 3H), 5.30 (q, 1H, *J* = 6.6 Hz), 1.92 (d, 3H, *J* = 6.6 Hz), 1.58 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 157.8, 152.4, 152.0, 150.1, 146.0, 132.4, 1318, 131.0, 130.3, 130.0, 129.8, 129.0, 128.5, 127.0, 122.2, 118.7, 116.7, 111.5, 107.3, 93.4, 91.3, 87.9, 87.1, 82.4, 77.6, 66.6, 28.5, 22.8; (ESI HRMS) *m/z* calcd. for $C_{32}H_{28}BrN_2O_3$ 567.1283 $(M+H)^+$, 567.1278.

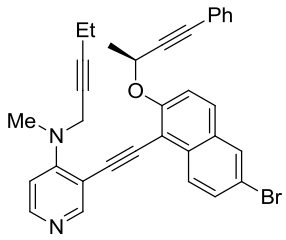
(*S*)-*tert*-Butyl (3-((6-bromo-2-((4-phenylbut-3-yn-2-yl)oxy)naphthalen-1-yl)ethynyl)pyridin-4-yl)(pent-2-yn-1-yl)carbamate (*S*-11a)



To a solution of carbamate *S*-**11** (1.14 g, 2.01 mmol, 1.0 eq.) in DMF (20 mL) was added sodium hydride (241 mg, 10.0 mmol, 5.0 eq.) and 1-bromo-2-pentyne (512 µl, 5.02 mmol, 2.5 eq.) dropwise. The resultant mixture was left to stir for 1 h at rt. The reaction was quenched *via* the addition of water (10 mL) and the organics extracted with EtOAc (4 × 5 mL). The organics were combined, washed with LiCl (sat.) (4 × 5 mL), brine (20 mL) and dried over $MgSO_4$ before being filtered and then concentrated under reduced pressure. The crude mixture was purified by flash chromatography (4:1 Pet/EtOAc) to afford the desired product as an orange oil (0.950 g, 75%). $[\alpha]_D^{25}$ = +158 (*c* 0.6, DCM); υ_{max} (film)/cm^{-1}: 2979, 2183, 2002, 1708; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 8.96 (s, 1H), 8.61 (d, 1H, *J* = 5.0 Hz), 8.21 (d, 1H, *J* = 8.8 Hz), 7.94 (d, 1H, *J* = 1.6 Hz), 7.73 (d, 1H, *J* = 9.1 Hz), 7.67 (d, 1H, *J* = 8.8 Hz), 7.55 (d, 1H, *J* = 9.1 Hz), 7.42–7.35 (m, 3H), 7.32–7.22 (m, 3H); 5.35 (q, 1H, *J* = 6.4 Hz), 4.67 (s, 2H), 2.09 (q, 2H, *J* = 7.4 Hz), 1.92 (d,

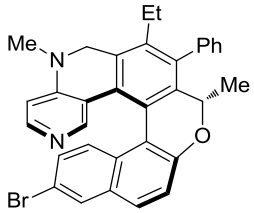
3H, *J* = 6.4 Hz), 1.43 (s, 9H), 1.01 (t, 3H, *J* = 7.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 157.8, 154.0, 153.2, 149.9, 149.3, 132.9, 131.9, 130.9, 130.2, 130.1, 129.6, 128.8, 128.4, 127.4, 123.5, 122.3, 120.1, 118.7, 117.3, 107.9, 93.5, 91.4, 88.0, 86.7, 81.8, 77.6, 74.6, 66.5, 39.0, 28.3, 22.6, 13.9, 12.5; (ESI HRMS) *m/z* calcd. for $C_{37}H_{34}BrN_2O_3$ 633.1753 $(M+H)^+$, found 633.1747.

