

## Supporting information

Synthesis and cycloaddition reactions of strained alkynes derived from 2,2'-dihydroxy-1,1'-biaryls.

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### **Contents:**

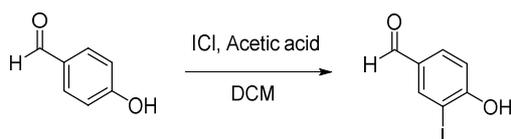
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## **Experimental data for compounds not listed in paper.**

### **General experimental section.**

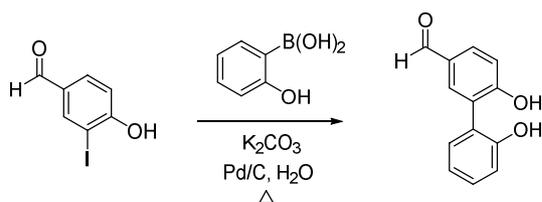
Solvents and reagents were degassed before use and all reactions were carried out under either a nitrogen atmosphere using vacuum line apparatus. Reactions were monitored by TLC using aluminium backed silica gel 60 (F254) plates, visualized using UV 254 nm and phosphomolybdic acid or potassium permanganate as appropriate. Flash column chromatography was carried out routinely on silica gel. Reagents were used as received from commercial sources unless otherwise stated. Dry solvents were purchased and used as received. <sup>1</sup>H NMR spectra were recorded on a Bruker DPX (300, 400 or 500 MHz) spectrometer. Chemical shifts are reported in  $\delta$  units, parts per million relative to the singlet at 7.26 ppm for chloroform and 0.00 ppm for TMS. Coupling constants (*J*) are measured in Hertz. Mass spectra for analysis of synthetic products were recorded on a Bruker Esquire 2000 or a Bruker MicroTOF mass spectrometer. IR spectra were recorded on a Perkin-Elmer Spectrum One FT-IR Golden Gate. Melting points were recorded on a Stuart Scientific SMP 1 instrument and are uncorrected. GPC was carried out on an Agilent 390LC MDS instrument equipped with differential refractive index (DRI), viscometry (VS) and dual angle light scatter (LS) detectors. The system was equipped with 2 x PLgel Mixed C columns (300 x 7.5 mm) and a PLgel 5  $\mu$ m guard column. The eluent was CHCl<sub>3</sub> with 2 % TEA (triethylamine) additive. Samples were run at 1ml/min at 30°C and narrow Poly(methyl methacrylate) standards were used to create a third order calibration between 1,568,000 – 550 gmol<sup>-1</sup>. Analyte samples were filtered through a GVHP membrane with 0.22  $\mu$ m pore size before injection. Respectively, experimental molar mass and dispersity values of synthesized polymers were determined by conventional calibration using Agilent GPC/SEC software. TGA measurements were performed on Mettler-Toledo DSC1 equipped with an autosampler. Samples were heated in 40 ul aluminium pans from 25-600 °C at a rate of 10 °C min under a nitrogen atmosphere. Samples were analysed used Mettler-Toledo STARe software. X-ray crystallography was carried out on a Rigaku Oxford Diffraction SuperNova diffractometer with a dual source (Cu at zero) equipped with an AtlasS2 CCD area detector or an Xcalibur Gemini diffractometer with a Ruby CCD area detector.

### **3-Iodo-4-hydroxybenzaldehyde 7.**



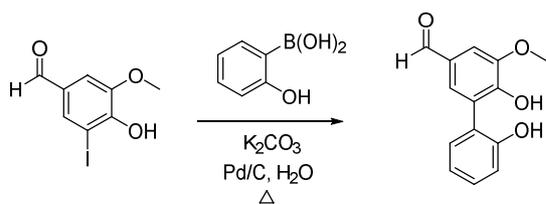
To a solution of 4-hydroxybenzaldehyde (1.22 g, 10 mmol, 1.0 eq.) in DCM (10 mL) was added to a solution of 1M iodine monochloride in DCM (21 mL, 21 mmol, 2.1 eq.) and acetic acid (1 mL) and left to stir for 2 days at rt. The solution was washed with  $\text{Na}_2\text{S}_2\text{O}_3$  (3 x 35 mL) thoroughly and the solvent collected dried with  $\text{Na}_2\text{SO}_4$  and the organics removed under vacuum to produce the product **7** as a white solid (1.95 g, 7.86 mmol, 79%).  $\delta_{\text{H}}$  (300 MHz,  $\text{CDCl}_3$ ) 9.79 (1 H, s, CHO), 8.22 (1 H, d,  $J$  1.8, ArH), 7.79 (1 H, dd,  $J$  8.4, 1.8, ArH), 7.11 (1 H, d,  $J$  8.4, ArH), 6.37 (1 H, br. s, OH). The data matched that reported; R. S. Harapanhalli, L. W. McLaughlin, R. W. Howell, D. V. Rao, S. J. Adelstein and A. I. Kassis, *J. Med. Chem.* 1996, **39**, 4804-4809.

### **2',6-Dihydroxybiphenyl-3-carbaldehyde 11.**



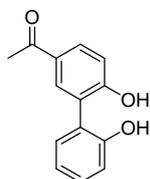
3-Iodo-4-hydroxybenzaldehyde **7** (0.49 g, 1.98 mmol, 1.0 eq.), 2-hydroxyphenyl boronic acid **10** (0.35 g, 2.57 mmol, 1.3 eq.), potassium carbonate (1.09 g, 7.92 mmol, 4.0 eq.) and 10% Pd/C (39.0 mg, 2 mol%) were added together and water (20 mL) added and left to stir at 80 °C. After 3 hours the mixture cooled and carefully acidified with 1M HCl, which was then extracted with ethyl acetate (3 x 35 mL). The organic layer combined and dried with  $\text{Na}_2\text{SO}_4$ , filtered and concentrated under vacuum. The crude solid was purified by column chromatography (1:1 hexane : diethyl ether) to afford **11** as an off-white solid (0.27 g, 1.26 mmol, 64 %).  $\delta_{\text{H}}$  (400 MHz,  $\text{CDCl}_3$ ) 9.91 (1 H, s, CHO), 7.87 (1 H, dd,  $J$  8.3, 2.0, 1H, ArH), 7.86 (1 H, d,  $J$  2.0, ArH), 7.39-7.34 (1 H, m, ArH), 7.30 (1 H, dd,  $J$  7.6, 1.4, ArH), 7.17 (1 H, d,  $J$  8.3, ArH), 7.10 (1 H, td,  $J$  7.6, 0.7, ArH), 7.04 (1 H, d,  $J$  8.2, ArH), 6.63 (1 H, s, OH), 5.87 (1 H, s, OH). Data matched that reported; B. Schmidt. M. Riemer and M. Karras, *J. Org. Chem.* 2013, **78**, 8680-8688.

### **2',6-Dihydroxy-5-methoxybiphenyl-3-carbaldehyde 12.**



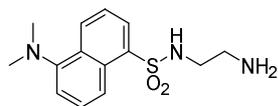
5-Iodovanillin **8** (2.50 g, 8.99 mmol, 1.0 eq.), 2-hydroxyphenyl boronic acid **10** (1.61 g, 11.7 mmol, 1.3 eq.), potassium carbonate (4.97 g, 3.6 mmol, 4.0 eq.) and 10% Pd/C (0.18 g, 2 mol%) were added together, water (90 mL) was added and the reaction was stirred at 80 °C. After 3 hours the mixture was cooled and carefully acidified with 1M HCl, then extracted with ethyl acetate (3 x 90 mL). The organic layers were combined and dried with Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated under vacuum. The crude solid was purified by column chromatography (1:1 hexane : diethyl ether) to afford **12** as an off-white solid (1.90 g, 7.78 mmol, 87 %).  $\delta_{\text{H}}$  (300 MHz, CDCl<sub>3</sub>) 9.89 (1 H, s, CHO), 7.50-7.47 (2 H, m, ArH), 7.35-7.30 (2 H, m, ArH), 7.10-7.04 (2 H, m, ArH), 6.77 (1 H, s, OH), 5.85 (1 H, s, OH), 4.05 (3 H, s, OCH<sub>3</sub>). The data matched that reported; B. Schmidt, M. Riemer and M. Karras, *J. Org. Chem.* 2013, **78**, 8680-8688.

### **3-Acetyl-2',6-Dihydroxy-biphenyl 13.**



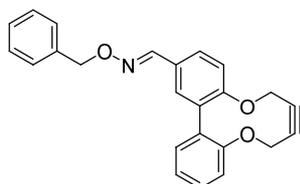
4-Hydroxy-3'-iodoacetophenone **9** (1.00 g, 3.82 mmol, 1.0 eq.), 2-hydroxyphenyl boronic acid **10** (646 mg, 5.08 mmol, 1.3 equiv), K<sub>2</sub>CO<sub>3</sub> (2.1 g, 15.2 mmol), and 10 wt% Pd/C (80 mg, 2 mol %) were added to a RBF and suspended in water (40 mL). The mixture was immersed in an oil bath preheated at 80 °C and heated at reflux for 2.5 hrs. The mixture was cooled to rt then, dropwise, was acidified to pH 5 with addition of HCl (aq, 1.0 M) and extracted with EtOAc (3 x 50 mL). The combined organic layers were dried with MgSO<sub>4</sub>, filtered and solvent removed via rotary evaporation. The residue was purified by column chromatography on silica, using hexane/EtOAc mixtures of increasing polarity until an equal gradient was attained (hexane/EtOAc 1:1), to give compound **13** (685 mg, 3.0 mmol, 78 %) as a white solid. Mp 130 °C;  $\delta_{\text{H}}$  (400 MHz, CDCl<sub>3</sub>) 7.94 (2 H, brs, ArH), 7.35 (1 H, t, *J* 7.5, ArH), 7.30-7.26 (1 H, m, ArH), 7.10-7.03 (3 H, m, ArH), 6.38 (2 H, brs, OH), 2.58 (3 H, s, CH<sub>3</sub>); *m/z* (ESI) 228.2 [M]<sup>+</sup>, 251.2 ([M+Na]<sup>+</sup>). The data matched that reported; J. P. Bachelet, P. Demerseman and R. Royer, *J. Heterocyclic Chem.* 1977, **14**, 1409-1411.

### N-(2-Aminoethyl)-5-(dimethylamino)naphthalene-1-sulfonamide 34.



(5-(Dimethylamino)naphthalene-1-sulfonyl chloride (3.0 g, 11.1 mmol) was dissolved in 1,2-diaminoethane (50 mL) and stirred for 18 h at ambient temperature. The solvent was removed under reduced pressure, redissolved in EtOAc (20 mL) and washed with sat. NaHCO<sub>3(aq)</sub> (2 × 20 mL) and H<sub>2</sub>O (20 mL). The organic layer was dried over MgSO<sub>4</sub>. Purification by column chromatography (silica; MeOH/H<sub>2</sub>O; 100:0→90:10) afforded the product **34** as a yellow solid (0.94 g, 3.20 mmol, 29%).  $\delta_{\text{H}}$  (500 MHz, CDCl<sub>3</sub>) 8.53 (1 H, d, *J* 8.6, ArH), 8.32 (1 H, d, *J* 8.6, ArH), 8.24 (1H, d, *J* 7.3, ArH), 7.54 (1H, t, *J* 8.0, ArH), 7.50 (1H, t, *J* 8.0, ArH), 7.16 (1H, d, *J* 7.5, ArH), 2.92 (2H, t, *J* 5.8, NCH<sub>2</sub>), 2.88 (6H, s, N(CH<sub>3</sub>)<sub>2</sub>), 2.72 (2H, d, *J* 5.8, CH<sub>2</sub>NH<sub>2</sub>).  $\delta_{\text{C}}$  (126 MHz, CDCl<sub>3</sub>) 152.0, 134.8, 130.5, 130.0, 129.7, 129.6, 128.5, 123.3, 118.9, 45.5, 45.4, 41.0. The data matched that reported; R. C. Knighton, M. R. Sambrook, J. C. Vincent, S. A. Smith, C. J. Serpell, J. Cookson, M. S. Vickers and P. D. Beer, *Chem. Commun.* 2013, **49**, 2293-2295. .

