Supplementary Material (ESI) for Chemical Communications

Supplementary Material

An alkynylboronic ester cycloaddition route to functionalised aromatic boronic esters

Patrick M. Delaney, [a] Jane E. Moore [a] and Joseph P. A. Harrity*[a]

[a]*Department of Chemistry, University of Sheffield, Brook Hill, Sheffield, S3 7HF*

General Procedures

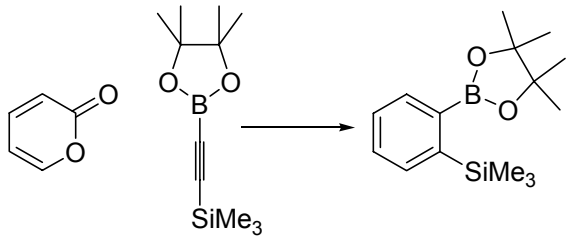
Infrared (IR) Spectra were recorded on a Perkin Elmer Paragon 100 FTIR spectrophotometer, ν_{max} in cm^{-1}. Samples were recorded as thin films using sodium chloride plates, as a DCM solution. Bands are characterised as broad (br), strong (s), medium (m), and weak (w). ^1H NMR spectra were recorded on a Bruker AC-250 (250 MHz) or AMX-400 (400 MHz) supported by an Aspect 3000 data system, unless otherwise stated. Chemical shifts are reported in ppm from tetramethylsilane with the residual protic solvent resonance as the internal standard ($CHCl_3$: δ7.27ppm). Data are reported as follows: chemical shift, integration, multiplicity (s=singlet, d=doublet, q=quartet, pent=pentet, sext=sextet, br=broad, m=multiplet, app=apparent), coupling constants (Hz), and assignment. ^{13}C NMR spectra were recorded on a Bruker AC-250 (62.9 MHz) or AMX-400 (100.6 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal reference ($CDCl_3$: δ77.0ppm). Low resolution mass spectra were recorded on Micromass Autospec, operating in E.I., C.I. or FAB mode; or a Perkin-Elmer Turbomass Benchtop GC-MS operating in either E.I. or C.I mode. High-resolution mass spectra (HRMS) recorded for accurate mass analysis, were performed on either a MicroMass LCT operating in Electrospray mode (TOF ES^+) or a MicroMass Prospec operating in FAB (FAB^+), EI (EI^+) or CI (CI^+) mode. Elemental microanalysis was performed using a Perkin-Elmer 2400 CHNS / O Series II Elemental Analyser. Melting points performed on recrystallised solids, were recorded on a Gallenkamp melting point apparatus and are uncorrected. All solvents and reagents were purified using standard laboratory techniques according to methods published in "Purification of Laboratory Chemicals" by Perrin, Armarego, and Perrin (Pergamon Press, 1966). Starting alkynylboronates[1] and pyranones[2] were prepared according to established procedures. Methyl coumalate was purchased from Aldrich chemical co. and used as received. Flash chromatography was performed on silica gel (BDH Silica Gel 60 43-60). Thin layer chromatography (TLC) was performed on aluminium backed plates pre-coated with

[1] H. C. Brown, N. G. Bhat , M. Srebink, *Tetrahedron Lett*, 1982, **29**, 2631.
[2] A. Haneda, H. Uenakai, T. Imawaga, M. Kawanisi, *Synth. Comm*, 1976, **6**, 141; I.W. Ashworth, M.C. Bowden, B. Dembofsky,D. Levin, W. Moss, E. Robinson, N. Szczur, J. Virica, *Org. Proc. Res. Dev.*, 2003, **7**, 74; C.G. Cho, Y.W. Kim, Y.K. Lim, J.S. Park, H. Lee, S. Koo, *J. Org. Chem*, 2002, **67**, 290.

silica (0.2 mm, Merck DC-alufolien Kieselgel 60 F_{254}) which were developed using standard visualizing agents: Ultraviolet light or potassium permanganate. X-Ray data for compounds **13** and **15** have been deposited with the CCDC, supplementary entry numbers: 608269 and 608270 respectively.

1.1. [4+2] Cycloaddition

Cycloaddition of 1 with 2a

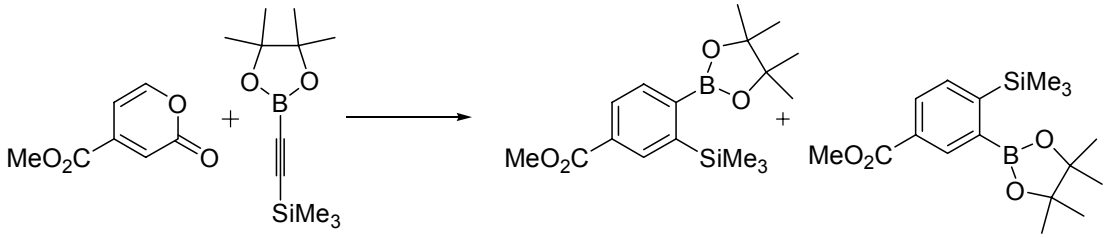


1 **2a** **3**

A mixture of **1** (0.1 g, 1.04 mmol) and **2a** (0.466 g, 2.08 mmol) was heated at 170 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compound **3** as crystalline colourless solid, wt. 0.247 g, 86% yield.

Mp 84-86 ^{o}C **^{1}H NMR** (250 MHz, $CDCl_3$): δ0.33 (9H, s, $SiMe_3$), 1.34 (12H, s, 4 x CH_3), 7.36 (2H, m, Ar-H) 7.61 (1H, m, Ar-*H*), 7.90 (1H, m, Ar-*H*); **^{13}C NMR** (62.9 MHz, $CDCl_3$) δ 1.1, 25.0, 83.8, 127.8, 129.7, 134.3, 136.1, 146.0; **FTIR**: ν_{max}/$CHCl_3$, 2981 (s) cm^{-1}; **HRMS (**EI^+) calcd for. $C_{15}H_{25}BO_2Si$: 276.1717. Found: 276.1715.

Cycloaddition of 4 with 2a



4 **2a** **6a** **6b**

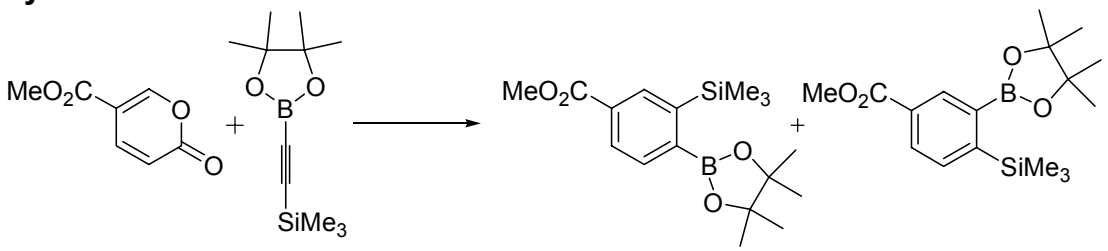
A mixture of **4** (0.1 g, 0.649 mmol) and **2a** (0.291 g, 1.298 mmol) was heated at 170 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give **6a** and **6b** (3:1 ratio) as colourless

oils, wt. 0.152 g, 70% yield. *The regiochemistry was determined by 2-D NMR experiment (nOesy) of the major regioisomer* **6a**.

6a : ^{1}H NMR (250 MHz, $CDCl_3$): δ0.29 (9H, s, $SiMe_3$), 1.29 (12H, s, CH_3), 3.86 (3H, s, CO_2CH_3), 7.62 (1H, d, *J*=8.0 Hz, Ar-*H*), 7.93 (1H, dd, *J*=8.0, 1.5 Hz, Ar-*H*), 8.43 (1H, d, *J*=1.5 Hz, Ar-*H*); **^{13}C NMR** (62.9 MHz, $CDCl_3$) δ0.8, 25.0, 52.0, 84.1, 129.7, 130.2, 134.4, 136.4, 153.3, 167.4; **FTIR:** $\nu_{max}/CHCl_3$, 2979 (m), 2951 (w), 2901 (w), 1726 (s), 1593 (w), 1551 (w) cm^{-1}; **HRMS** (EI^+) calcd. for $C_{17}H_{27}O_4BSi$ 335.1850 Found: 335.1857

6b : **^{1}H NMR** (250 MHz, $CDCl_3$) δ0.36 (9H, s, $SiMe_3$), 1.35 (12H, s, CH_3), 3.91 (3H, s, CO_2CH_3), 7.96 (2H, m, Ar-*H*), 8.24 (1H, d, *J*=1.5 Hz, Ar-*H*); ^{13}C NMR (62.9 MHz, $CDCl_3$) δ0.4, 25.0, 52.1, 84.2, 128.5, 130.5, 134.8, 135.9, 147.3, 167.5; FTIR $\nu_{max}/CHCl_3$ 2979 (m), 2952 (w), 1726 (s), 1595 (w), 1548 (w) cm^{-1}; HRMS (EI^+) calcd. for $C_{17}H_{27}O_4BSi$ 335.1850. Found: 335.1839

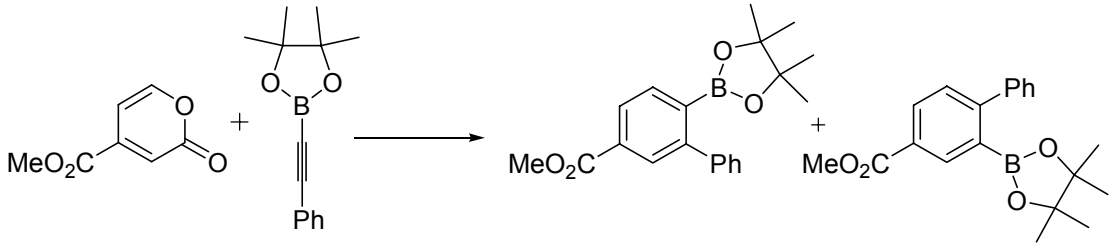
Cycloaddition of 5 with 2a



5 **2a** **6a** **6b**

A mixture of **5** (0.2 g, 1.298 mmol) and **2a** (0.582 g, 2.595 mmol) was heated at 170 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give the separated title compounds **6a** and **6b** (1:1 ratio) as colourless oils, wt. 0.384 g, 83% yield.