(*S*)-3-((6-Bromo-2-((4-phenylbut-3-yn-2-yl)oxy)naphthalen-1-yl)ethynyl)-N-methyl-N-(pent-2-yn-1-yl)pyridin-4-amine (*S*-12)



To a solution of carbamate *S*-**11**a (1.00 g, 1.58 mmol, 1.0 eq.) in DCM (15 mL) was added triethylamine (550 µL, 3.95 mmol, 2.5 eq.) followed by trimethylsilyl trifluoromethanesulfonate (714 µL, 3.95 mmol, 2.5 eq.) dropwise. The reaction was left to stir at rt for 1 h. The reaction was quenched *via* addition of NH_4Cl (sat.) (10 mL) and the organics extracted with DCM (3 × 10 mL). The organics were combined, washed with brine (15 mL) and dried over $MgSO_4$ before being concentrated under reduced pressure. The residue was re-dissolved in DMF (25 mL) before sodium hydride (241 mg, 10.02 mmol, 5.0 eq.) and iodomethane (312 µl, 5.01 mmol, 2.5 eq.) were subsequently added. The reaction was left to stir at rt for 1 h. The reaction was quenched by addition of water (10 mL) and the organics extracted with EtOAc (4 × 5 mL). The organics were combined, washed with LiCl (sat.) (4 × 5 mL), brine (15 mL) and dried over $MgSO_4$ before being filtered and then concentrated under reduced pressure. The resultant residue was purified *via* flash chromatography (1:1 Pet/EtOAc) to give the title product as an orange oil (516 mg, 60%). $[\alpha]_D^{25}$ = +163 (*c* 0.4, DCM); υ_{max} (film)/cm^{-1}: 2977, 2937, 2232, ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 8.67 (s, 1H), 8.29(d, 1H, *J* = 6.0 Hz), 8.21 (d, 1H, *J* = 8.8 Hz), 7.95 (s, 1H), 7.72 (d, 1H, *J* = 9.1 Hz), 7.61 (dd, 1H, *J* = 9.1, 1.7 Hz), 7.54 (d, 1H, *J* = 8.8 Hz), 7.40–7.36 (m, 2H), 7.33–7.24 (m, 3H), 6.80 (d, 1H, *J* = 6.0 Hz), 5.33 (d, 1H, *J* = 6.5 Hz), 4.45 (s, 2H), 3.28 (s, 3H), 2.15 (q, 2H, *J* = 7.6 Hz), 1.86 (d, 3H, *J* = 6.5 Hz), 1.07 (t, 3H, *J* = 7.6 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 157.6, 156.4, 155.5, 149.3, 133.0, 132.0, 130.8, 130.4, 130.3, 129.0, 128.9, 128.6, 127.6, 122.4, 118.7, 117.3, 111.2, 109.2, 108.9, 96.6, 91.3, 88.2, 87.4, 86.8, 74.2, 66.5, 43.9, 39.5, 22.8, 14.2, 12.7; (ESI HRMS) *m/z* calcd. for $C_{33}H_{28}BrN_2O$ 547.1385 $(M+H)^+$, found 547.1380.

(*P*,*S*)-13-Bromo-4-ethyl-2,6-dimethyl-3-phenyl-5,6-dihydro-2*H*-naphtho[2',1':3,4]isochromeno[6,5-c][1,6]naphthyridine (5)