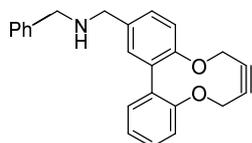
### Oxime ether 29.



To a solution of aldehyde alkyne **15** (50 mg, 0.17 mmol, 1.0 eq) in MeOH (1 mL) was added NaOAc (35 mg, 0.43 mmol, 2.5 eq) and benzylhydroxylamine (32 mg, 0.2 mmol, 1.2 eq). The mixture was stirred at 45 °C overnight. The mixture was cooled to rt and the MeOH removed under vacuum. Saturated NaHCO<sub>3</sub> solution (20 mL) was added and the product extracted with DCM (3 x 20 mL). The combined organic layers were dried over MgSO<sub>4</sub> and concentrated under vacuum to give the crude product, which was purified by flash column chromatography (eluent = DCM) to give the product **29** as a thick colourless oil (45 mg, 0.11 mmol, 66 %). *R*<sub>f</sub> = 0.70 (DCM); (Found(ESI)) [M + Na]<sup>+</sup> 392.1257 C<sub>24</sub>H<sub>19</sub>NNaO<sub>3</sub> requires 392.1257 (no molecular ion found by LRMS);  $\nu_{\text{max}}$  3028, 2962, 2916, 2864, 1497, 1475, 1452, 1346, 1194 and 970 cm<sup>-1</sup>;  $\delta_{\text{H}}$  (500 MHz, CDCl<sub>3</sub>) 8.15 (1 H, s, N=CH), 7.68 (1 H, dd, *J* 8.4, 1.9, ArH), 7.36 – 7.46 (6 H, m, ArH), 7.31 – 7.35 (1 H, m, ArH), 1.17 – 7.24 (4 H, m, ArH), 5.21 (2 H, s, PhCH<sub>2</sub>O), 4.56 (2 H, d, *J* 16.2, 2 x CHH), 4.36 (2 H, d, *J* 12.5, 2 x CHH)

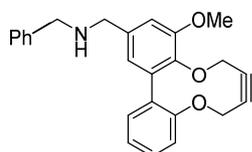
;  $\delta_C$  (125 MHz,  $CDCl_3$ ) 155.8, 154.4, 148.3, 137.5, 136.3, 135.3, 131.9, 131.2, 129.3, 128.4, 128.3, 128.3, 127.9, 127.4, 124.3, 123.1, 122.6, 86.9, 86.4, 76.4, 63.6 and 63.5.

### **Benzyl amine derivative 30.**



Aldehyde **15** (100 mg, 0.378 mmol) and benzylamine (40 mg, 0.378 mmol) in EtOH (2 mL) were refluxed for 1 h. The mixture was cooled to 0 °C and  $NaBH_4$  (35.7 mg, 0.945 mmol) was added. The reaction was stirred for 24 h and at the end of this time the solvent was removed under vacuum. DCM (10 mL) and water (10 mL) were added and the organic layer was separated. The water layer was extracted with further DCM (2 x 10 mL) and the combined organic extracts were dried ( $MgSO_4$ ), filtered and the solvent removed to leave 125 mg of crude product. The product was purified by flash chromatography (EtOAc:hexane 7:3, silica) to give the product **30** as a clear oil (60 mg, 0.169 mmol, 45%) (40 mg of impure product was also isolated). TLC EtOAc:hexane 7:3, silica,  $R_f$  0.30; (Found (ESI+):  $[M+H]^+$  356.1648.  $C_{24}H_{22}NO_2$  requires 356.1645);  $\nu_{max}$  2860, 1497, 1474, 1449, 1251, 1119, 968, 759, 731  $cm^{-1}$ ;  $\delta_H$  (500 MHz,  $CHCl_3$ ) 7.45-7.15 (12 H, m, ArH), 4.56 (2 H, d,  $J$  14.0, OCHH), 4.36 (2 H, d,  $J$  14.0, CHH), 3.86 (2 H, s,  $CH_3$ ), 3.82 (2 H, s,  $CH_3$ ), 1.75 (1 H, brs, NH);  $\delta_C$  (125 MHz,  $CDCl_3$ ) 154.5 (C), 153.4 (C), 140.3 (C), 138.8 (C), 136.1 (C), 132.7 (CH), 132.0 (CH), 131.8 (CH), 131.1 (CH), 128.9 (CH), 128.4 (CH), 127.0 (CH), 124.2 (CH), 122.6 (CH), 122.5 (CH), 86.8 (C), 86.7 (C), 62.55 ( $CH_2$ ), 53.3 ( $CH_2$ ), 52.7 ( $CH_2$ );  $m/z$  (ES-API+) 355.9 ( $[M+H]^+$ ).

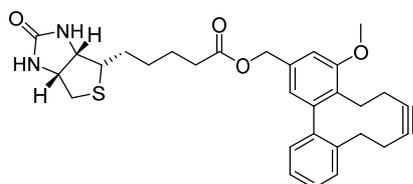
### **Benzyl amine derivative 31.**



Aldehyde **16** (50 mg, 0.17 mmol) and benzylamine (18 mg, 0.17 mmol) in EtOH (1 mL) were refluxed for 1 h. The mixture was cooled to 0 °C and  $NaBH_4$  (32 mg, 0.86 mmol) was added. The reaction was stirred for 24 h and at the end of this time the solvent was removed under vacuum. DCM (10 mL) and water (10 mL) were added and the organic layer was separated. The water layer was extracted with further DCM (2 x 10 mL) and the combined organic

extracts were dried (MgSO<sub>4</sub>), filtered and the solvent removed to leave 64 mg of crude product. The product was purified by flash chromatography (EtOAc:hexane 7:3, silica) to give the product **31** as a clear oil (53 mg, 0.138 mmol, 81%). TLC EtOAc:hexane 7:3, silica, Rf 0.3; (Found (ESI+): [M+H]<sup>+</sup> 386.1751. C<sub>25</sub>H<sub>24</sub>NO<sub>3</sub> requires 386.1751);  $\nu_{\max}$  2926, 2836, 1584, 1450, 1194, 1134, 906, 730, 698 cm<sup>-1</sup>;  $\delta_{\text{H}}$  (500 MHz, CHCl<sub>3</sub>) 7.40-7.15 (9 H, m, ArH), 7.03 (1 H, s, ArH), 6.77 (1 H, s, ArH), 4.65 (1 H, d, *J* 15.0, OCH), 4.58 (1 H, d, *J* 16.0, OCH), 4.40 (1 H, d, *J* 16.0, OCH), 4.37 (1 H, d, *J* 15.0, OCH), 3.95 (3 H, s, OCH<sub>3</sub>), 3.86 (2 H, s, CH<sub>2</sub>), 3.83 (2 H, s, CH<sub>2</sub>) 1.90 (1 H, brs, NH);  $\delta_{\text{C}}$  (125 MHz, CDCl<sub>3</sub>) 154.6 (C), 154.4 (C), 153.1 (C), 146.4 (C), 141.3 (C), 132.9 (CH), 128.8 (CH), 128.3 (CH), 128.0 (CH), 127.3 (CH), 125.5 (CH), 124.4 (CH), 124.2 (CH), 111.4 (CH), 87.6 (C), 86.1 (C), 63.6 (CH<sub>2</sub>), 60.3 (CH<sub>2</sub>), 60.3 (CH<sub>3</sub>), 53.5 (CH<sub>2</sub>), 53.3 (CH<sub>2</sub>). *m/z* (ES-API+) 386.0 ([M+H]<sup>+</sup>). The cyclisation reaction of **32** with benzyl azide **20** was followed over time and full details are given in the Supporting Information.

### **Biotin functionalised alkyne 41.**

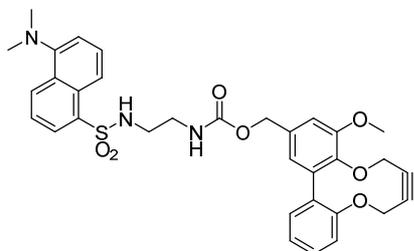


To a solution of alcohol **19** (50 mg, 0.17 mmol, 1.2 eq) in DCM (5 mL), EDC.HCl (32.6 mg, 0.17 mmol, 1.2 eq), biotin (35 mg, 0.14 mmol, 1.0 eq) and DMAP (1 mg, catalytic, 0.01 eq) were added and stirred at rt for 3 days. After this time the reaction was diluted with DCM (20 mL) and washed with water (3 x 20 mL). The organic layer was then dried over MgSO<sub>4</sub> and concentrated under vacuum. The crude product was then purified by flash column chromatography (eluent gradient of EtOAc – Pet. Ether 8:2 to EtOAc – MeOH 9:1) to give the pure product **41** as a white solid (31.6 mg, 0.06 mmol, 43 %).

Rf = 0.35 (EtOAc – MeOH 95:5), (Found(ESI)) [M + Na]<sup>+</sup> 545.1718. C<sub>28</sub>H<sub>30</sub>N<sub>2</sub>NaO<sub>6</sub>S requires 545.1717);  $\nu_{\max}$  3243, 3013, 2927, 2862, 1698, 1494, 1337, 1135, 966 and 746 cm<sup>-1</sup>;  $\delta_{\text{H}}$  (500 MHz, CDCl<sub>3</sub>) 7.35 – 7.41 (1 H, m, ArH), 7.15 – 7.21 (3 H, m, ArH), 6.95 (1 H, s, ArH), 6.79 (1 H, s, ArH), 5.77 (1 H, brs, NH), 5.21 (1 H, brs, NH), 5.09 (2 H, s, CH<sub>2</sub>OCO), 4.61 (1 H, d, *J* 15.3, CHH), 4.55 (1 H, d, *J* 15.5, CHH), 4.44 – 4.49 (1 H, m, NHCH), 4.41 (1 H, d, *J* 15.5, CHH), 4.35 (1 H, d, *J* 15.3, CHH), 4.25 – 4.30 (1 H, m, NHCH), 3.93 (3 H, s, OCH<sub>3</sub>), 3.09 – 3.15 (1 H, m, SCH), 2.88 (1 H, dd, *J* 4.9, 12.8, SCHH), 2.69 (1 H, d, *J* 12.8, SCHH), 2.40 (2 H, t, *J* 7.5, CH<sub>2</sub>), 1.60 – 1.76 (4 H, m, 2 x ), 1.36 – 1.52 (2H, m, CH<sub>2</sub>);  $\delta_{\text{C}}$

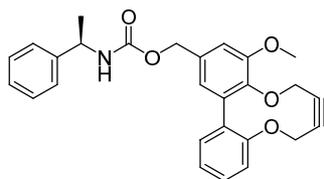
(125 MHz, CDCl<sub>3</sub>) 173.4, 163.4, 154.3, 153.2, 142.32, 137.1, 135.6, 131.9, 131.9, 129.2, 124.2, 123.8, 122.5, 111.1, 87.4, 86.2, 66.1, 63.6, 61.7, 60.3, 60.0, 55.8, 55.3, 40.5, 33.8, 28.2, 28.1 and 24.7; *m/z* (ESI) 545 ([M+Na]<sup>+</sup>).