Cycloaddition of 4 with 2b



4 **2a** **7a** **7b**

A mixture of **4** (0.1 g, 0.649 mmol) and **2a** (0.296 g, 1.298 mmol) was heated at 170 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting

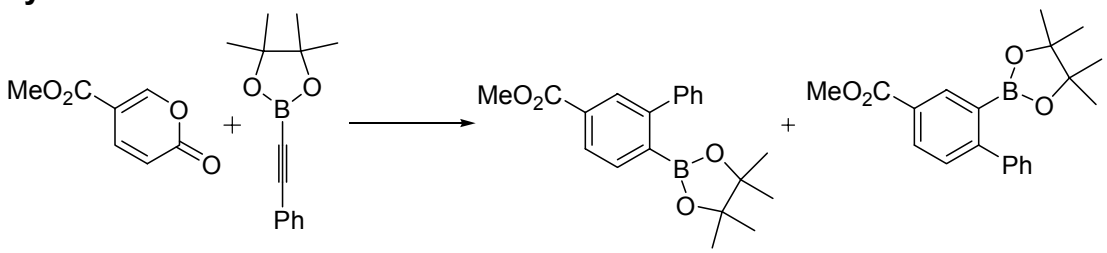
solvent petroleum ether/ethyl acetate 25:1 ratio) to give **7a** and **7b** (1:1 ratio) as a clear oil, 0.092g, 42%.

7a ^{1}**H NMR**: δ1.23 (12H, s, 4 x CH_3), 3.95 (3H, s, CO_2CH_3), 7.35-7.48 (6H, m, Ar-*H*), 8.12 (1H, dd, *J*=8.0, 2.0 Hz, Ar-*H*) 8.38 (1H, dd, *J*=2.0, 0.5 Hz, Ar-*H*); ^{13}**C NMR** (62.9 MHz, $CDCl_3$) δ24.6, 52.1, 84.0, 127.0, 127.5, 127.9, 128.9, 129.0, 130.0, 135.6, 142.1, 151.9, 167.0.

7b ^{1}**H NMR**: δ1.21 (12H, s, 4 x CH_3), 3.92 (3H, s, CO_2CH_3), 7.35-7.48 (5H, m, Ar-*H*), 7.56 (1H, d, *J*=8.0 Hz, Ar-*H*), 7.98 (1H, dd, *J*=8.0, 1.5 Hz, Ar-*H*), 8.04 (1H, dd, *J*= 1.0, 0.5 Hz, Ar-*H*); ^{13}**C NMR** (62.9 MHz, $CDCl_3$) δ24.7, 52.2, 84.1, 127.0, 127.2, 127.5, 128.0, 129.0, 129.1, 135.7, 142.0, 147.9, 167.0.

FTIR: ν_{max}/$CHCl_3$, 2991 (w), 2979 (w), 2940 (w), 1723 (s), 1600 (w) cm^{-1}; **HRMS** (EI^+) calcd. for $C_{20}H_{23}O_4B$: 338.1689 Found: 338.1687.

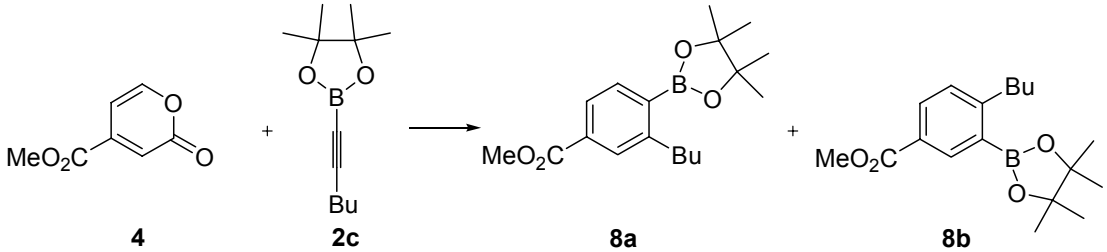
Cycloaddition of 5 with 2b



5 **2b** **7a** **7b**

A mixture of **5** (0.1 g, 0.649 mmol) and **2b** (0.296 g, 1.298 mmol) was heated at 170 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compounds **7a** and **7b** (1:14 ratio) as a clear oil, 0.127g, 57% yield. *The regiochemistry was determined by 2-D NMR experiment (nOesy) of the major regioisomer* **7b**.

Cycloaddition of 4 with 2c



4 **2c** **8a** **8b**

A mixture of **4** (0.1 g, 0.649 mmol) and **2c** (0.270 g, 1.298 mmol) was heated at 170 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compounds **8a** and **8b** (10:1 ratio) as

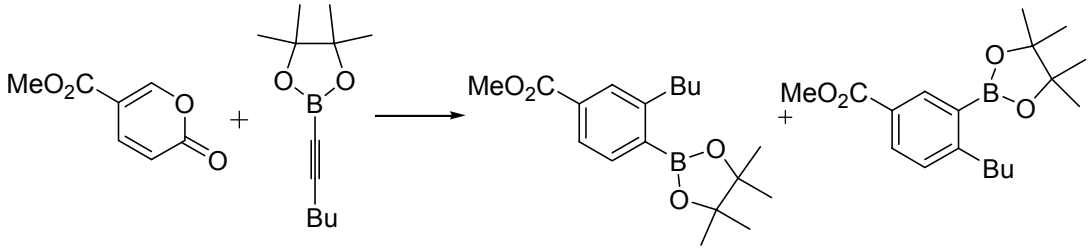
a clear oil, wt. 0.050 g, 24% yield. *The regiochemistry was determined by 2-D NMR experiment (nOesy) of the major regioisomer* **8a**.

8a ^{1}H NMR: δ0.85 (3H, t, *J*=7.5 Hz, CH_2CH_3), 1.28 (12H, s, 4 x CH_3), 1.22-1.37 (2H, m, CH_2CH_3), 1.38-1.56 (2H, m, $CH_2CH_2CH_3$), 2.85 (2H, app t, *J*=8.0 Hz, C=CCH_2), 3.88 (3H, s, CO_2CH_3), 7.16 (1H, d, *J*=8.0 Hz, Ar-*H*), 7.92 (1H, dd, *J*=8.0, 2.0 Hz, Ar-*H*), 8.34 (1H, d, *J*=2.0 Hz, Ar-*H*); **^{13}C NMR** (62.9 MHz, $CDCl_3$) δ13.9, 22.7, 24.8, 35.3, 35.6, 51.9, 83.7, 126.8, 129.3, 131.8, 137.2, 155.5, 167.3.

8b ^{1}H NMR: δ0.85 (3H, t, *J*=7.5 Hz, CH_2CH_3), 1.28 (12H, s, 4 x CH_3), 1.22-1.37 (2H, m, CH_2CH_3), 1.38-1.56 (2H, m, $CH_2CH_2CH_3$), 2.85 (2H, app t, *J*=8.0 Hz, C=CCH_2), 3.89 (3H, s, CO_2CH_3), 7.79 (2H, s, Ar-*H*), 7.81 (1H, s, Ar-*H*); **^{13}C NMR** (62.9 MHz, $CDCl_3$) δ13.9, 22.7, 24.8, 35.2, 35.4, 52.0, 83.8, 125.6, 129.9, 131.8, 135.8, 150.2, 167.3.

FTIR: ν_{max}/$CHCl_3$,2977 (m), 2956 (m), 2931 (m), 2870 (m), 1723 (s)cm^{-1} **HRMS** (EI^+) calcd. for $C_{18}H_{27}O_4B$: 318.2002 Found: 318.2012

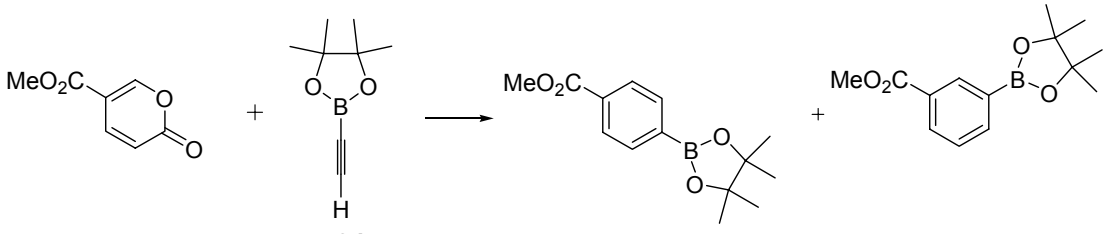
Cycloaddition of 5 with 2c



5 **2c** **8a** **8b**

A mixture of **5** (0.1 g, 0.649 mmol) and **2c** (0.270 g, 1.298 mmol) was heated at 170 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compounds **8a** and **8b** (1: 3 ratio) as a clear oil, wt. 0.127 g, 59% yield. *The regiochemistry was determined by 2-D NMR experiment (nOesy) of the major regioisomer* **8b**.

Cycloaddition of 5 with 2d



5 **2d** **9a** **9b**

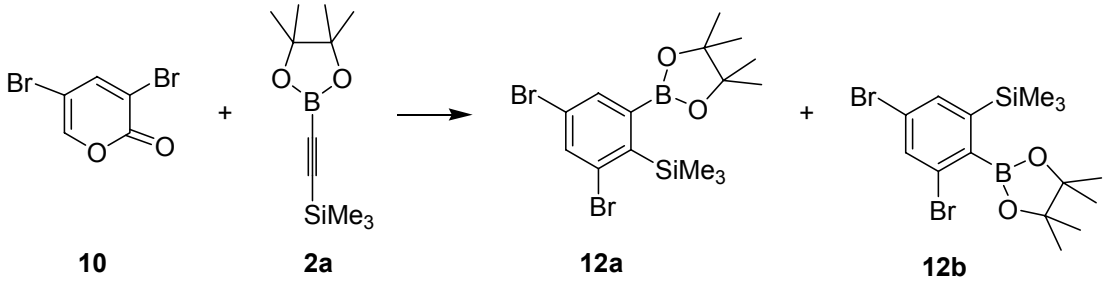
A mixture of **5** (0.1 g, 0.649 mmol), **2d** (0.09 g, 0.649 mmol) and diphenylether (1 mL) were heated at 170 °C with stirring, for 15 hours under a nitrogen atmosphere. The product was purified by flash column chromatography (eluting solvent 100: 1 petroleum ether: EtOAc) to give colourless crystalline solid of regioisomers in 5: 1 ratio (Major regioisomer was **9a**) wt. 0.130 g, 77% yield.

9a[3]: **^{1}H NMR** (250 MHz, $CDCl_3$): δ1.28 (12H, s, C*H*$_3$), 3.84 (3H, s, C*H*$_3$OCO), 7.79 (2H, d, *J*=8.5 Hz, Ar*H*), 7.96 (2H, d, *J*=8.5 Hz, Ar*H*). **^{13}C NMR** (62.9 MHz, $CDCl_3$): δ24.8, 52.1, 84.1, 127.8, 135.8, 134.6, 167.1

9b: **^{1}H NMR** δ1.28 (12H, s, C*H*$_3$), 3.84 (3H, s, C*H*$_3$OCO), 7.38 (1H, t, *J*=8.0 Hz, Ar*H*), 7.90 (1H, m, Ar*H*), 8.05 (1H, dt, *J*=8.0, 1.0 Hz, Ar*H*), 8.39 (1H, br, Ar*H*). **^{13}C NMR** (62.9 MHz, $CDCl_3$): δ24.8, 52.0, 84.1, 128.6 (x 2), 132.3 (x 2), 139.1, 167.1.