To a solution of triyne *S*-**12** (123 mg, 0.220 mmol, 1.0 eq.) in PhMe (6 mL) at rt was added a solution of tris(triphenylphosphine)rhodium(I) chloride (12.0 mg, 10 µmol, 5 mol %) in PhMe (1 mL) dropwise. The solution was heated to 110 ^{o}C and left to stir for 12 h. Following completion of the reaction, all solvents were removed under reduced pressure. The resultant crude mixture was analysed by ^{1}H NMR to demonstrate a 95:5 *dr* of the desired product. The crude was subjected to flash chromatography (3:2 Pet/EtOAc + 1% NEt_3) to yield the major (*P*, *S*) diastereomer as a yellow amorphous solid (108 mg, 88%). $[\alpha]_D^{25}$ = + 527 (*c* 0.8, DCM); υ_{max} (film)/cm^{-1}: 2968, 2148, 2025; ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 7.94 (d, 1H, *J* = 5.7 Hz), 7.82 (d, 1H, *J* = 2.2 Hz), 7.61 (d, 1H, *J* = 8.9 Hz), 7.54–7.42 (m, 3H), 7.39 (s, 1H), 7.32–7.28 (m, 3H), 7.25 (d, 1H, *J* = 8.9 Hz), 7.01 (dd, 1H, *J* = 8.9, 2.2 Hz), 6.62 (d, 1H, *J* = 5.7 Hz), 5.09 (q, 1H, *J* = 6.6 Hz), 4.39 (d, 1H, *J* = 14.1 Hz), 4.09 (d, 1H, *J* = 14.1 Hz), 3.05 (s, 3H), 2.41 (q, 2H, *J* = 7.6 Hz), 1.05–1.00 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ_C 153.6, 153.1, 148.7, 148.4, 140.1, 138.7, 137.5, 137.2, 136.2, 131.8, 130.6, 129.5, 129.3, 129.0, 128.7, 128.6, 127.9, 127.7, 127.3, 122.4, 121.2, 120.4, 118.8, 117.5, 107.4, 73.8, 52.8, 38.4, 23.2, 18.4, 14.9; (ESI HRMS) *m/z* calcd. for $C_{33}H_{28}BrN_2O$ 547.1385 $(M+H)^+$, found 547.1368.

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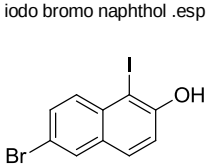

6

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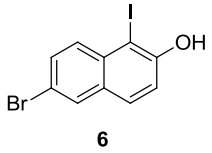

6

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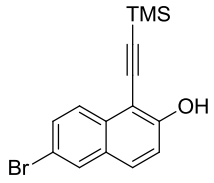
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	203.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3080.0195	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	25.000

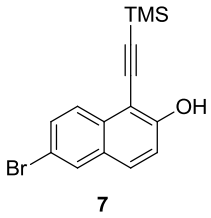
6BrTMS naphthol.esp



7

Acquisition Time (sec)	1.1010	***Comment***	5 mm PABBO BB/19F-1H/D Z-GRD Z800701/0088			***Date***	20 Dec 2010 09:36:00
Date Stamp	20 Dec 2010 09:36:00			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\olddata\04-01-2011\data\mrc\nmr\mrc 573\2\PDATA\1\1r		
Frequency (MHz)	125.76	***Nucleus***	13C	***Number of Transients***	300	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	362.00	***SW(cyclical) (Hz)***	29761.90	***Solvent***	CHLOROFORM-d	***Spectrum Offset (Hz)***	12618.2520
Spectrum Type	STANDARD	***Sweep Width (Hz)***	29761.00	***Temperature (degree C)***	25.000		