### **Dansyl carbamate derivative 42.**



To a solution of **19** (50 mg, 0.17 mmol, 1.0 eq) in MeCN (0.5 mL) was added TEA (68.5 mg, 0.68 mmol, 4.0 eq) and DSC (65 mg, 0.25 mmol, 1.5 eq) and the reaction was stirred for 3 h at rt. The MeCN was removed under vacuum and the resulting residue dissolved in DMF (0.2 mL). The solution was then added to a separate solution of DIPEA (54.8 mg, 0.07 mL, 0.43 mmol, 2.5 eq) and dansyl amine (100 mg, 0.34 mmol, 2 eq) in DMF (0.5 mL) and the reaction stirred overnight. The mixture was diluted with DCM (20 mL), washed with water (3 x 20 mL) and dried over MgSO<sub>4</sub> before being concentrated under vacuum to give the crude product. The product was then purified by flash column chromatography to give the pure product **42** as a thick light green oil (55.6 mg, 0.090 mmol, 53 %). *R<sub>f</sub>* = 0.3 (DCM – EtOAc 9:1) (Found(ESI) [M + Na]<sup>+</sup> 638.1931. C<sub>33</sub>H<sub>33</sub>N<sub>3</sub>NaO<sub>7</sub>S requires 638.1931);  $\nu_{\max}$  3301, 2927, 2868, 2789, 1699, 1453, 1136 and 788 cm<sup>-1</sup>;  $\delta_{\text{H}}$  (500 MHz, CDCl<sub>3</sub>) 8.53 (1 H, d, *J* 8.5, ArH) 8.19 – 8.27 (2 H, m, ArH), 7.50 (1 H, t, *J* 7.9, ArH), 7.36 – 7.42 (1 H, m, ArH), 7.13 – 7.22 (4 H, m, ArH), 6.91 (1 H, s, ArH), 6.74 (1 H, s, ArH), 5.26 – 5.31 (1 H, brs, NH), 5.02 (2 H, s, PhCH<sub>2</sub>O) 4.84 – 4.93 (1 H, brs, NH), 4.60 (1 H, d, *J* 15.4, CHH), 4.55 (1 H, d, *J* 15.6, CHH), 4.40 (1 H, d, *J* 15.6, CHH), 4.34 (1 H, d, *J* 15.3, CHH), 3.91 (3 H, s, OCH<sub>3</sub>), 3.12 – 3.27 (2 H, m, NHCH<sub>2</sub>), 2.98 – 3.09 (2 H, m, NHCH<sub>2</sub>), 2.87 (6 H, s, N(CH<sub>3</sub>)<sub>2</sub>);  $\delta_{\text{C}}$  (125 MHz, CDCl<sub>3</sub>) 156.7, 154.3, 153.3, 152.1, 142.2, 137.04, 135.7, 134.5, 132.4, 132.0, 130.7, 129.9, 129.7, 129.5, 129.2, 128.5, 124.3, 123.3, 122.6, 118.5, 115.3, 110.8, 87.5, 86.2, 66.6, 63.7, 60.4, 55.9, 45.4, 43.4 and 40.8; *m/z* (ESI) 638 ([M+Na]<sup>+</sup>). Fluorescence (MeCN;  $\lambda_{\text{ex}}$  = 259 nm);  $\lambda_{\text{em}}$  = 519 nm; UV-Vis (MeCN)  $\lambda_{\text{max}}$  ( $\epsilon/\text{M}^{-1} \text{cm}^{-1}$ ): 342 (3057) nm.

### **Carbamate alkyne 43.**



Alkyne methoxy alcohol **19** (60 mg, 0.202 mmol) and DMAP (one crystal, catalytic) were dissolved in CH<sub>2</sub>Cl<sub>2</sub> (2 mL). (*R*)-(1-isocyanatoethyl)benzene (34 μL, 0.242 mmol) was added and the reaction was stirred under N<sub>2</sub> at rt for 18 hours. The solvent was removed in vacuo and the crude mixture was purified by column chromatography (SiO<sub>2</sub>; EtOAc/Hex; 20:80→50:50) to afford product **43** as a white solid (79 mg, 0.18 mmol, 89%). Mp 82-84 °C; (found (ESI) [M + Na]<sup>+</sup>, 466.1630. C<sub>27</sub>H<sub>25</sub>NNaO<sub>5</sub> requires 466.1625). ν<sub>max</sub> 3324, 2968, 3934, 1701, 1450, 1135, 964 and 699 cm<sup>-1</sup>; δ<sub>H</sub> (500 MHz, CDCl<sub>3</sub>) 7.40 – 7.35 (1 H, m, ArH), 7.31 (3 H, brs, ArH), 7.25 (2 H, brs, ArH), 7.22 – 7.11 (4 H, m, ArH), 6.93 (1 H, s, ArH), 6.79 (1 H, s, ArH), 5.12 – 5.01 (2 H, m, ArCH<sub>2</sub>O), 4.91 – 4.81 (1 H, m, ArCHCH<sub>3</sub>), 4.71 – 4.50 (2 H, m, OCH<sub>2</sub>), 4.44 – 4.27 (2 H, m, OCH<sub>2</sub>), 3.89 (3 H, s, OCH<sub>3</sub>), 1.48 (3 H, d, *J* 6.9, ArCHCH<sub>3</sub>). δ<sub>C</sub> (126 MHz, CDCl<sub>3</sub>) 154.5, 153.3, 137.1, 135.8, 132.6, 132.2, 129.3, 128.8, 127.5, 126.1, 124.3, 123.7, 122.6, 111.1, 87.6, 86.3, 66.7, 63.7, 60.5, 55.9, 50.9, 22.6; *m/z* (ESI) 466 ([M+Na]<sup>+</sup>, 100%) and 482 ([M+K]<sup>+</sup> 40).

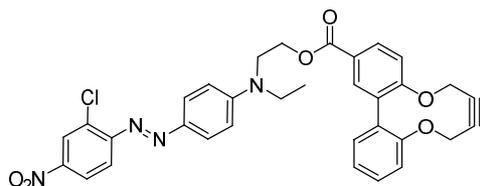
#### **Carbamate 44.**



To a solution of acid **5** (74 mg, 0.264 mmol) in DCM (4 mL) at 0 °C was added trimethylamine (0.029 mL, 0.28 mmol). A solution of diphenylphosphoryl azide (95 mg, 0.074 mL) in DCM (4 mL) was then added dropwise over 30 min. The reaction was stirred at 0 °C for 5 h then the solvent was evaporated, methanol (5 mL) was added and the reaction was heated overnight at 65 °C. At the end of this time, the solvent was removed and the product was purified by chromatography on silica gel (hexane:EtOAc 8:2) to give the crude product as a white solid (30 mg). TLC showed the presence of a side product at similar R<sub>f</sub> therefore the product was washed with hexane (10 x 2 mL) to leave the product **44** as a white solid (25 mg, 0.0809 mmol, 31%). TLC hexane: EtOAc 1:1, silica, R<sub>f</sub> 0.75; M.p. 119-121 °C; (Found (ESI<sup>+</sup>): [M+Na]<sup>+</sup> 332.0888. C<sub>18</sub>H<sub>15</sub>NNaO<sub>4</sub> requires 332.0893); ν<sub>max</sub> 3301, 1731, 1614, 1536, 1503, 1476, 1227, 1187, 941, 700, 518 cm<sup>-1</sup>; δ<sub>H</sub> (500 MHz, CHCl<sub>3</sub>) 7.62 (1 H,

brs, ArH), 7.40-7.34 (1 H, m, ArH), 7.22-7.10 (4 H, m, ArH), 7.03 (1 H, d, *J* 2.0, ArH), 6.62 (1 H, brs, NH), 4.61-4.52 (2 H, m, OCH), 4.38-4.30 (2 H, m, OCH), 3.76 (3 H, s, CH<sub>3</sub>);  $\delta_C$  (125 MHz, CDCl<sub>3</sub>) 154.3 (C), 150.3 (C), 136.3 (C), 135.5 (C), 133.8 (C), 131.9 (CH), 129.3 (CH), 125.4 (CH), 124.3 (CH), 122.6 (CH), 86.7 (C), 86.6 (C), 63.6 (CH<sub>2</sub>), 63.5 (CH<sub>2</sub>), 52.4 (CH<sub>3</sub>); *m/z* (ES-API+) 331.8 ([M+Na]<sup>+</sup>).

### **Ester linked to disperse red 45.**

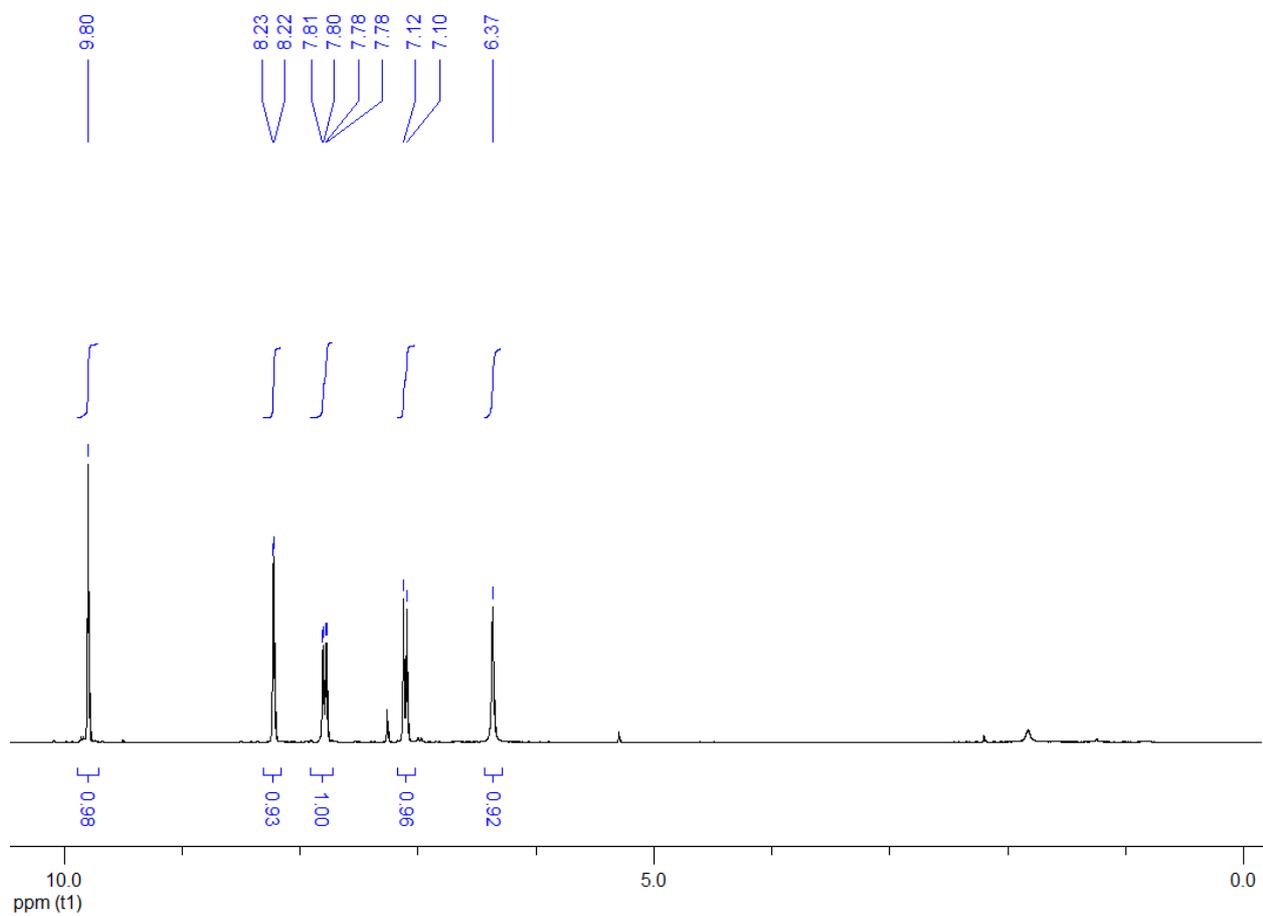
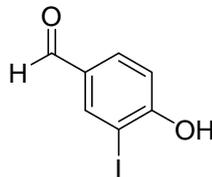