FTIR: $\nu_{max}/CHCl_3$, cm^{-1} 2979 (w), 1727 (s), 1606 (w), 1510 (w), 1399 (m), 1361 (s). **HRMS** (EI$^+$) Calcd for $C_{29}H_{24}O_4$ 262.1376 Found: 262.1377.

Cycloaddition of 10 with 2a



10 **2a** **12a** **12b**

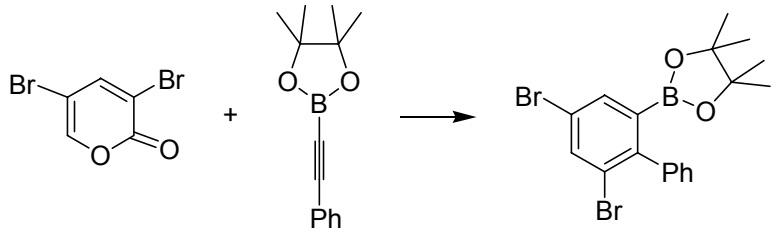
A mixture of **10** (0.100 g, 0.394 mmol) and **2a** (0.176 g, 0.788 mmol) and mesitylene (1 mL) was heated at 155 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compounds **12a** and **12b** (1:1 ratio) as a brown oil, wt. 0.111 g, 65% yield.

12a/**b** mixture:**^{1}H NMR** δ0.29 (9H, s, C*H*$_3$), 0.37 (9H, s, C*H*$_3$),1.29 (12H, s, C*H*$_3$), 1.37 (12H, s, C*H*$_3$),7.47 (1H, d, *J*=2.0 Hz, Ar*H*), 7.51 (1H, d, *J*=2.0 Hz, Ar*H*), 7.59 (1H, d, *J*=2.0 Hz, Ar*H*), 7.64 (1H, d, *J*=2.0 Hz, Ar*H*).**^{13}C NMR** (62.9 MHz, $CDCl_3$): δ-0.5, 1.8, 24.7, 25.3, 84.5, 85.5, 112.5(x2), 134.3(x2), 134.8(x2), 135.0(x2), 136.3(x2).

FTIR: $\nu_{max}/CHCl_3$, cm^{-1} 2980 (s), 1315 (s), 846 (s). **HRMS** (EI$^+$) Calcd for $C_{15}H_{23}BBr_2O_2Si$ 431.9927 Found: 431.9909.

[3] N. Miyaura, M. Murata, I. Tatsuo, *J. Org. Chem.*, 1995, **60**, 7508.

Cycloaddition of 10 with 2b

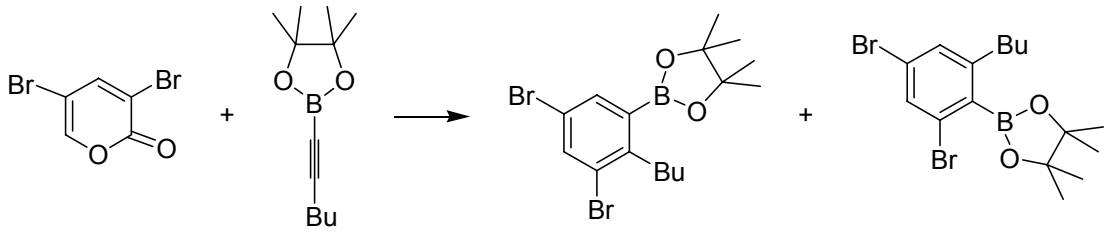


10 **2b** **13**

A mixture of **10** (0.200 g, 0.788 mmol) and **2b** (0.269 g, 1.182 mmol) and mesitylene (1 mL) was heated at 155 °C and stirred for 15 h under N_2. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compound **13** as brown solid, 0.214g, 62% yield. *The regiochemistry was determined by X-ray crystallography.*

Mp 54-56 °C. **^{1}H NMR** δ1.06 (12H, s, C*H*$_3$), 7.28 (2H, m, Ar*H*), 7.36 (3H, m, Ar*H*), 7.72 (1H, d, *J*=2.0 Hz, Ar*H*), 7.85 (1H, d, *J*=2.0 Hz, Ar*H*). **^{13}C NMR** (62.9 MHz, $CDCl_3$): δ24.4, 84.2, 113.3, 114.0, 115.0, 117.5, 122.0, 127.6, 129.5, 135.4, 136.3. **FTIR**: $\nu_{max}/CHCl_3$, cm^{-1} 2979 (w), 2979(s), 1458 (w), 1145 (w). **HRMS** (EI$^+$) Calcd for $C_{18}H_{19}BBr_2O_2$ 381.0490 Found: 381.0486.

Cycloaddition of 10 with 2c



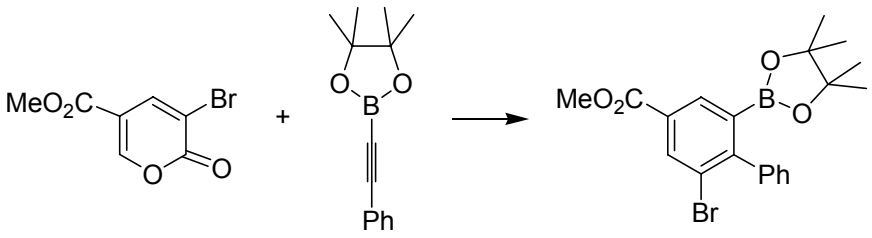
10 **2c** **14a** **14b**

A mixture of **10** (0.100 g, 0.394 mmol) and **2c** (0.123 g, 0.591 mmol) and mesitylene (1 mL) was heated at 155 °C and stirred for 15 h under N_2.The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compounds **14a** and **14b** as a brown oil, wt. 0.077 g,47% yield. *The regiochemistry was determined by NMR experiment (nOe) of the major regioisomer* **14a**.

14a: **^{1}H NMR** δ0.87 (3H, t, CH_2CH_3), 1.26 (12H, s, C*H*$_3$), 1.27-1.36 (4H, m, $CH_2CH_2CH_3$), 2.92 (2H, t, *J*=8.0 Hz, C=CCH_2), 7.66 (1H, d, *J*=1.0 Hz, Ar*H*), 7.73 (1H, d, *J*=1.0 Hz Ar*H*). **^{13}C NMR** (62.9 MHz, $CDCl_3$): δ13.9, 22.9, 24.8, 34.4, 35.4, 81.5, 125.6, 130.4, 134.9, 137.3, 137.7.

FTIR: $\nu_{max}/CHCl_3$, cm^{-1} 2958 (s). **HRMS** (EI^+) Calcd for $C_{16}H_{23}BBr_2O_2$ 416.0158 Found: 416.0162.

Cycloaddition of 11 with 2b

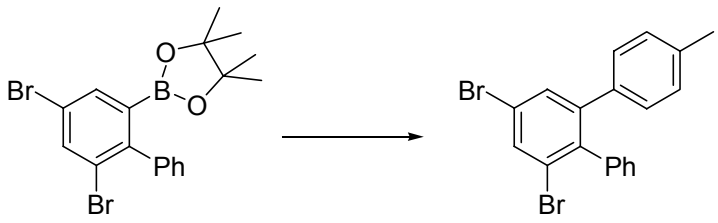


11 **2b** **15**

A mixture of **11** (0.1 g, 0.429 mmol) and **2b** (0.391 g, 1.716 mmol) was heated at 170 °C and stirred for 15 h under vacuum 10 mmHg. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 25:1 ratio) to give compound **89** as brown solid, 0.099 g, 56% yield. *The regiochemistry was determined by X-ray crystallography.*

Mp 109-111 °C.**^{1}H NMR** δ1.01 (12H, s, C*H*$_3$), 3.85 (3H, s, C*H*$_3$OCO), 7.30 (2H, m, Ar*H*), 7.71 (3H, m, Ar*H*), 8.17 (1H, d, *J*= 2.0 Hz, Ar*H*), 8.28 (1H, d, *J*= 2.0 Hz Ar*H*). **^{13}C NMR** (62.9 MHz, $CDCl_3$): δ24.9, 52.6, 82.1, 122.9, 128.5, 128.8, 129.5, 130.9, 131.6, 134.7, 140.6, 147.4, 166.1. **FTIR**: $\nu_{max}/CHCl_3$, cm^{-1} 2978 (w), 1727 (s). **HRMS** (EI^+) Calcd for $C_{20}H_{22}BBrO_4$ 417.0873 Found: 417.0886.

Suzuki reaction of 13 with *p*-iodotoluene



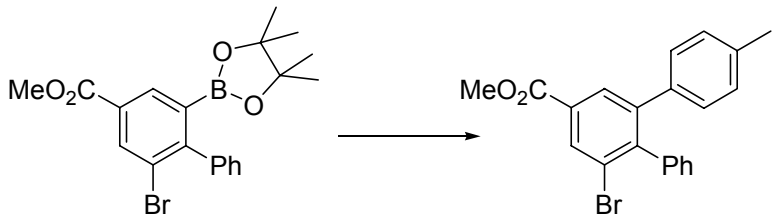
13 **16**

A round bottom flask was charged with **13** (0.260 g, 0.594 mmol), $PdCl_2$(dppf)DCM (0.043 g, 0.059 mmol), K_3PO_4 (0.378 g, 1.780 mmol), dioxane (1 ml) and iodotoluene (0.259 g, 1.187 mmol). The flask was fitted with a reflux condenser and heated at 85 °C under N_2. After stirring for 48 h the reaction was cooled to room temperature and quenched by the addition of distilled water (10 ml), the product was extracted into dichloromethane (3 x 10 ml), dried ($MgSO_4$), filtered and conc. *in vacuo*. The product was purified by flash column

chromatography (eluting solvent petroleum ether/ethyl acetate 15:1 ratio) to give compound **16** as clear oil, wt. 0.133 g, 56% yield.

^{1}H NMR δ2.24 (3H, s, C*H*$_3$), 6.86-6.95 (4H, m, Ar*H*), 7.24-7.20 (2H, m, Ar*H*), 7.49-7.50 (3H, m , Ar*H*), 7.49 (1H, d, *J*=2.0 Hz, Ar*H*), 7.81 (1H, d, *J*=2.0, Hz, Ar*H*). **^{13}C NMR** (62.9 MHz, $CDCl_3$): δ21.3, 113.1, 113.9, 114.2, 115.1, 126.5, 127.3, 127.7, 128.5, 129.3, 130.1, 130.4, 133.8, 134.3, 135.0. **FTIR**: $\nu_{max}/CHCl_3$, cm^{-1} 2923 (w). **HRMS** (EI$^+$) Calcd for $C_{19}H_{14}Br_2$ 399.9462 Found: 399.9462.