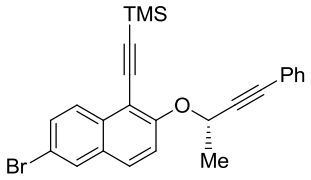
6BrTMS naphthol carbon.esp



7

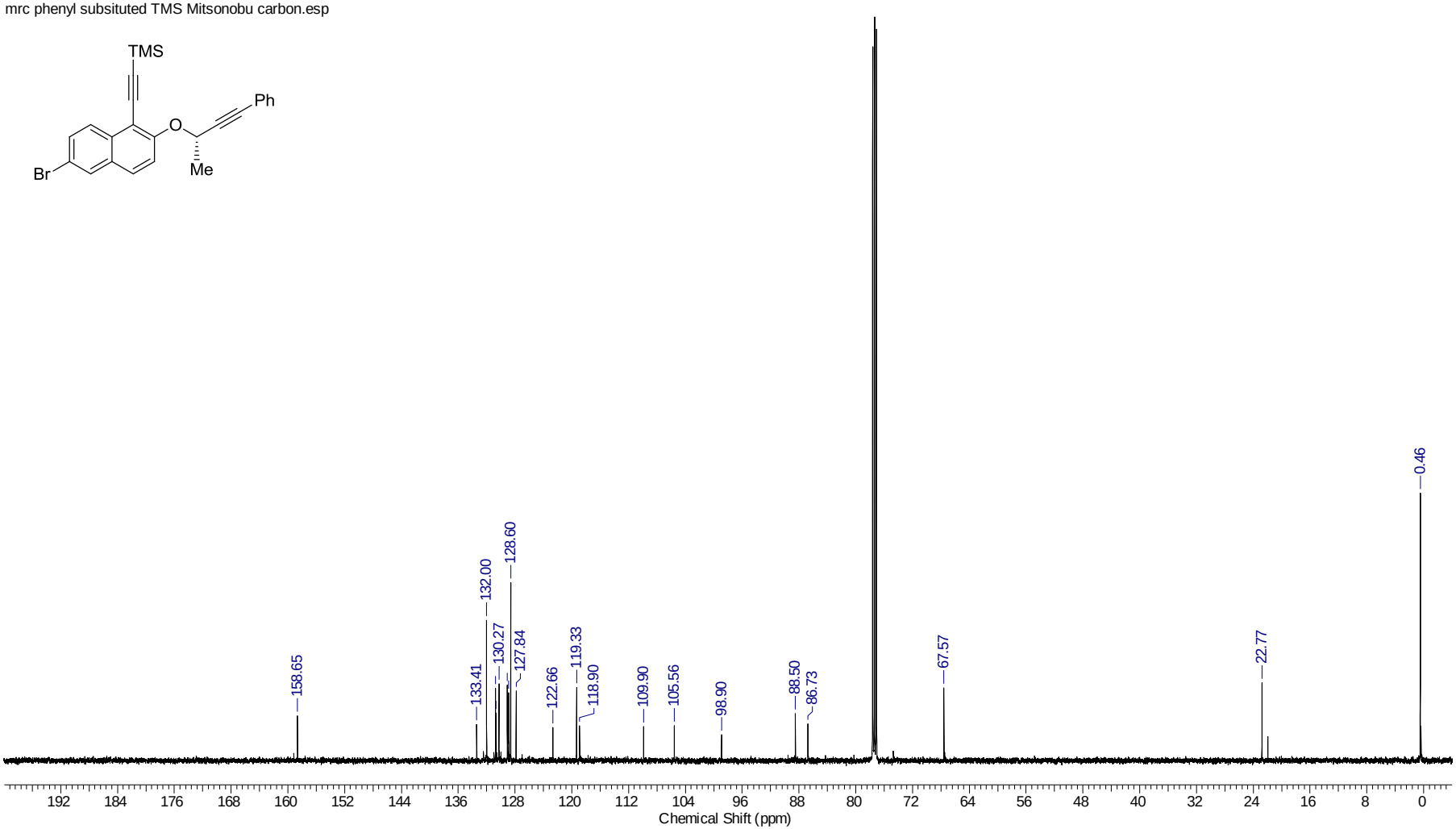
Acquisition Time (sec)	3.1719	***Comment***	Mitsonoubu phenyl protected prop alcohol			***Date***	13 Jun 2011 13:09:36
Date Stamp	13 Jun 2011 13:09:36			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc698\1\PDATA\1\1r		
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	203.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3064.8870	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	25.000

mrc phenyl subsituted TMS Mitsonobu.esp



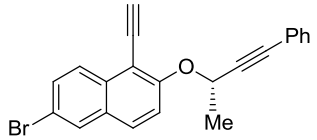
Acquisition Time (sec)	1.1010	***Comment***	5 mm PABBO BB/19F-1H/D Z-GRD Z800701/0088			***Date***	13 Jun 2011 13:48:00
Date Stamp	13 Jun 2011 13:48:00			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc698\2\PDATA\1\1r		
Frequency (MHz)	125.76	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	575.00	***SW(cyclical) (Hz)***	29761.90	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12618.2559	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	29761.00	***Temperature (degree C)***	25.000