A solution of acid **5** (45 mg, 0.161 mmol), disperse red 13 (67 mg, 0.193 mmol), DMAP (1 mg) and EDC.HCl (37 mg, 0.193 mmol) in DCM (5.0 mL) was stirred at rt overnight. At the end of this time, EtOAc (20 mL) was added and the solution was washed with water (3 x 20 mL). The organic phase was dried with Na<sub>2</sub>SO<sub>4</sub>, filtered and the solvent removed under vacuum to give the crude product. Product **45** was purified by chromatography on silica gel (100% DCM) and was isolated as a red solid (60 mg, 0.098 mmol, 61%). TLC DCM, silica, R<sub>f</sub> 0.60; M.p. 120-123 °C; (Found (ESI+): [M+H]<sup>+</sup> 611.1687. C<sub>33</sub>H<sub>28</sub><sup>35</sup>ClN<sub>4</sub>NaO<sub>6</sub> requires 611.1692);  $\nu_{\max}$  2971, 1713, 1597, 1513, 1332, 1227, 1137, 665 cm<sup>-1</sup>;  $\delta_H$  (500 MHz, CHCl<sub>3</sub>) 8.28 (1 H, brs, ArH), 8.07 (1 H, dd, *J* 7.0, 2.0, ArH), 7.45 (1 H, dd, *J* 8.0, 2.0, ArH), 7.85-7.81 (3 H, m, ArH), 7.69 (1 H, d, *J* 8.0, ArH), 7.32-7.28 (1 H, m, ArH), 7.15-7.05 (4 H, m, ArH), 6.77 (2 H, d, *J* 10.0, ArH), 4.55-4.45 (4 H, m, OCH<sub>2</sub>), 4.30-4.20 (2 H, m, OCH<sub>2</sub>), 3.73 (2 H, t, *J* 6.5, NCH<sub>2</sub>), 3.52 (2 H, q, *J* 6.5, CH<sub>2</sub>CH<sub>3</sub>), 1.15 (3 H, t, *J* 7.0, CH<sub>3</sub>);  $\delta_C$  (125 MHz, CDCl<sub>3</sub>) 170.9 (C), 158.9 (C), 154.5 (C), 153.1 (C), 151.8 (C), 147.2 (C), 144.5 (C), 136.3 (C), 134.8 (C), 133.9 (C), 133.0 (CH), 131.7 (CH), 130.8 (CH), 129.4 (CH), 127.0 (CH), 126.0 (CH), 125.6 (C), 124.3 (CH), 122.7 (CH), 122.6 (CH), 118.0 (CH), 111.6 (CH), 87.3 (C), 86.1 (C), 63.8 (CH<sub>2</sub>), 63.4 (CH<sub>2</sub>), 61.8 (CH<sub>2</sub>), 48.9 (CH<sub>2</sub>), 45.8 (CH<sub>2</sub>), 12.4 (CH<sub>3</sub>); *m/z* (ES-API+) 611.0 ([M+H]<sup>+</sup>).

**NMR and fluorescence Spectra and X-ray crystallographic data.**

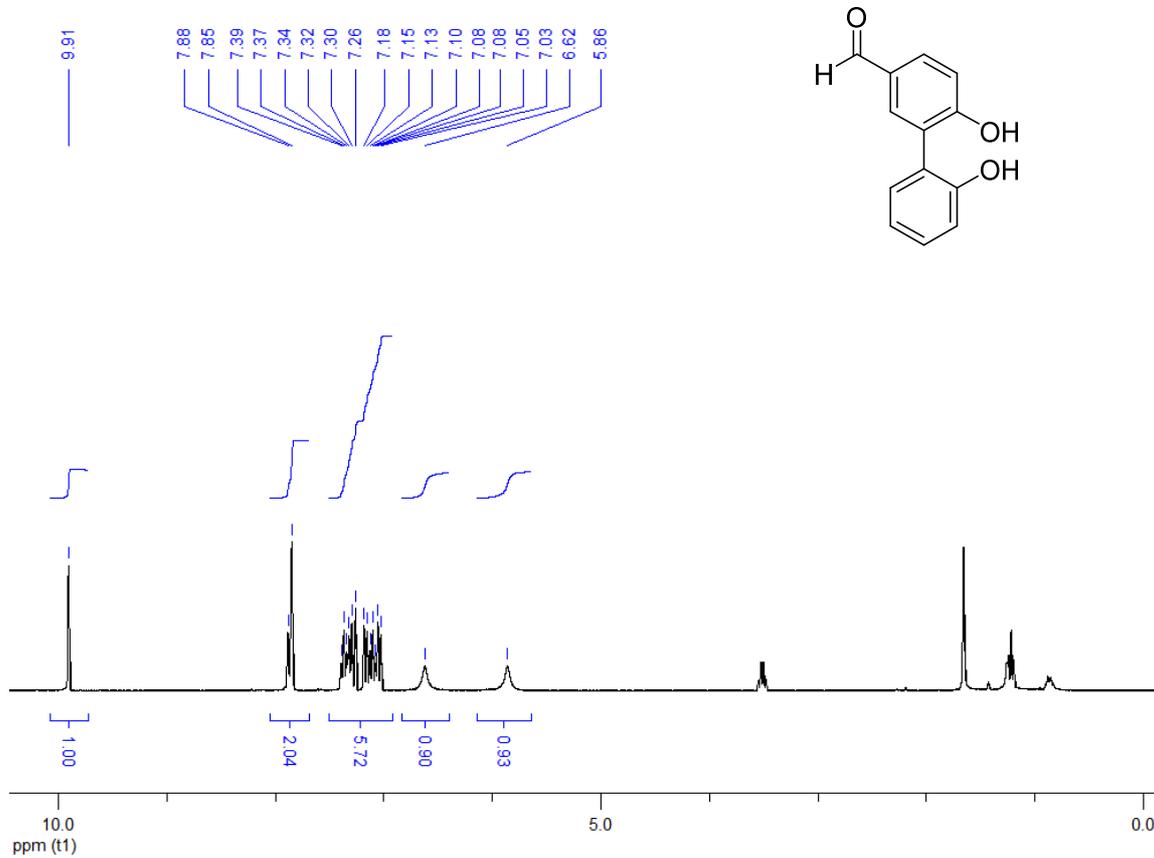
**3-Iodo-4-Hydroxybenzaldehyde 7.**

$\delta_{\text{H}}$  (300 MHz,  $\text{CDCl}_3$ ).



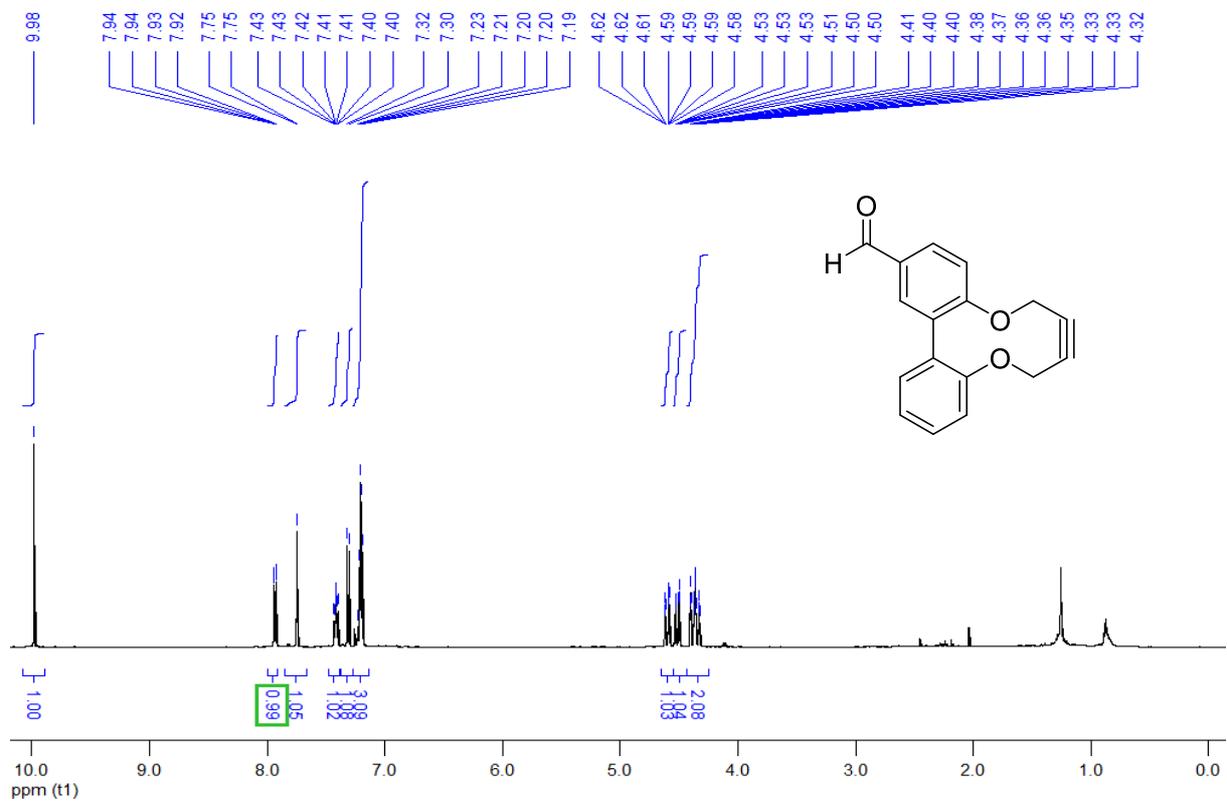
**2',6-Dihydroxybiphenyl-3-carbaldehyde 11.**

$\delta_H$  (300 MHz,  $CDCl_3$ )

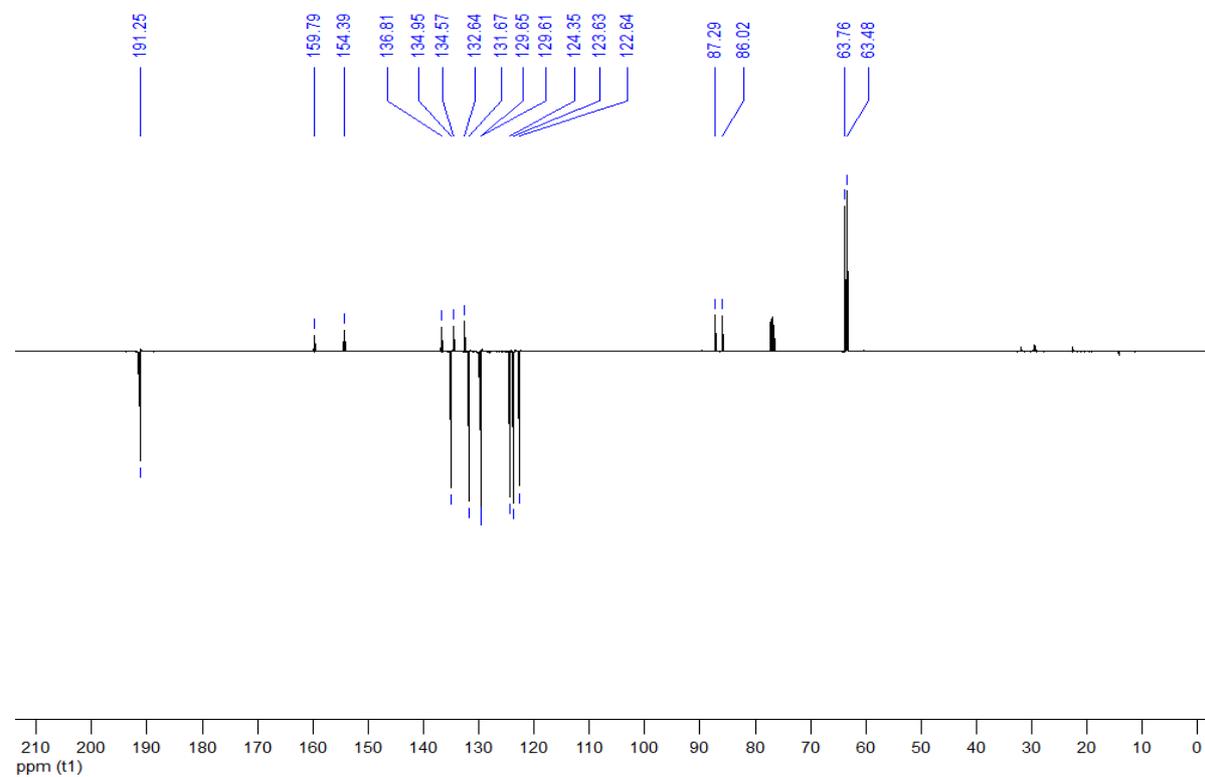


## Alkyne 15

$\delta_H$  (500 MHz,  $CDCl_3$ )