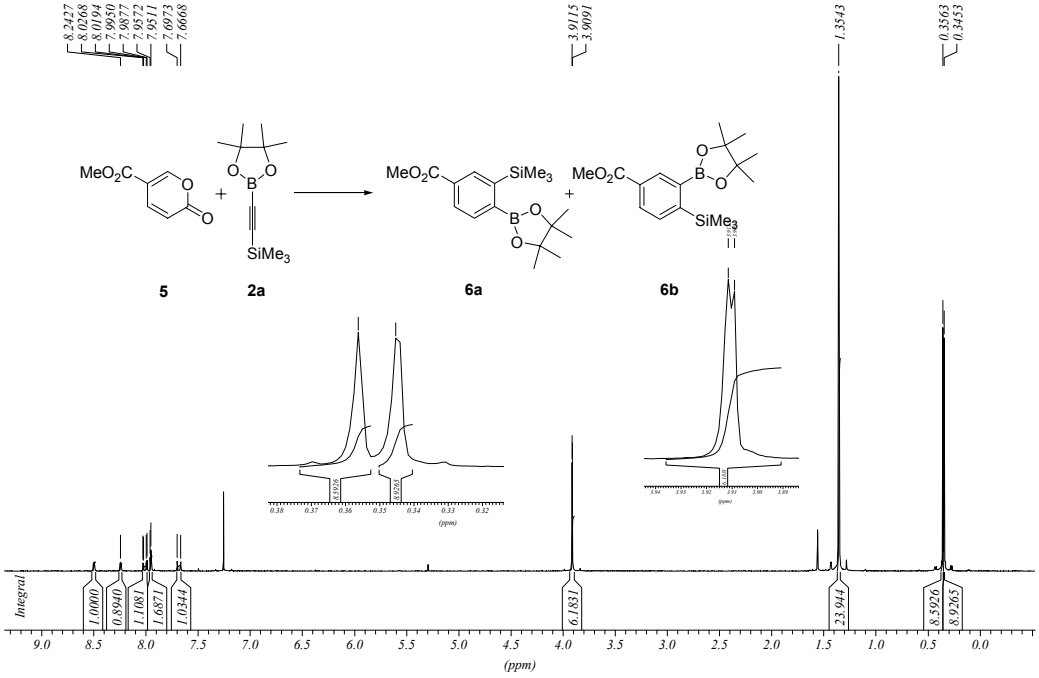
Suzuki reaction of 13 with *p*-iodotoluene