mrc phenyl subsituted TMS Mitsonobu carbon.esp



Acquisition Time (sec)	3.1719	***Comment***	deprotected mitsonobu product			***Date***	17 Jun 2011 15:09:04
Date Stamp	17 Jun 2011 15:09:04			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc704\1\PDATA\1\1r		
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	181.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3064.5720	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	25.000

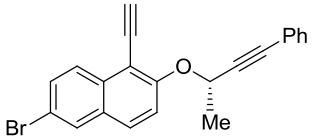
mrcTMS deprotected chiral acetylene .esp



***S*-9**

Acquisition Time (sec)	1.1010	***Comment***	5 mm PABBO BB/19F-1H/D Z-GRD Z800701/0088			***Date***	17 Jun 2011 15:43:12
Date Stamp	17 Jun 2011 15:43:12			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc704\2\PDATA\1\1r		
Frequency (MHz)	125.76	***Nucleus***	13C	***Number of Transients***	300	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	362.00	***SW(cyclical) (Hz)***	29761.90	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12616.4395	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	29761.00	***Temperature (degree C)***	25.000

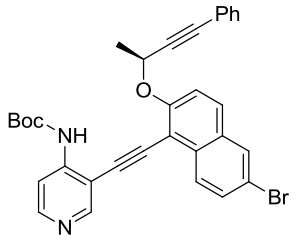
mrcTMS deprotected chiral acetylene carbon.esp



S-**9**

Acquisition Time (sec)	3.1719	***Comment***	purified first	***Date***	29 Jun 2011 12:07:44				
Date Stamp	29 Jun 2011 12:07:44			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc711\1\PDATA\1\1r				
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect	***Original Points Count***	32768
Owner	chemist	***Points Count***	32768	***Pulse Sequence***	zg30	***Receiver Gain***	36.00	***SW(cyclical) (Hz)***	10330.58
Solvent	CHLOROFORM-d			***Spectrum Offset (Hz)***	3085.6516	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26
Temperature (degree C)	25.000								

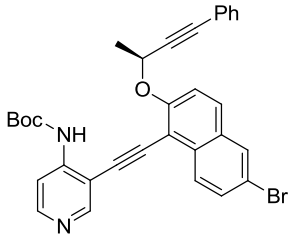
mrc 711 chiral prydine diyne .esp



S-**11**

Acquisition Time (sec)	1.1010	***Comment***	5 mm PABBO BB/19F-1H/D Z-GRD Z800701/0088			***Date***	29 Jun 2011 12:29:04
Date Stamp	29 Jun 2011 12:29:04			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc711\2\PDATA\1\1r		
Frequency (MHz)	125.76	***Nucleus***	13C	***Number of Transients***	256	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	1150.00	***SW(cyclical) (Hz)***	29761.90	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12599.1826	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	29761.00	***Temperature (degree C)***	25.000