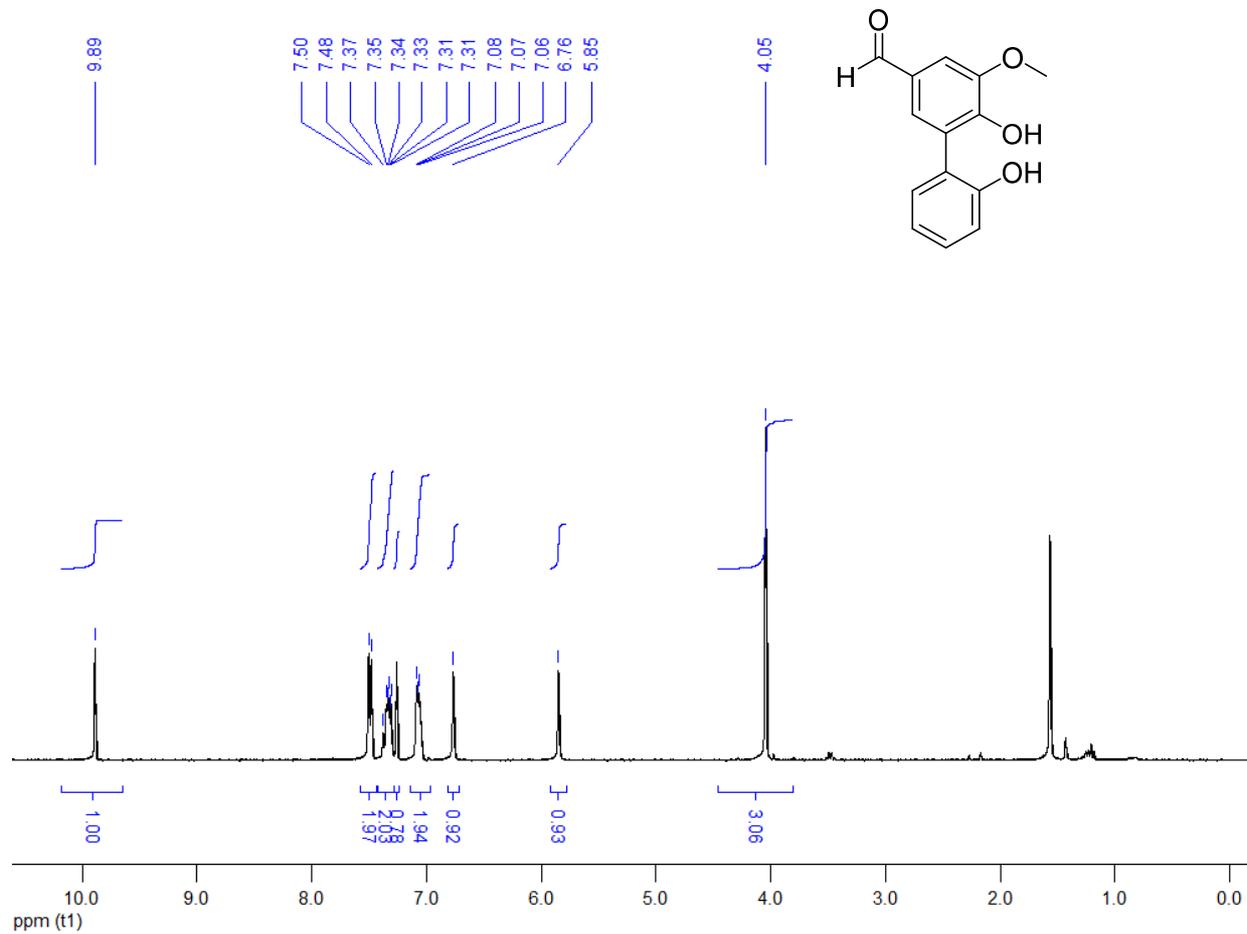


$\delta_C$  (126 MHz,  $CDCl_3$ )



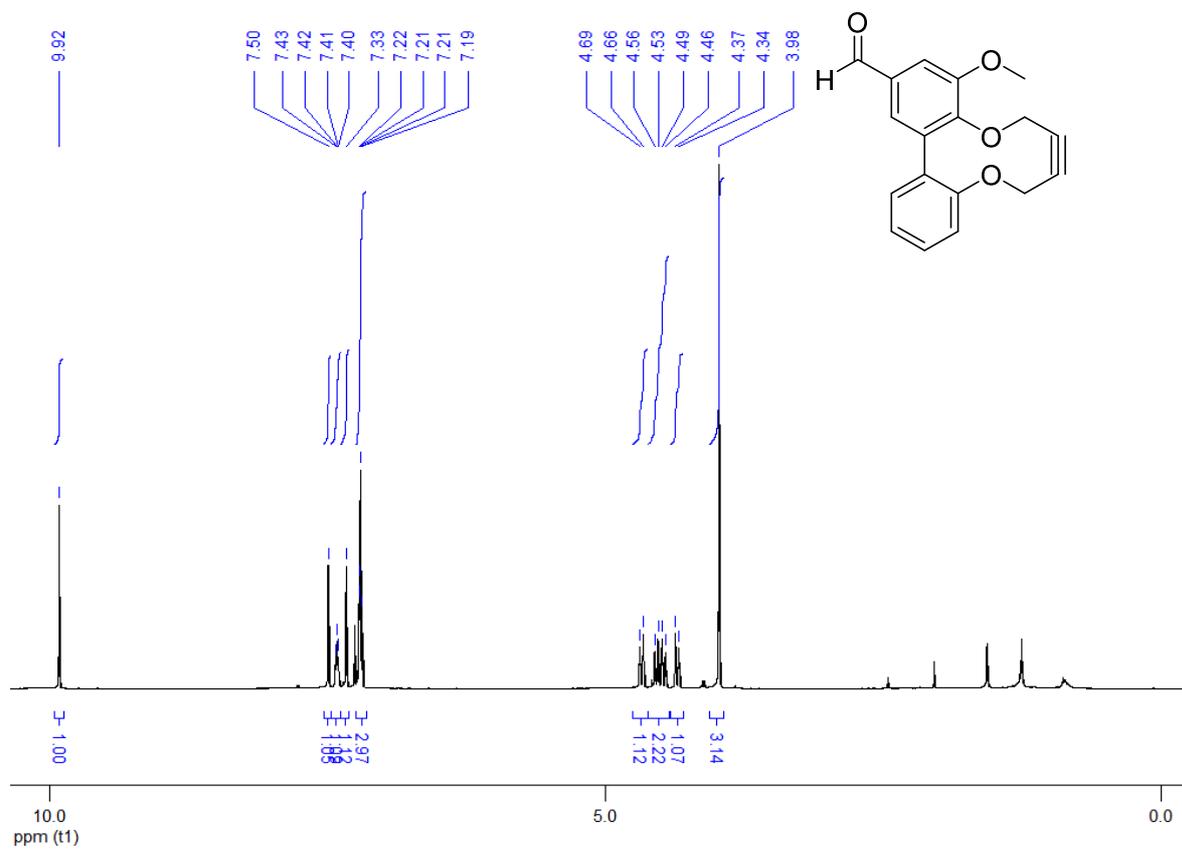
**2',6-Dihydroxy-5-methoxybiphenyl-3-carbaldehyde 12.**

$\delta_H$  (300 MHz,  $CDCl_3$ )

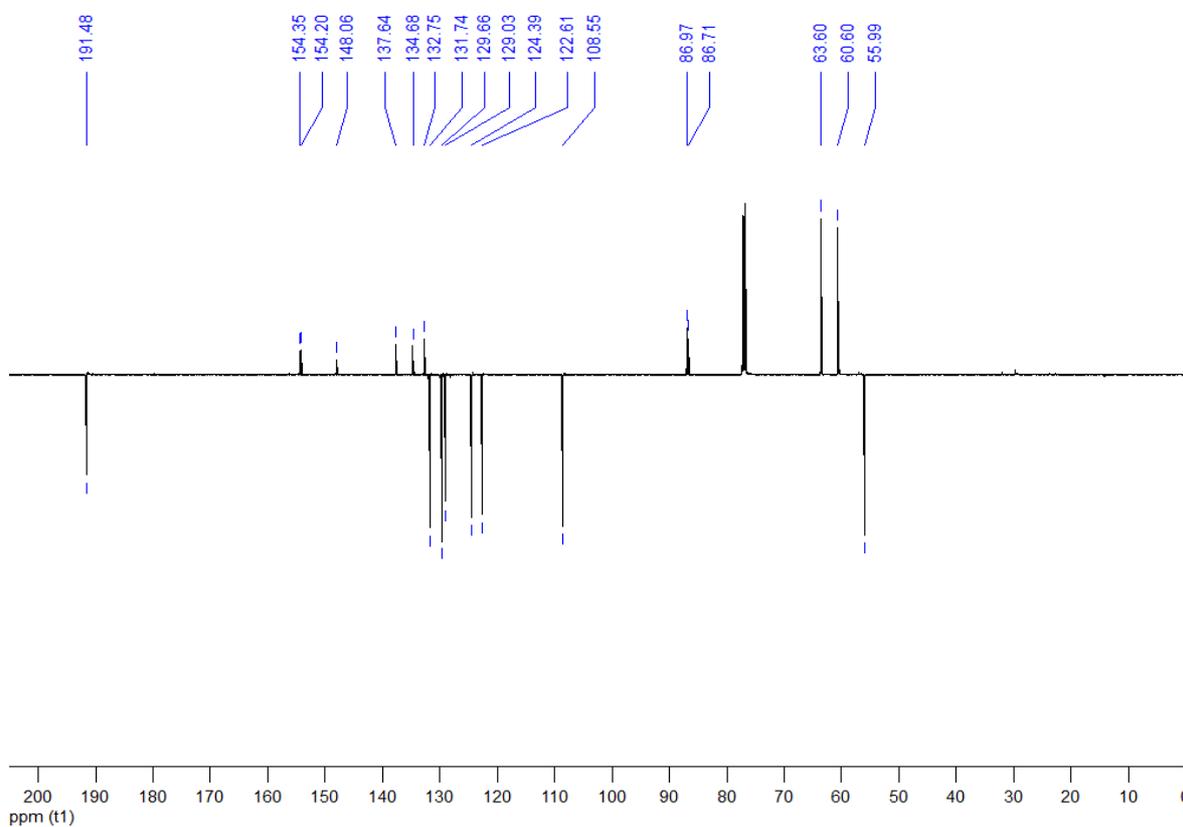


## Alkyne 16

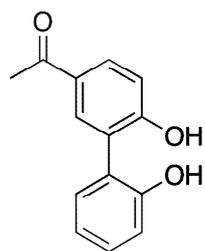
$\delta_H$  (500 MHz,  $CDCl_3$ )



$\delta_C$  (126 MHz,  $CDCl_3$ )



## Diol 13. (ZD10).



$\delta_H$  (400 MHz,  $CDCl_3$ ).