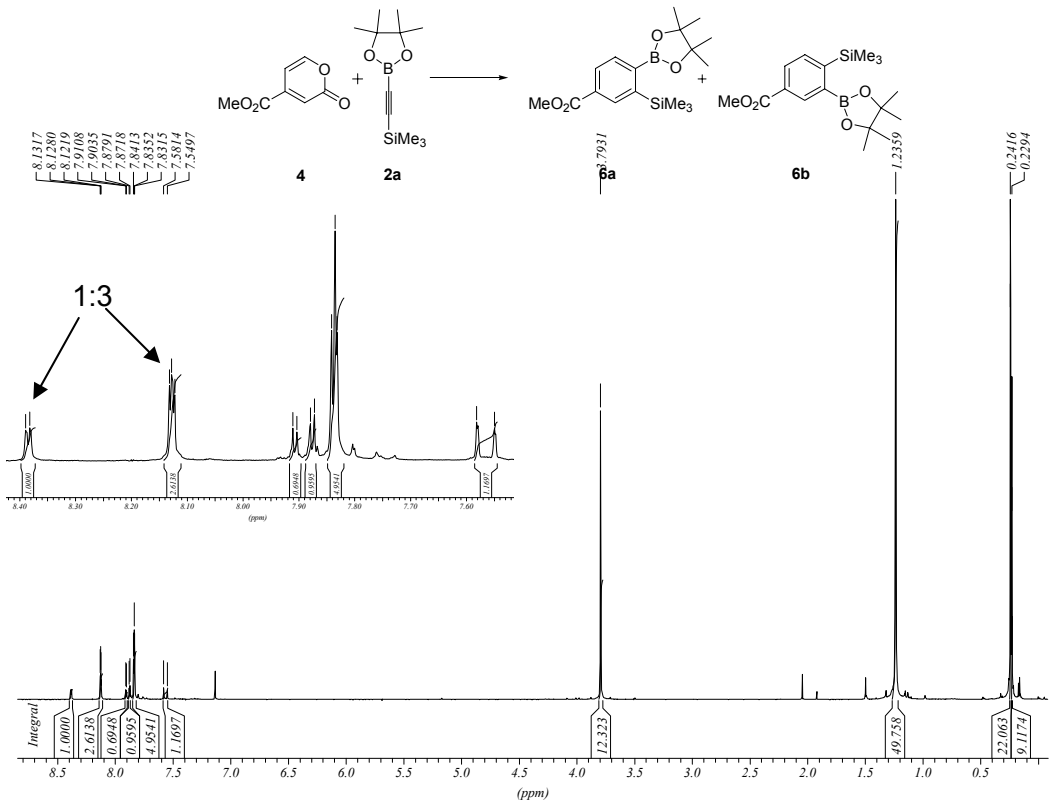


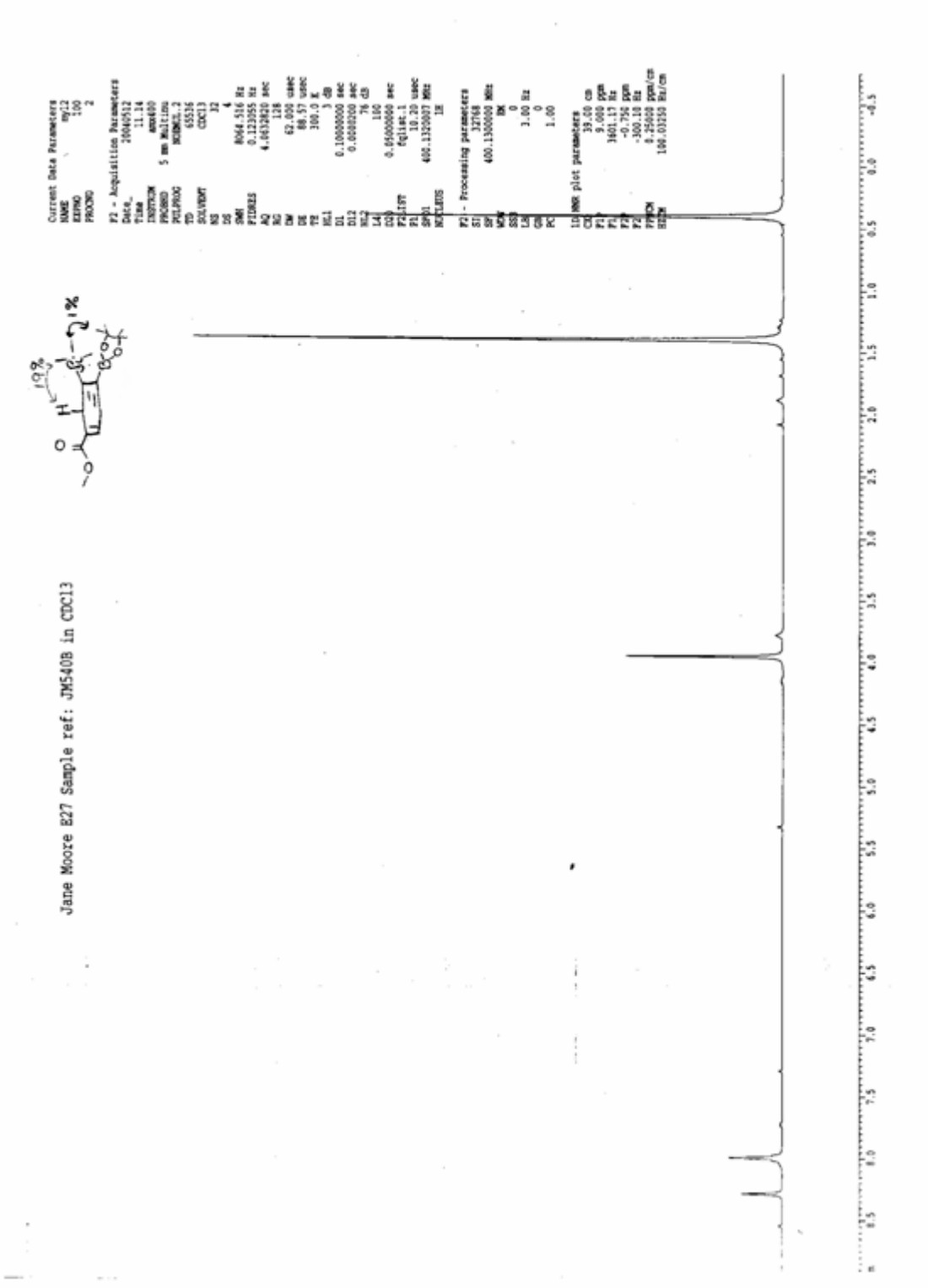
14 **17**

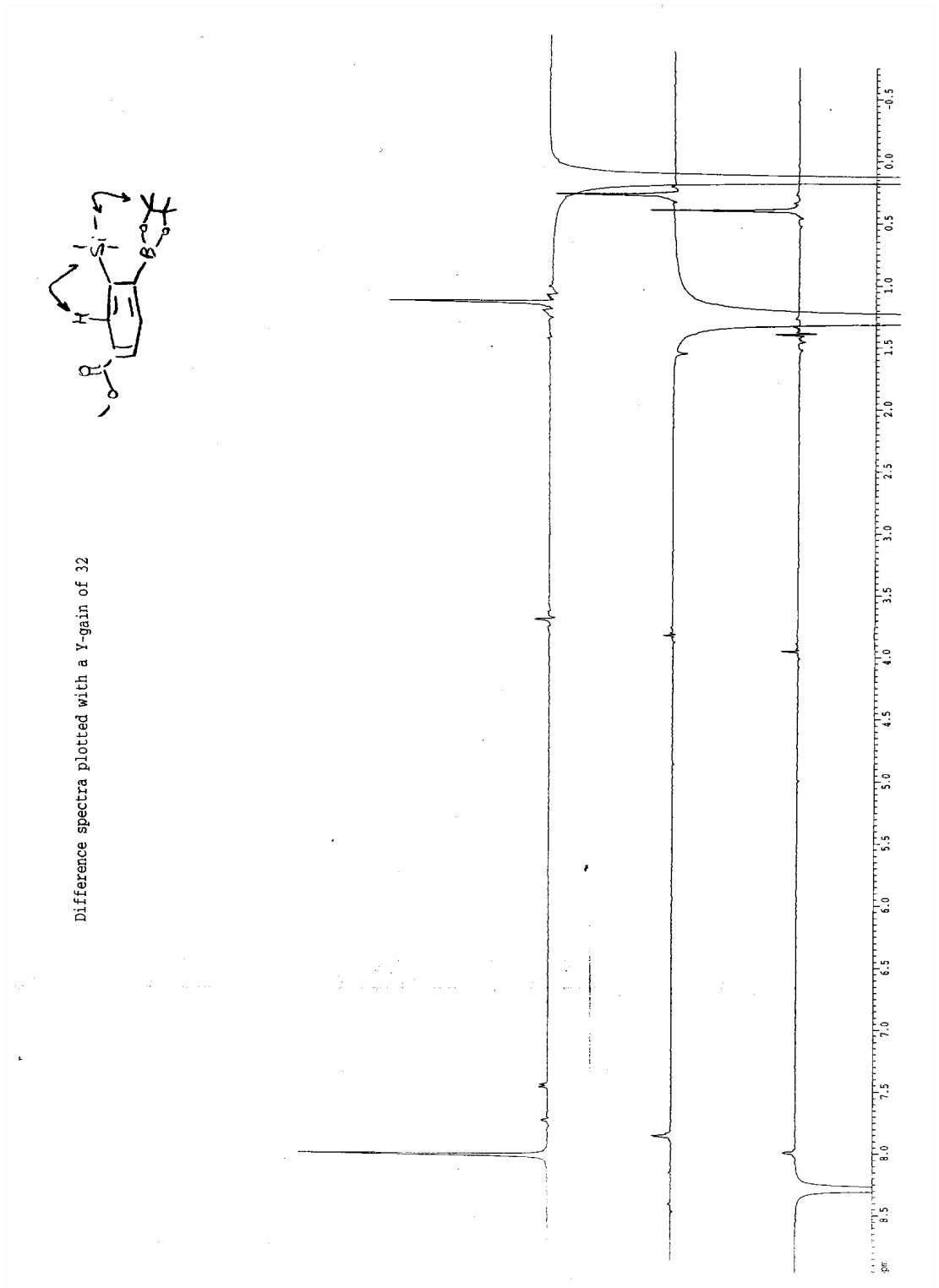
A round bottom flask was charged with **14** (0.088 g, 0.211 mmol), $PdCl_2$(dppf)DCM (0.015 g, 0.021 mmol), K_3PO_4 (.134 g, 0.633 mmol), dioxane (1 ml) and iodotoluene (0.092 g, 0.422 mmol). The flask was fitted with a reflux condenser and heated at 85 ^oC under N_2. After stirring for 48 h the reaction was cooled to room temperature and quenched by the addition of distilled water (10 ml), the product was extracted into dichloromethane (3 x 10 ml), dried ($MgSO_4$), filtered and conc. *in vacuo*. The product was purified by flash column chromatography (eluting solvent petroleum ether/ethyl acetate 15:1 ratio) to give compound **17** as colourless oil, wt. 0.076 g, 70% yield.

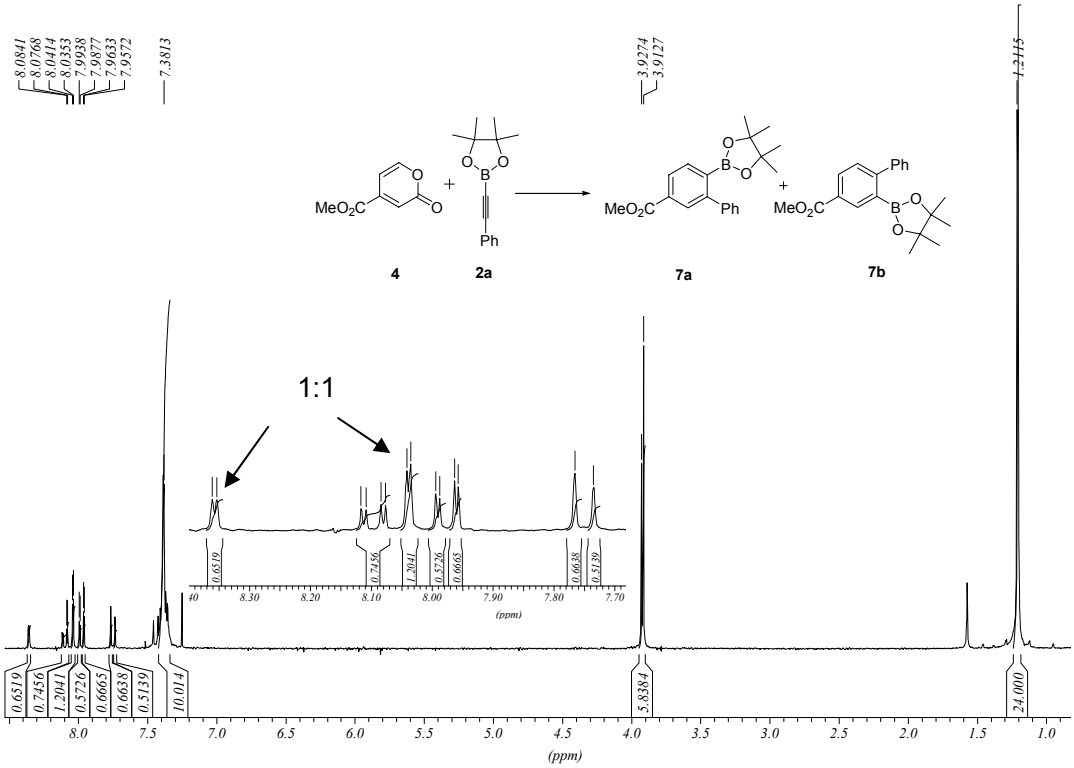
^{1}H NMR δ2.18 (3H, s, C*H*$_3$), 3.85 (3H, s, C*H*$_3$OCO), 6.82-6.90 (4H, m, Ar*H*), 6.98-7.02 (2H, m, Ar*H*), 7.18 (3H, m, Ar*H*), 7.94 (1H, d, *J*=1.5 Hz, Ar*H*), 8.25 (1H, d, *J*=1.5 Hz, Ar*H*). **^{13}C NMR** (62.9 MHz, $CDCl_3$): δ21.1, 52.4, 124.7, 127.4, 127.7, 128.5, 129.4, 130.2, 130.4, 132.5, 133.4, 136.7, 137.2, 139.5, 143.7, 145.5, 165.7. **FTIR**: $\nu_{max}/CHCl_3$, cm^{-1} 2360 (w), 1725 (s), 1240 (s). **HRMS** (EI$^+$) Calcd for $C_{21}H_{17}BrO_2$ 380.0412 Found: 380.0413.