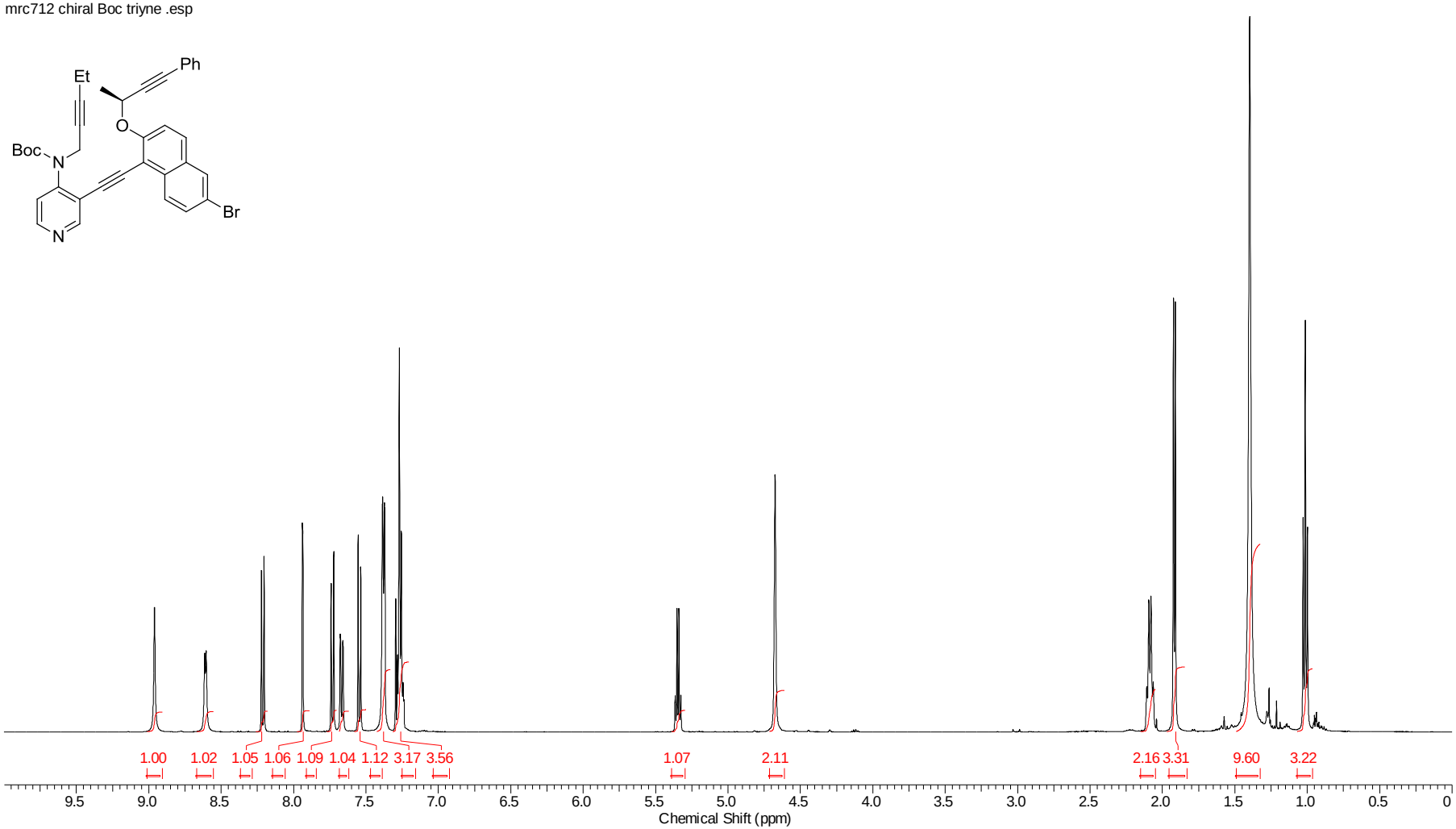
mrc 711 chiral prydine diyne carbon.esp



S-**11**

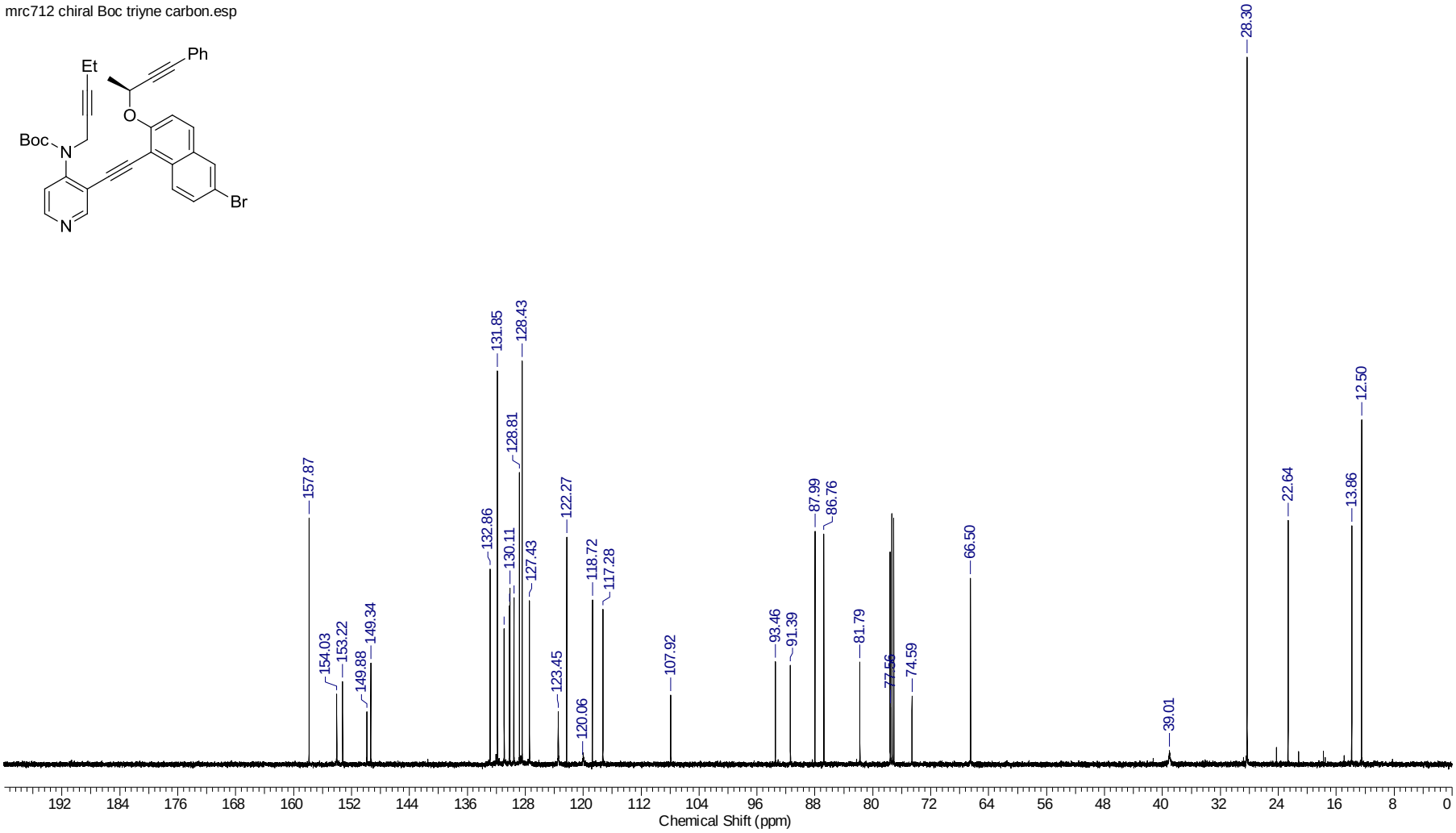
Acquisition Time (sec)	3.1719	***Comment***	chiral Boc triyne columned			***Date***	01 Jul 2011 12:12:00
Date Stamp	01 Jul 2011 12:12:00			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc712\1\PDATA\1\1r		
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	25.40	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3097.9468	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	25.000