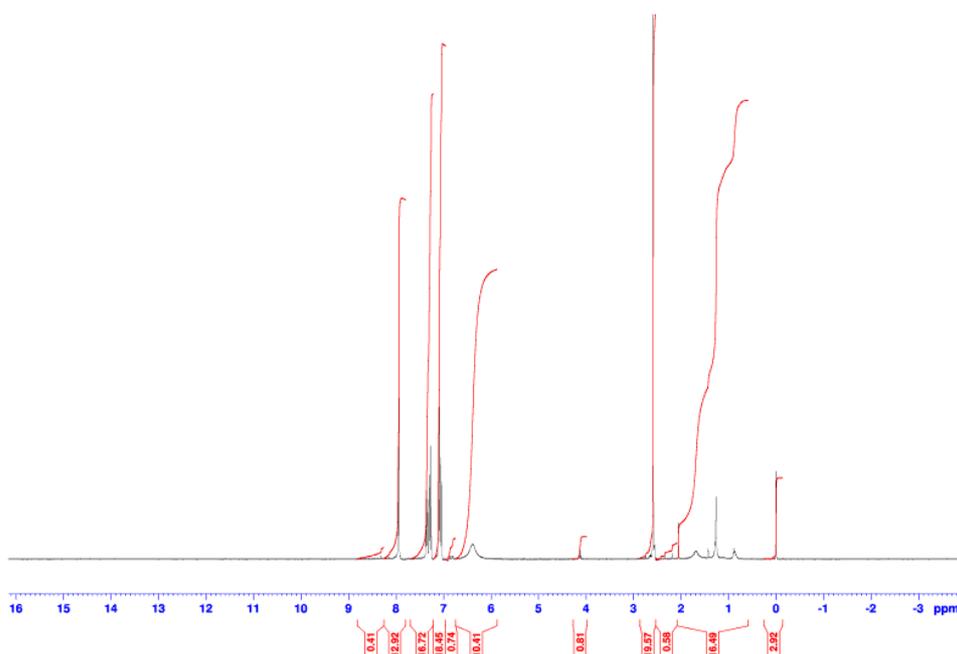
ZD 10 ketone diol  
PROTON.w CDCl3 /opt/topspin3.5pl2 ZD 26



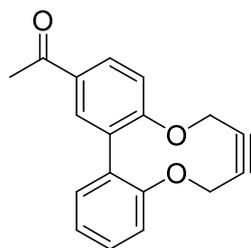
```
Current Data Parameters
NAME      Nov20-2017
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20171120
Time      17.02 h
INSTRUM   spect
PROBHD    zgpg30
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8012.820 Hz
FIDRES     0.122266 Hz
AQ         4.0894465 sec
RG         179.42
DM         62.400 usec
DE         6.50 usec
TE         298.0 K
D1         1.0000000 sec
TDO        1
SFO1      400.1324708 MHz
NUC1       1H
P1         14.00 usec
PLM1      12.92099953 W

F2 - Processing parameters
SI         65536
SF         400.1300182 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
```



## Strained alkyne 17. ZD11.



$\delta_H$  (500 MHz,  $CDCl_3$ ).

Chemist Zak Dualeh  
ZD 11  
PROTON.w CDCI3 /opt/topspin3.2 ZD 31

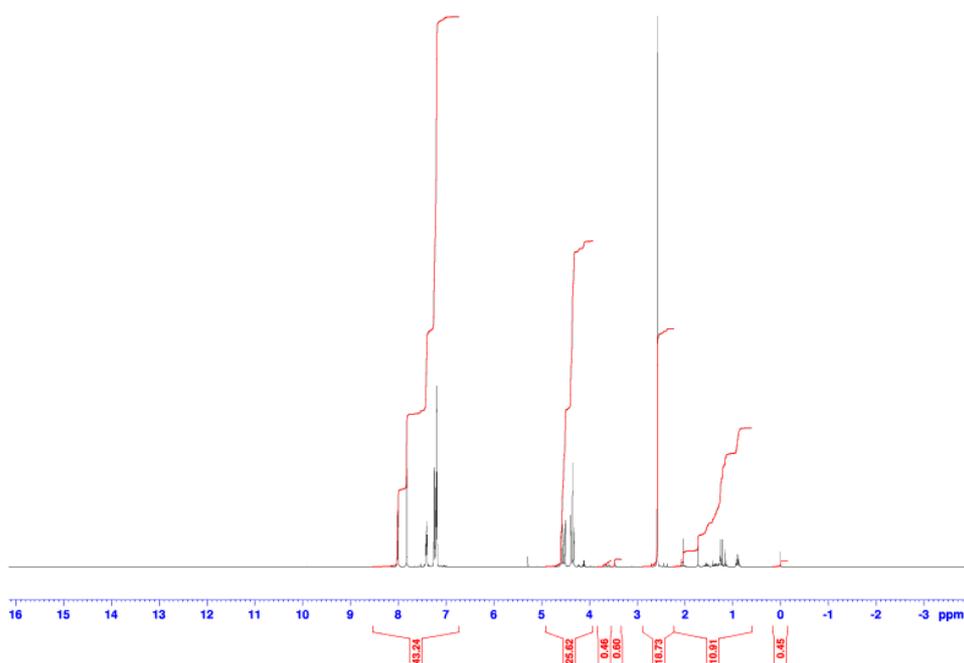


```
Current Data Parameters
NAME      Dec08-2017
EXPNO     10
PROCNO    1

F2 - Acquisition Parameters
Date_     20171208
Time      23.12
INSTRUM   spect
PROBHD    5 mm CPDQ3 13C
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         14
DS         2
SWH        10000.000 Hz
FIDRES     0.1525888 Hz
AQ         3.2767999 sec
RG         47.05
DM         50.000 usec
DE         40.00 usec
TE         298.0 K
D1         1.00000000 sec
TDO        1

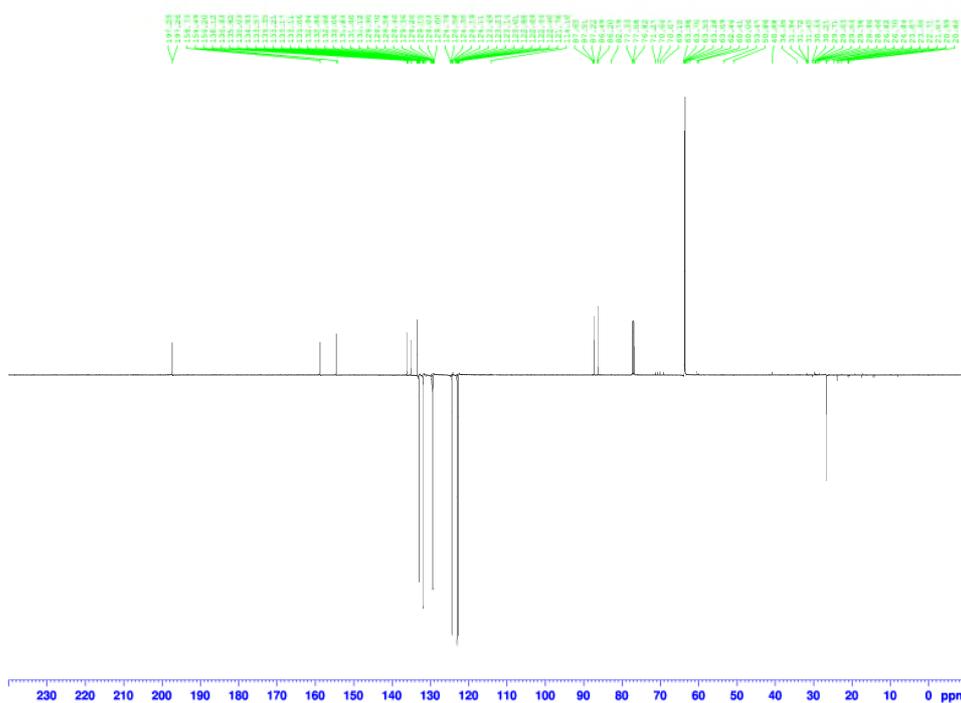
===== CHANNEL f1 =====
SFO1      500.1330885 MHz
NUC1      1H
P1         9.10 usec
PLW1      13.00000000 W

F2 - Processing parameters
SI         65536
SF         500.1300122 MHz
MGM        EM
SGB        0
LB         0.30 Hz
GB         0
PC         1.00
```



$\delta_C$  (126 MHz, CDCl<sub>3</sub>).

Chemist Zak Dualeh  
ZD 11  
C13APT.w CDCl<sub>3</sub> /opt/topspin3.2 ZD 31



```
Current Data Parameters
NAME      Dec08-2017
EXPNO    14
PROCNO   1

F2 - Acquisition Parameters
Date_    20171209
Time     0.16
INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  jmod
TD        65536
SOLVENT  CDCl3
NS        256
DS        4
SWH       32894.738 Hz
FIDRES   0.501934 Hz
AQ        0.9961472 sec
RG        185.92
DW        15.200 usec
DE        66.43 usec
TE        298.0 K
CNST2    145.000000
CNST11   1.000000
D1        2.0000000 sec
D20      0.00689653 sec
TD0       1

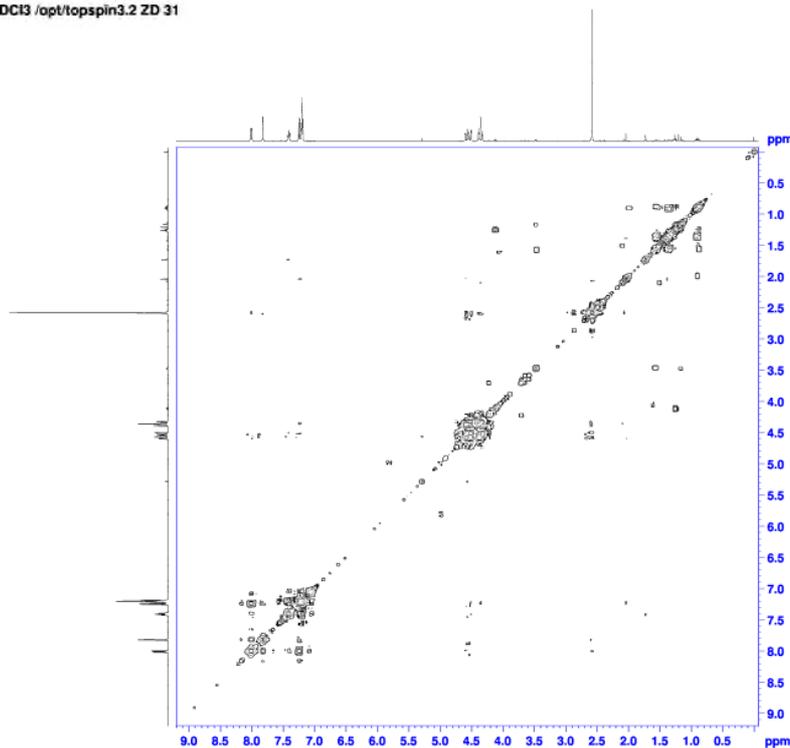
===== CHANNEL f1 =====
SFO1     125.7728795 MHz
NUC1     13C
P1       9.00 usec
P2       19.00 usec
PLW1    24.0000000 W

===== CHANNEL f2 =====
SFO2     500.1320000 MHz
NUC2     1H
CPDPRG2  waltz16
PCPD2    65.00 usec
PLW2    13.0000000 W
PLW12   0.2678400 W

F2 - Processing parameters
SI       32768
SF       125.7727785 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
```