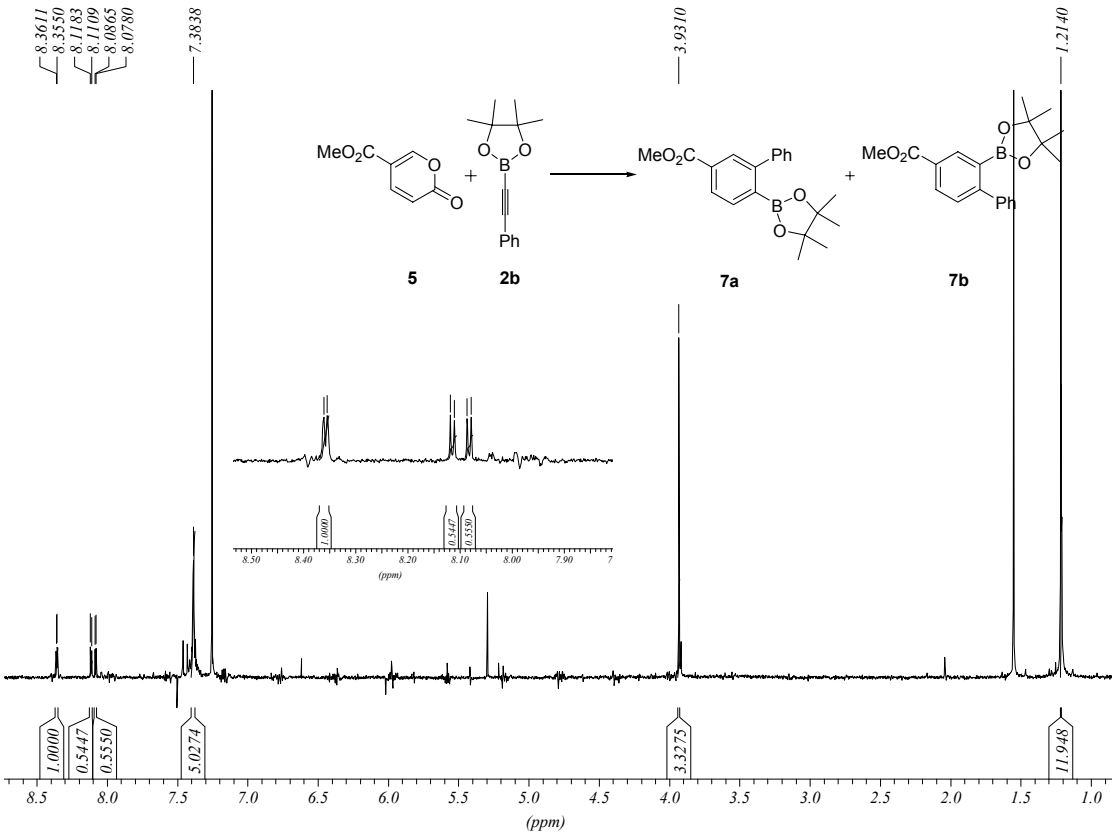


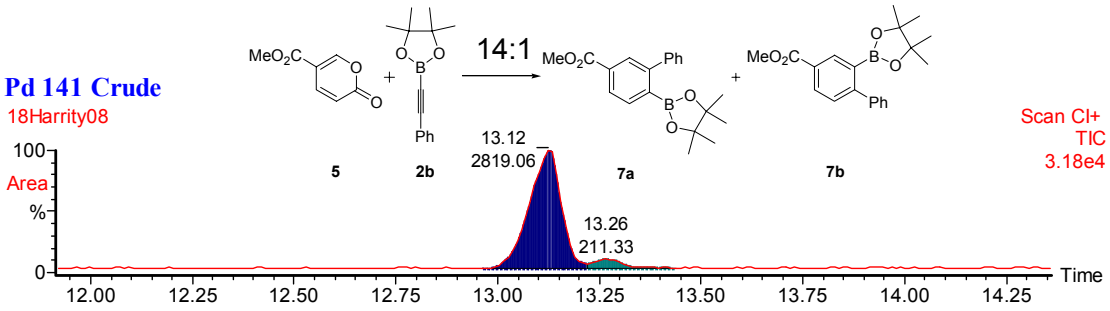


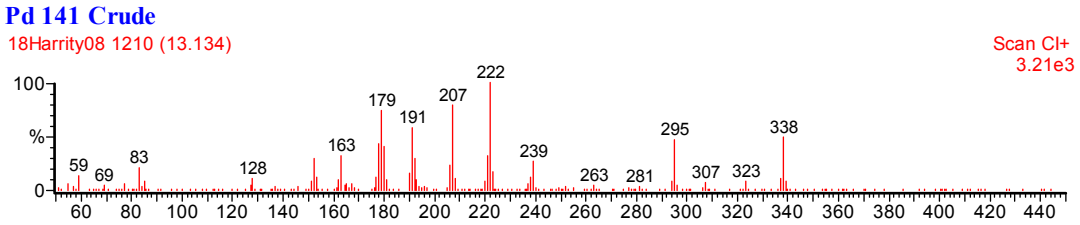


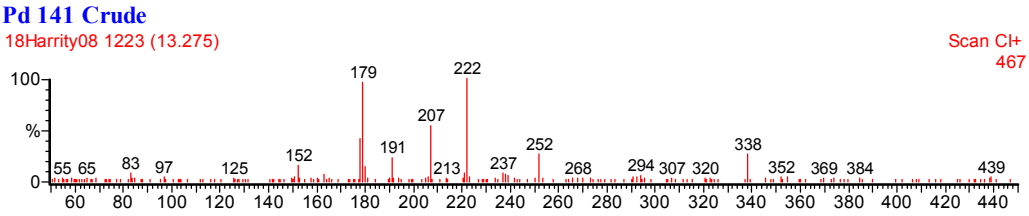


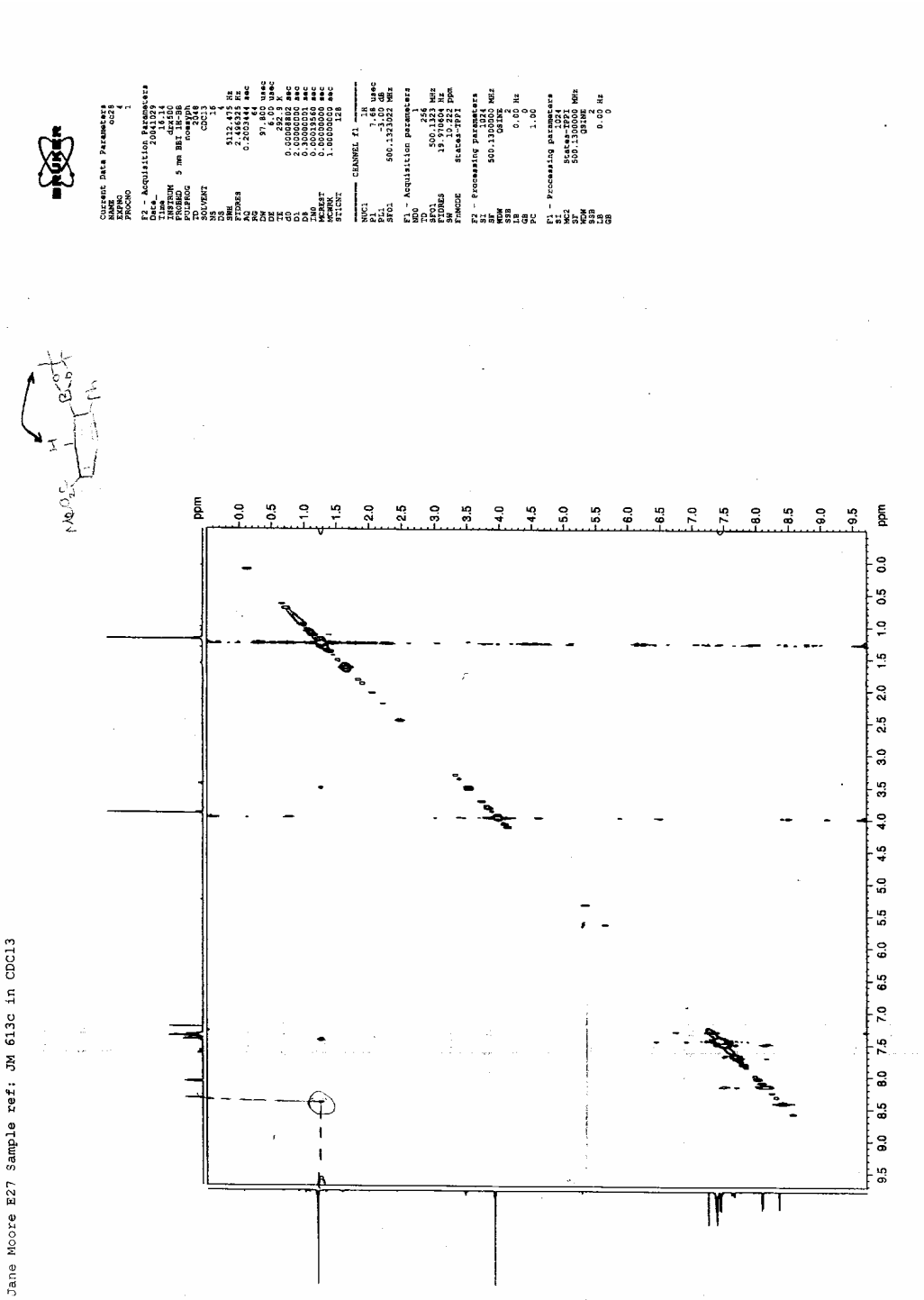


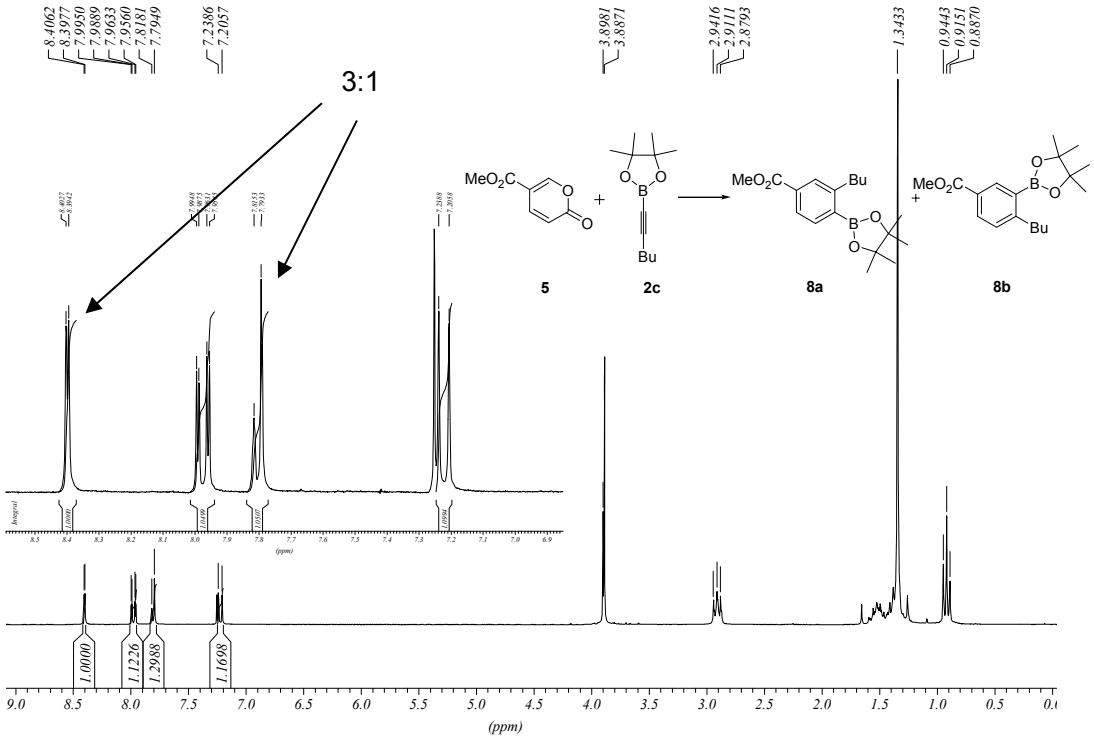








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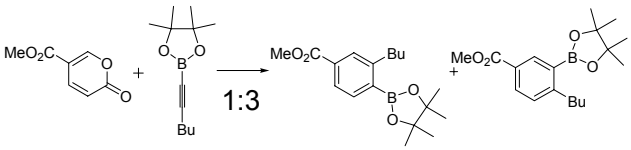
7.7949
7.2386
7.2057
3.8981
3.8871
2.9416
2.9111
2.8793
1.3433
0.9443
0.9151
0.8870
1.0000
1.1226
1.2988
1.1698
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
(ppm)
MeO2C
Bu
5
2c
8a
8b