mrc712 chiral Boc triyne .esp



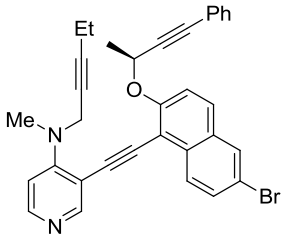
Acquisition Time (sec)	1.1010	***Comment***	5 mm PABBO BB/19F-1H/D Z-GRD Z800701/0088			***Date***	01 Jul 2011 12:37:36
Date Stamp	01 Jul 2011 12:37:36			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc712\2\PDATA\1\1r		
Frequency (MHz)	125.76	***Nucleus***	13C	***Number of Transients***	300	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	812.00	***SW(cyclical) (Hz)***	29761.90	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12595.5488	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	29761.00	***Temperature (degree C)***	25.000

mrc712 chiral Boc triyne carbon.esp



Acquisition Time (sec)	3.1719	***Comment***	chiral methyl bromo triyne			***Date***	03 Jul 2011 15:02:40
Date Stamp	03 Jul 2011 15:02:40			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc714\1\PDATA\1\1r		
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zg30
Receiver Gain	64.00	***SW(cyclical) (Hz)***	10330.58	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	3085.6516	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26	***Temperature (degree C)***	25.000

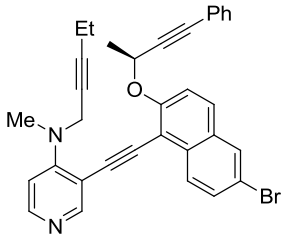
mrc714 methyl pyridine chiral triyne .esp



S-**12**

Acquisition Time (sec)	1.1010	***Comment***	5 mm PABBO BB/19F-1H/D Z-GRD Z800701/0088			***Date***	03 Jul 2011 15:26:08
Date Stamp	03 Jul 2011 15:26:08			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc714\2\PDATA\1\1r		
Frequency (MHz)	125.76	***Nucleus***	13C	***Number of Transients***	300	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	575.00	***SW(cyclical) (Hz)***	29761.90	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12611.8984	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	29761.00	***Temperature (degree C)***	25.000

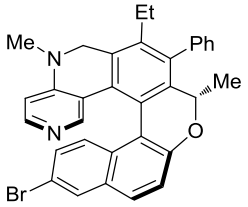
mrc714 methyl pyridine chiral triyne carbon.esp



S-**12**

Acquisition Time (sec)	3.1719	***Comment***	Helical DMAP	***Date***	18 Jul 2011 09:06:24				
Date Stamp	18 Jul 2011 09:06:24			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc721\5\PDATA\1\1r				
Frequency (MHz)	500.13	***Nucleus***	1H	***Number of Transients***	16	***Origin***	spect	***Original Points Count***	32768
Owner	chemist	***Points Count***	32768	***Pulse Sequence***	zg30	***Receiver Gain***	181.00	***SW(cyclical) (Hz)***	10330.58
Solvent	CHLOROFORM-d			***Spectrum Offset (Hz)***	3085.3362	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	10330.26
Temperature (degree C)	25.000								

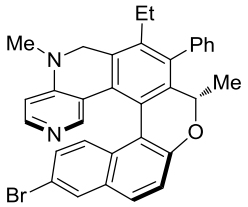
mrc721 HeAP chiral dr reaction.esp



(P,S)-**5**

Acquisition Time (sec)	1.1010	***Comment***	5 mm PABBO BB/19F-1H/D Z-GRD Z800701/0088			***Date***	18 Jul 2011 09:32:00
Date Stamp	18 Jul 2011 09:32:00			***File Name***	\\chpc-nmr500.campus.bath.ac.uk\data\mrc\nmr\mrc721\6\PDATA\1\1r		
Frequency (MHz)	125.76	***Nucleus***	13C	***Number of Transients***	300	***Origin***	spect
Original Points Count	32768	***Owner***	chemist	***Points Count***	32768	***Pulse Sequence***	zgpg30
Receiver Gain	575.00	***SW(cyclical) (Hz)***	29761.90	***Solvent***	CHLOROFORM-d		
Spectrum Offset (Hz)	12616.4395	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	29761.00	***Temperature (degree C)***	24.900

mrc721 HeAP chiral dr reaction carbon.esp



(P,S)-**5**