COSY:

Chemist Zak Dualeh  
ZD 11  
COSY.w CDCl<sub>3</sub> /opt/topspin3.2 ZD 31



```
Current Data Parameters
NAME      Dec08-2017
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20171208
Time     23.14
INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  waltz16
SOLVENT  CDCl3
NS        1
DS        4
SWH       4638.219 Hz
FIDRES   2.264755 Hz
AQ        0.207716 sec
RG        34.43
DW        107.800 usec
DE        40.00 usec
TE        298.0 K
D0        0.0000300 sec
D1        1.8077897 sec
D11       0.0300000 sec
D12       0.0000200 sec
D13       0.0000040 sec
D14       0.0000000 sec
IN0       0.0002150 sec

===== CHANNEL f1 =====
SFO1     500.132262 MHz
NUC1     1H
P1       9.10 usec
P2       9.10 usec
P12      2500.00 usec
PLW1    13.0000000 W
PLW12   1.3284893 W

===== GRABF2 CH CHANNEL =====
GRAMM[1] SMOG10.100
WEIC     10.00 %
P16      1000.00 usec

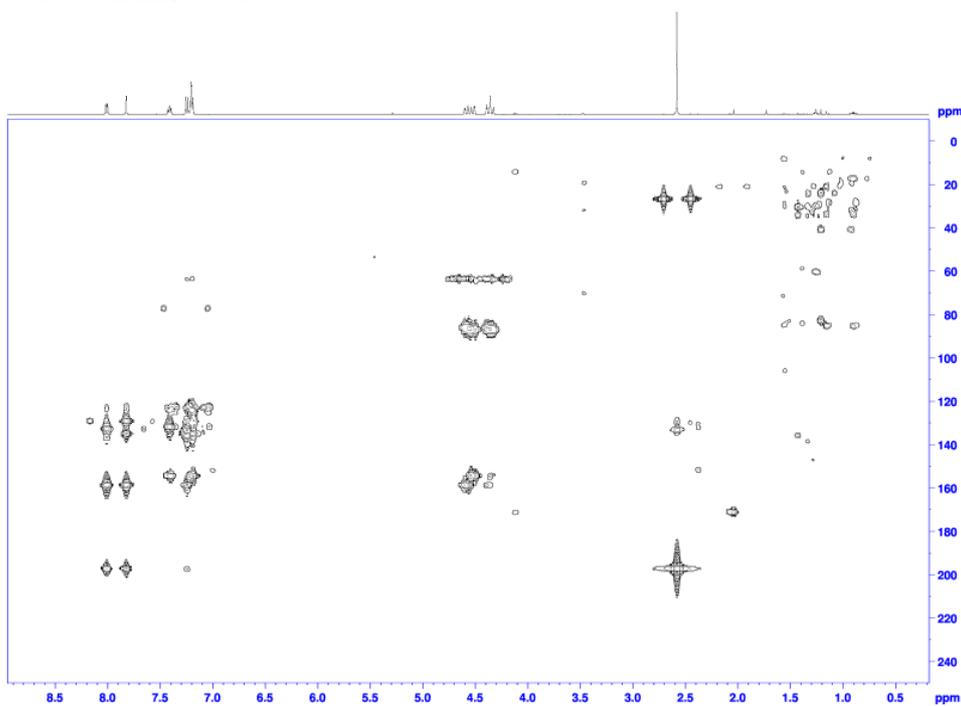
F1 - Acquisition parameters
TD        65536
SFO1     500.1323 MHz
FIDRES   36.202053 Hz
SK        9.245 ppm
FANGRES  0

F2 - Processing parameters
SI       1024
SF       500.1301122 MHz
WDW      QDINE
SSB      0
LB       0 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1024
NUC1     13C
SF       500.1301122 MHz
WDW      QDINE
SSB      0
LB       0 Hz
GB       0
```

# HMBC:

Chemist Zak Dualeh  
ZD 11  
HMBC.w CDCl3 /opt/topspin3.2 ZD 31



```

Current Data Parameters
NAME      hmc08-2011
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20110908
Time     23.24
INSTRUM  spect
PROBHD   5 mm CPDQX 13C
PULPROG  hmcopzgpg04
TD        1024
SOLVENT  CDCl3
NS        8
DS        4
SWH       4380.940 Hz
FIDRES    4.283049 Hz
AQ        0.1147940 sec
RG         655
AQ        0.1147940 sec
RG         655
TE        300.2 K
CMT02    145.0000000
D0        0.0000000 sec
D1        1.42948000 sec
D2        0.00348828 sec
D3        0.0000000 sec
D16       0.0000000 sec
TMS       0.0000000 sec

===== CHANNEL f1 =====
NUC1      13C
MPC1      18
P1         9.10 usec
P2         18.20 usec
===== CHANNEL f2 =====
NUC2      1H
MPC2      2
P2         24.0000000 usec

===== GRADIENT CHANNEL =====
GPMAX[1]  SMG10.100
GPMAX[2]  SMG10.100
GPMAX[3]  SMG10.100
CPL1      20.00 %
CPL2      20.00 %
CPL3      40.00 %
P15       1000.00 usec

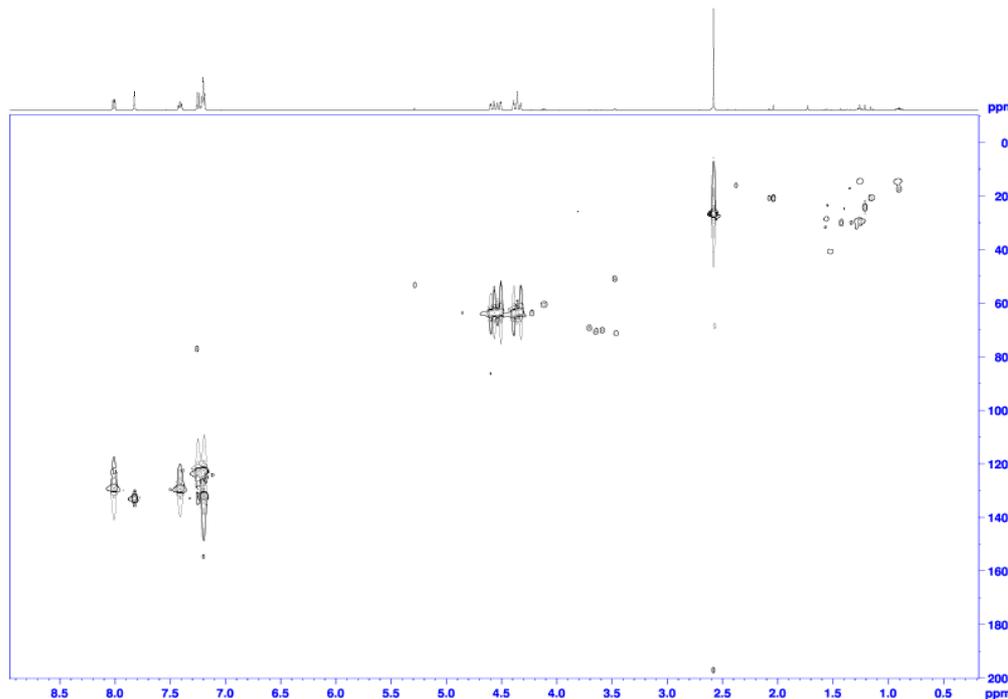
F1 - Acquisition parameters
SI         2
SF         125.7725 MHz
FIDRES    200.30465 Hz
SW         250.813 ppm
NUC1       13C

F2 - Processing parameters
SI         2
SF         500.136312 MHz
WDW        0
SSB        0
LB         0 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MPC1      18
P1         9.10 usec
SF         125.7725 MHz
WDW        0
SSB        0
LB         0 Hz
GB         0
PC         1.40
    
```

# HSQC:

Chemist Zak Dualeh  
ZD 11



```

Current Data Parameters
NAME      hmc08-2011
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20110908
Time     23.19
INSTRUM  spect
PROBHD   5 mm CPDQX 13C
PULPROG  hmcopzgpg04
TD        1024
SOLVENT  CDCl3
NS        8
DS        4
SWH       4380.940 Hz
FIDRES    4.283049 Hz
AQ        0.1147940 sec
RG         655
AQ        0.1147940 sec
RG         655
TE        300.2 K
CMT02    145.0000000
D0        0.0000000 sec
D1        1.42948000 sec
D2        0.00348828 sec
D3        0.0000000 sec
D16       0.0000000 sec
TMS       0.0000000 sec

===== CHANNEL f1 =====
NUC1      13C
MPC1      18
P1         9.10 usec
P2         18.20 usec
===== CHANNEL f2 =====
NUC2      1H
MPC2      2
P2         24.0000000 usec

===== GRADIENT CHANNEL =====
GPMAX[1]  SMG10.100
GPMAX[2]  SMG10.100
GPMAX[3]  SMG10.100
CPL1      20.00 %
CPL2      20.00 %
CPL3      40.00 %
P15       1000.00 usec

F1 - Acquisition parameters
SI         2
SF         125.7697 MHz
FIDRES    200.30465 Hz
SW         250.813 ppm
NUC1       13C

F2 - Processing parameters
SI         2
SF         500.136312 MHz
WDW        0
SSB        0
LB         0 Hz
GB         0
PC         1.40

F1 - Processing parameters
SI         1024
MPC1      18
P1         9.10 usec
SF         125.7697 MHz
WDW        0
SSB        0
LB         0 Hz
GB         0
PC         1.40
    
```

**Single crystal X-ray crystallographic structure of 16**

CCDC 1852221.

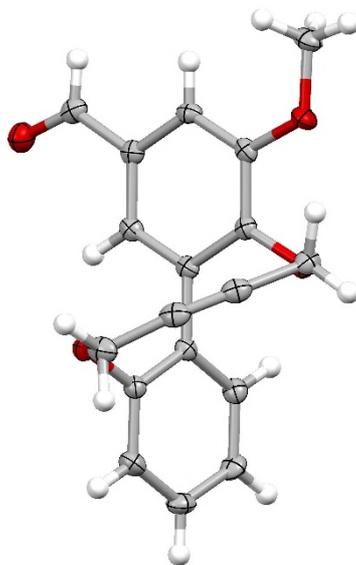


Figure 1: Single crystal X-ray structure of **16** (ellipsoids are plotted at the 50% probability level)

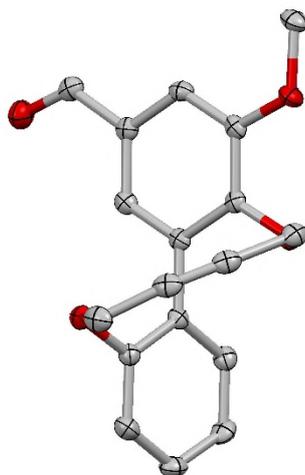


Figure 2: Single crystal X-ray structure of **16** (ellipsoids are plotted at the 50% probability level)

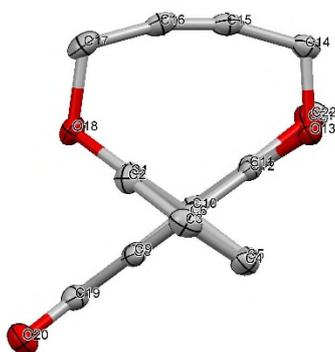


Figure 3: Single crystal X-ray structure of **16** (ellipsoids are plotted at the 50% probability level, hydrogen atoms are omitted for clarity)

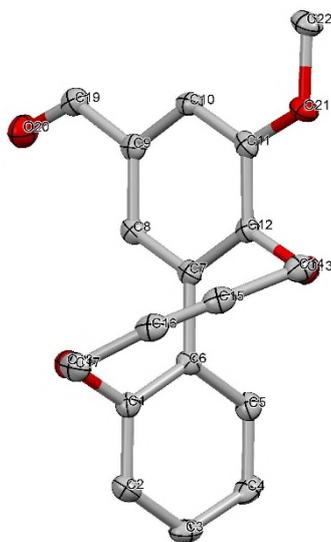


Figure 4: Single crystal X-ray structure of **16** (ellipsoids are plotted at the 50% probability level, hydrogen atoms are omitted for clarity)

CCDC 1852221 contains the supplementary crystallographic data for this paper. These can be obtained free of charge from the Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

Single crystals of **16** were grown from vapour diffusion of *n*-hexane into a chloroform solution of the compound over several days. A suitable crystal was selected and mounted on a glass fibre with Fomblin oil and placed on a Rigaku Oxford Diffraction SuperNova diffractometer with a dual source (Cu at zero) equipped with an AtlasS2 CCD area detector at 150(2) K. The structure was solved using Olex2<sup>1</sup> and the ShelXT<sup>2</sup> structure solution program using Direct Methods and refined with the ShelXL<sup>3</sup> refinement package using Least Squares refinement.