MeO2C
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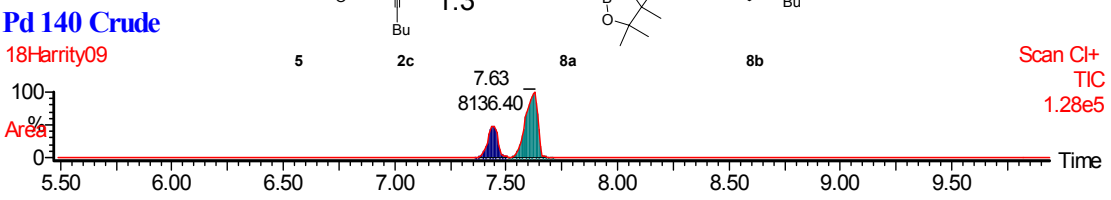
5
2c
8a
8b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18Harrity09
Scan CI+
TIC
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8136.40
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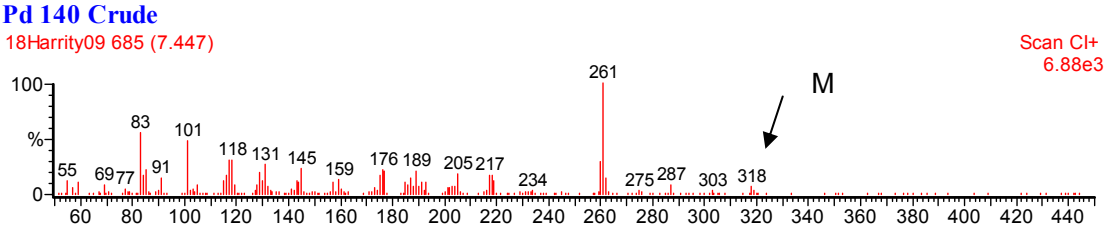
%
Area
0
5.50 6.00 6.50 7.00 7.50 8.00 8.50 9.00 9.50
Time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100
%
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101
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131
145
159
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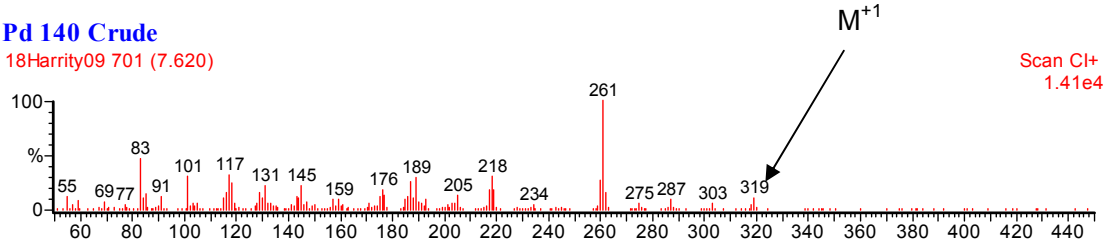
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Scan CI+
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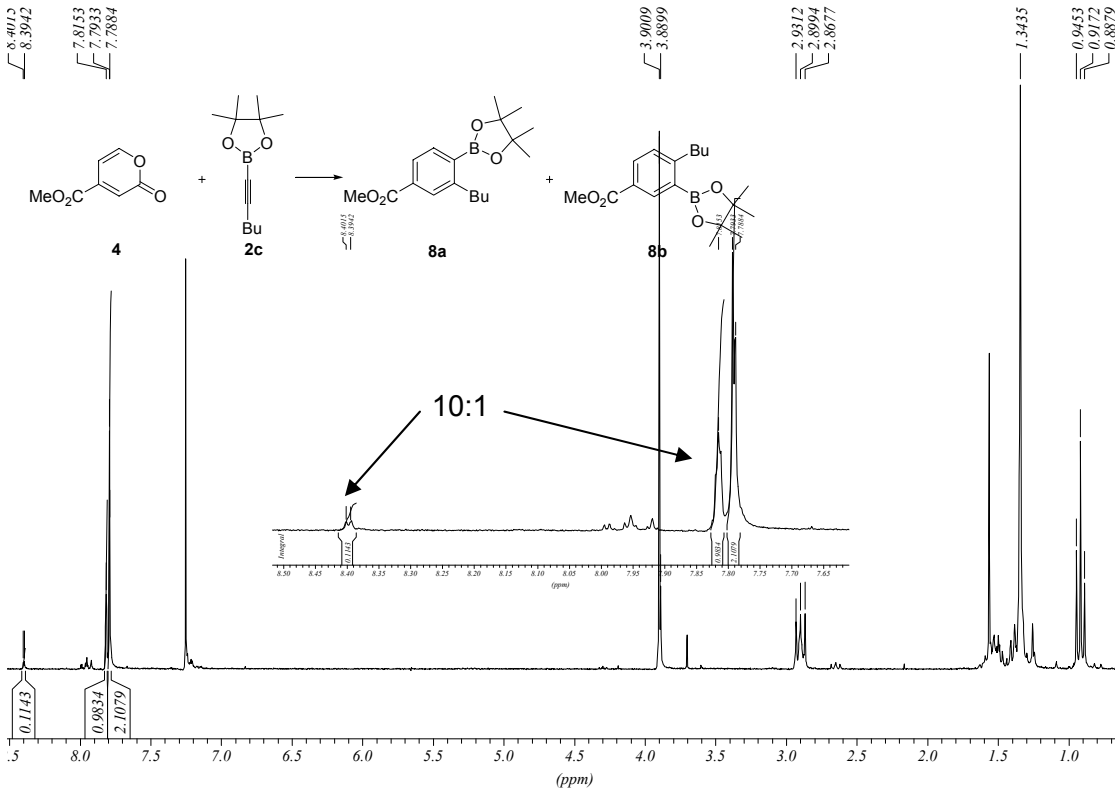
M+1
261
100
%
0
83
101
117
131
145
159
176
189
205
218
234
275
287
303
319
55
69
77
91
60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440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3.8899
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Bu
4
2c
8a
8b
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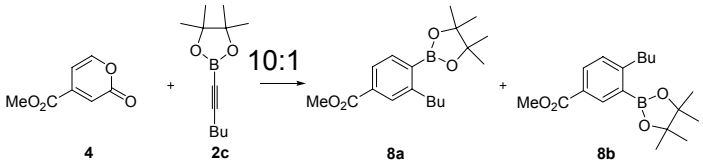
(ppm)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Bu
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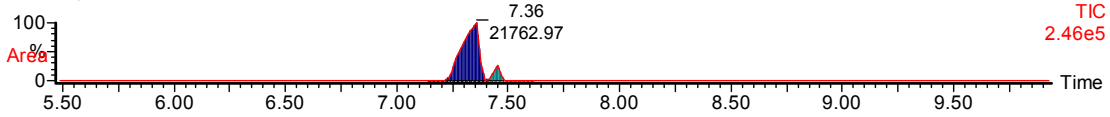
4
2c
8a
8b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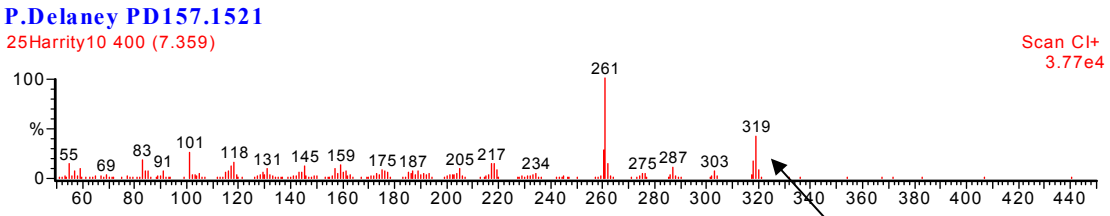
Area
Time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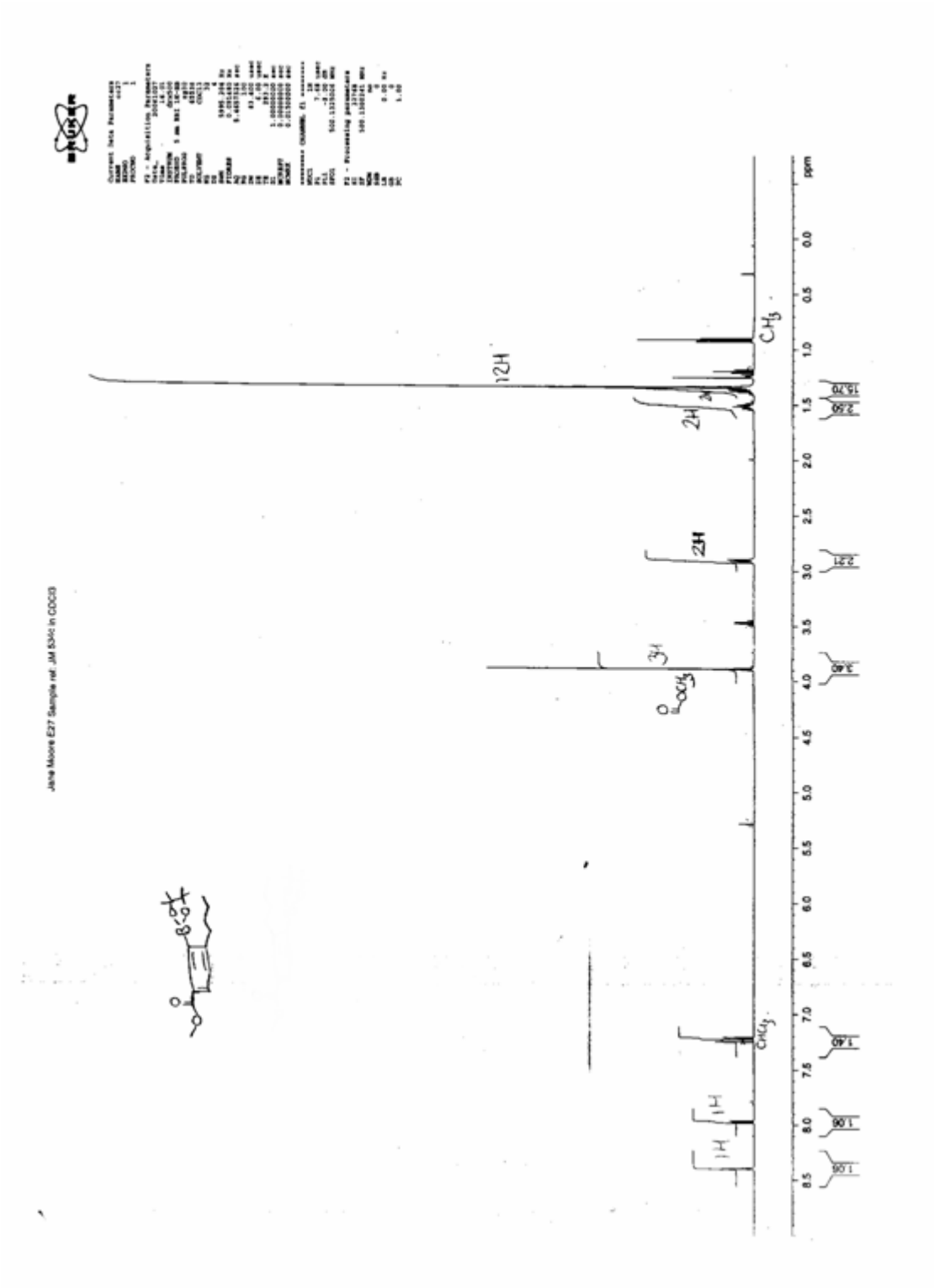
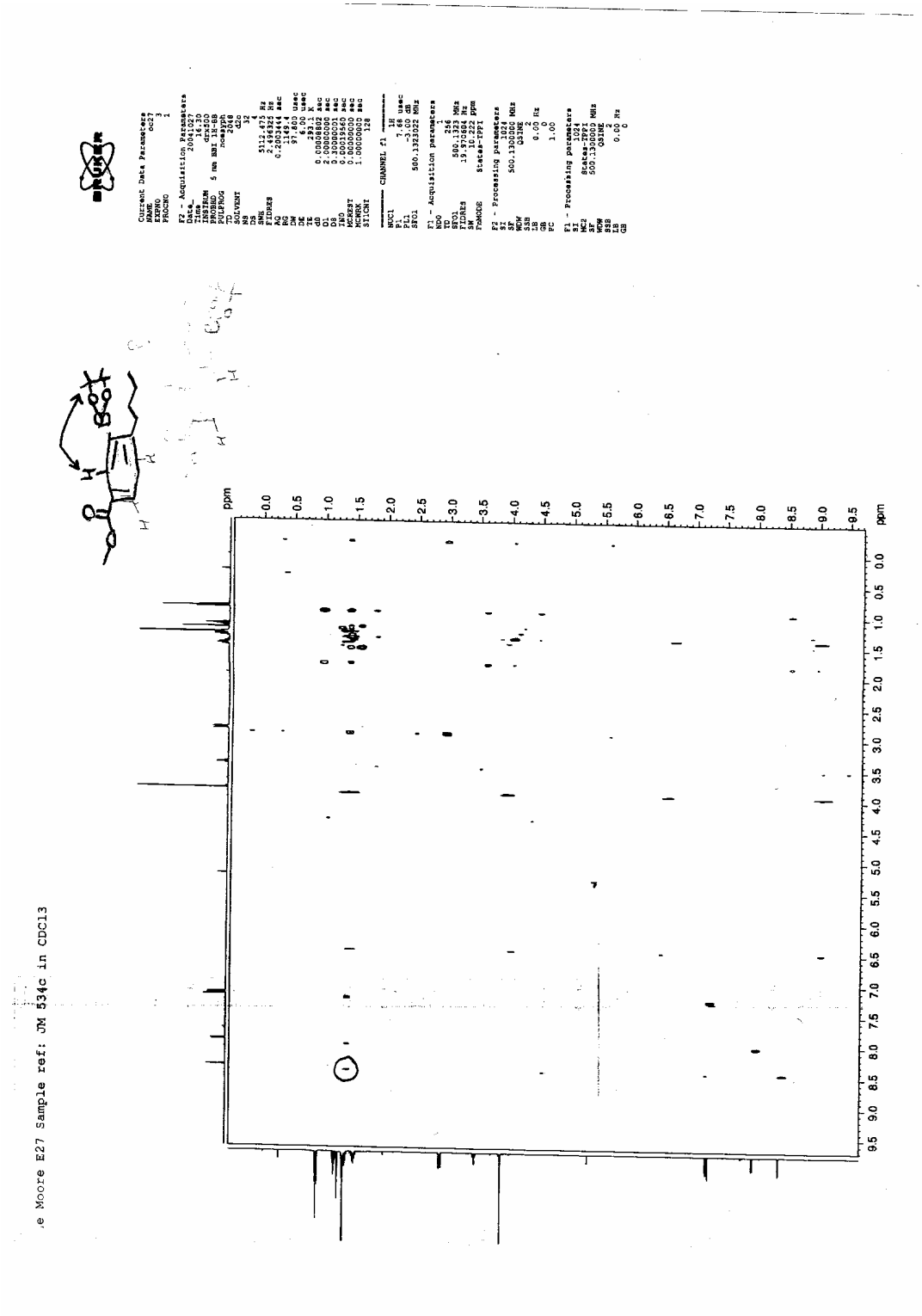
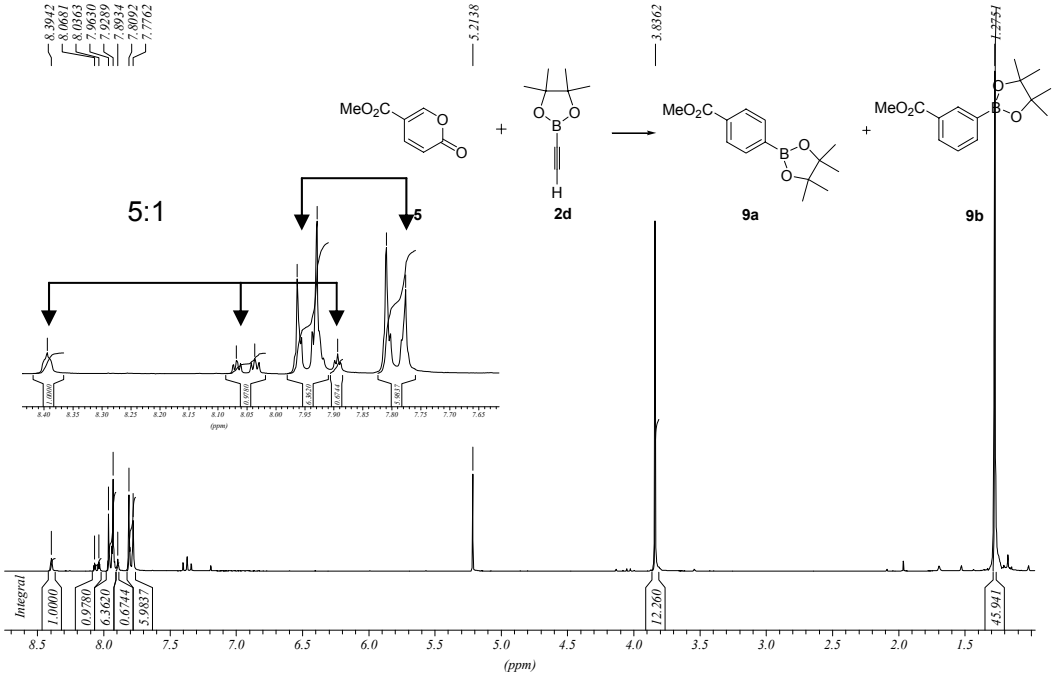
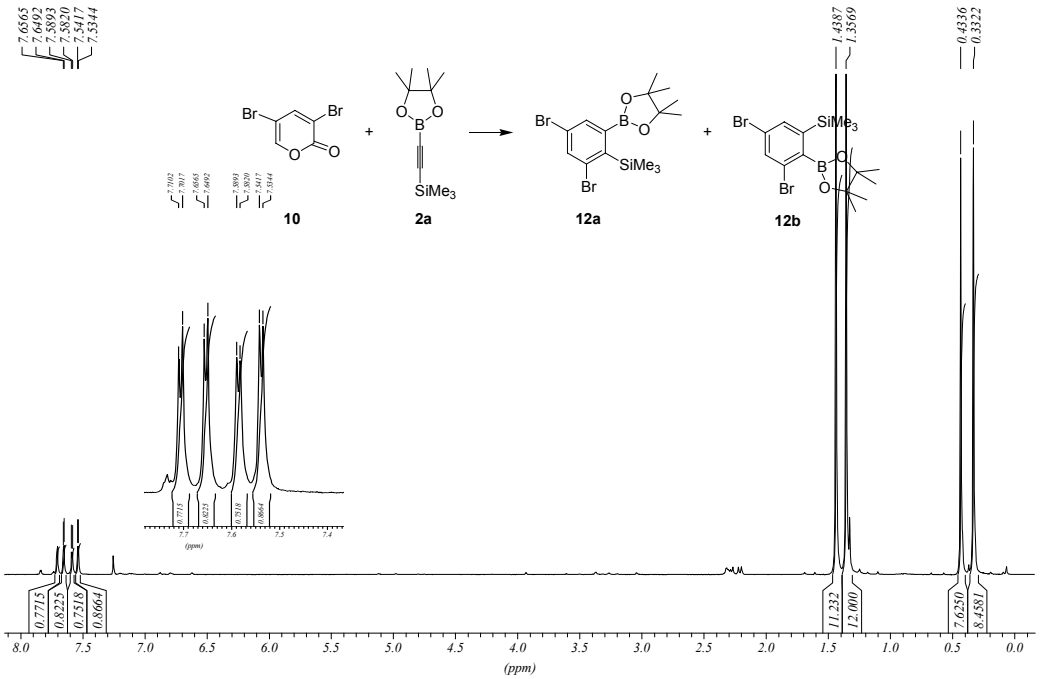
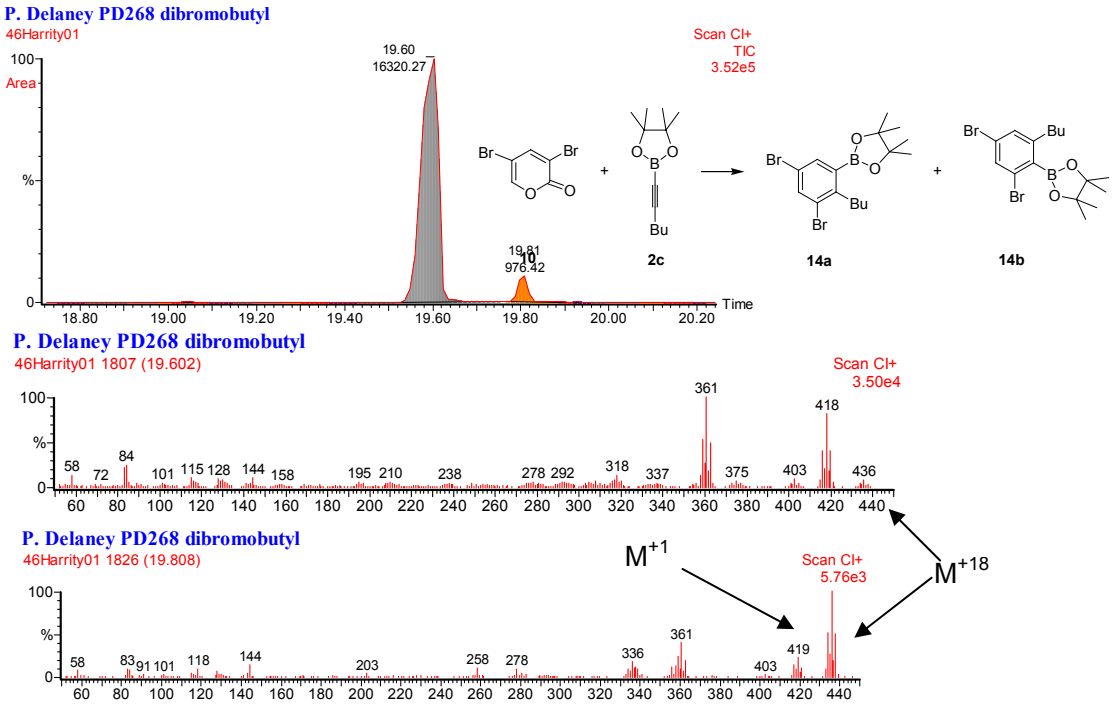
M+1
261
319











M^{+18} = mass ion + NH_3