The asymmetric unit contains one crystallographically distinct molecule, with *Z* = 4. The crystal packing indicates aromatic donor-acceptor ( $\pi$ - $\pi$ ) interactions of the biphenyl moieties of adjacent molecules. The alkyne group displays significant deviation from linearity, the C14-C15-C16 and C15-C16-C17 bond angles are 165.23(13)° and 165.32(13)° respectively. This is accompanied by a large biphenyl torsion angle (C5-C6-C7-C8) of 72.24(15)°.

**Table 1:** Single-crystal X-ray data for compound **16**.

Compound Reference	Compound 16
Chemical Formula	C <sub>18</sub> H <sub>14</sub> O <sub>4</sub>
Formula Mass	294.29
Crystal system	Monoclinic
<i>a</i> / Å	10.80656(8)
<i>b</i> / Å	17.94783(10)
<i>c</i> / Å	7.75427(6)
$\alpha$ / °	90
$\beta$ / °	110.5855(8)
$\gamma$ / °	90
Unit cell volume/ Å <sup>3</sup>	1407.942(18)
Temperature/ K	150(2) K
Space group	- <i>P</i> 2 <i>yc</i>
Crystal size/ mm	0.12 × 0.40 × 0.40
Radiation	Cu K $\alpha$
Goodness-of-fit on F <sup>2</sup>	1.052
No. of formula units per unit cell, Z	4
No. of reflections measured	27406
No. of independent reflections	2829
Final R <sub>1</sub> vaules ( <i>I</i> > 2 $\sigma$ ( <i>I</i> ))	0.0451
Final <i>wR</i> (F <sup>2</sup> ) values ( <i>I</i> > 2 $\sigma$ ( <i>I</i> ))	0.1206
Final <i>R</i> <sub>I</sub> values (all data)	0.0459
Final <i>wR</i> (F <sup>2</sup> ) (all data)	0.1219

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Sheldrick, G.M. (2015). Acta Cryst. A71, 3-8.
3. Sheldrick, G.M. (2015). Acta Cryst. C71, 3-8

**X-ray crystallographic structure of ketone 17.**

**CCDC 1852222**

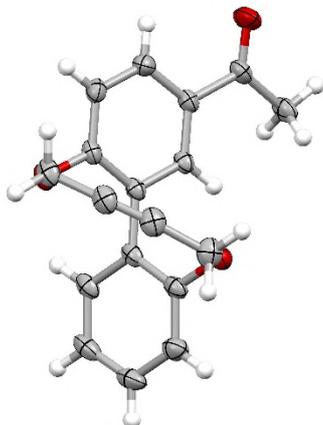


Figure 1: Single crystal X-ray structure of **17** (ellipsoids are plotted at the 50% probability level)

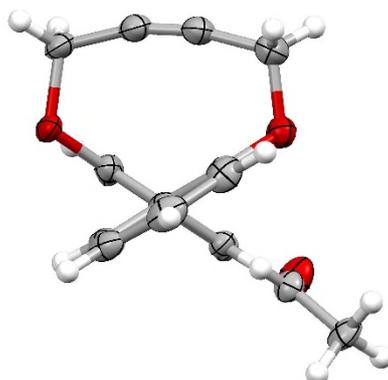


Figure 2: Single crystal X-ray structure of **17** (ellipsoids are plotted at the 50% probability level)

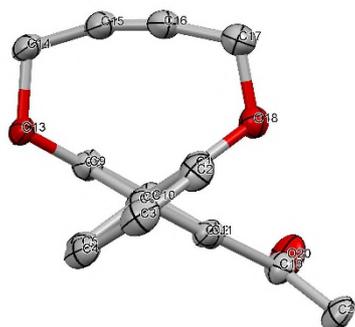


Figure 3: Single crystal X-ray structure of **17** (hydrogen atoms are omitted for clarity; ellipsoids are plotted at the 50% probability level)

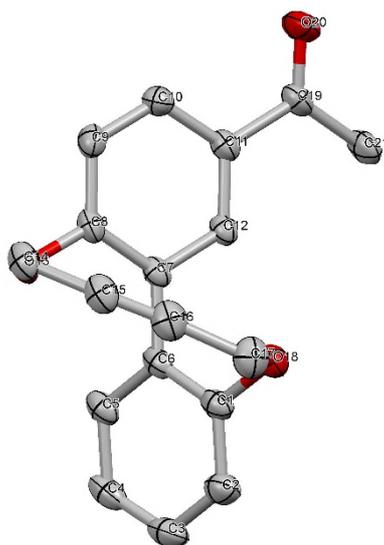


Figure 4: Single crystal X-ray structure of **17** (ellipsoids are plotted at the 50% probability level, hydrogen atoms are omitted for clarity)

**CCDC 1852222** contains the supplementary crystallographic data for this paper. These can be obtained free of charge from the Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

Single crystals of **X** were grown from vapour diffusion of *n*-hexane into a chloroform solution of the compound over several days. A suitable crystal was selected and mounted on a glass fibre with Fomblin oil and placed on a Rigaku Oxford Diffraction SuperNova diffractometer with a dual source (Cu at zero) equipped with an AtlasS2 CCD area detector at 150(2) K. The structure was solved using Olex2<sup>1</sup> and the ShelXT<sup>2</sup> structure solution program using Direct Methods and refined with the ShelXL<sup>3</sup> refinement package using Least Squares refinement.

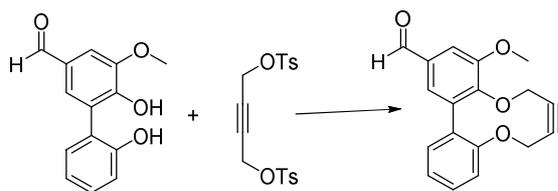
The asymmetric unit contains one crystallographically distinct molecules, with *Z* = 8. The crystal packing indicates aromatic donor-acceptor ( $\pi$ - $\pi$ ) interactions of the biphenyl moieties of adjacent molecules. The alkyne group displays significant deviation from linearity, the C14-C15-C16 and C15-C16-C17 bond angles are 165.1298(6)° and 168.5519(3)° respectively. This is accompanied by a large biphenyl torsion angle (C5-C6-C7-C8) of 66.378(2)°.

**Table 1:** single-crystal X-ray data for compound **17**.

Compound Reference	Compound 17
Chemical Formula	C <sub>18</sub> H <sub>14</sub> O <sub>3</sub>
Formula Mass	278.31
Crystal system	Monoclinic
<i>a</i> / Å	18.8350(8)
<i>b</i> / Å	7.1072(3)
<i>c</i> / Å	21.7952(9)
$\alpha$ / °	90
$\beta$ / °	105.284(4)
$\gamma$ / °	90
Unit cell volume/ Å <sup>3</sup>	2814.4(2)
Temperature/ K	150(2) K
Space group	<i>I</i> 1 2/ <i>a</i> 1
Crystal size/ mm	0.12 × 0.15 × 0.20
Radiation	Cu K $\alpha$
Goodness-of-fit on F <sup>2</sup>	1.0443
No. of formula units per unit cell, Z	8
No. of reflections measured	14083
No. of independent reflections	2812
Final R <sub>1</sub> values ( <i>I</i> > 2 $\sigma$ ( <i>I</i> ))	0.0508
Final <i>wR</i> (F <sup>2</sup> ) values ( <i>I</i> > 2 $\sigma$ ( <i>I</i> ))	0.1342
Final <i>R<sub>I</sub></i> values (all data)	0.0559
Final <i>wR</i> (F <sup>2</sup> ) (all data)	0.1432

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H. (2009), *J. Appl. Cryst.* 42, 339-341.
2. Sheldrick, G.M. (2015). *Acta Cryst.* A71, 3-8.
3. Sheldrick, G.M. (2015). *Acta Cryst.* C71, 3-8

## Conversion /time for cyclisation to form aldehyde strained alkyne **16**:



Conditions for HPLC: This reaction was also monitored by HPLC over 14 days, using a Chiralcel IB column, 15:85 IPA:Hexane, 1ml/min.

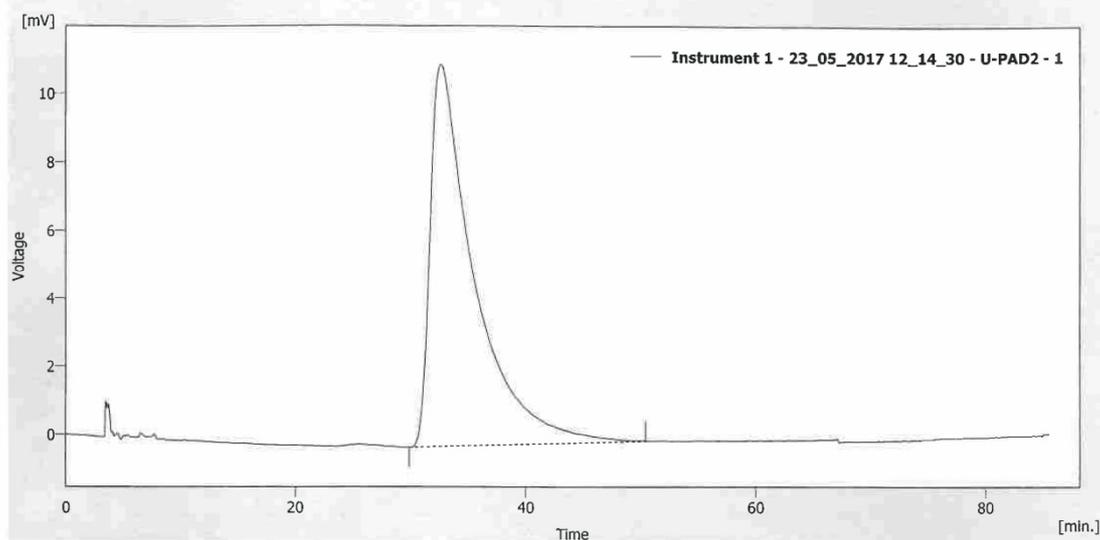
### Starting material:

23/05/2017 14:18

Chromatogram C:\Clarity\WORK2\DATA\Instrument 1 - 23\_05\_2017 12\_14\_30.PRM

Page 1 of 1

 **Clarity - Chromatography SW**  
DataApex 2006  
www.dataapex.com



Result Table (Uncal - Instrument 1 - 23\_05\_2017 12\_14\_30 - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	32.576	2973.594	11.191	100.0	100.0	3.55	
	Total	2973.594	11.191	100.0	100.0		

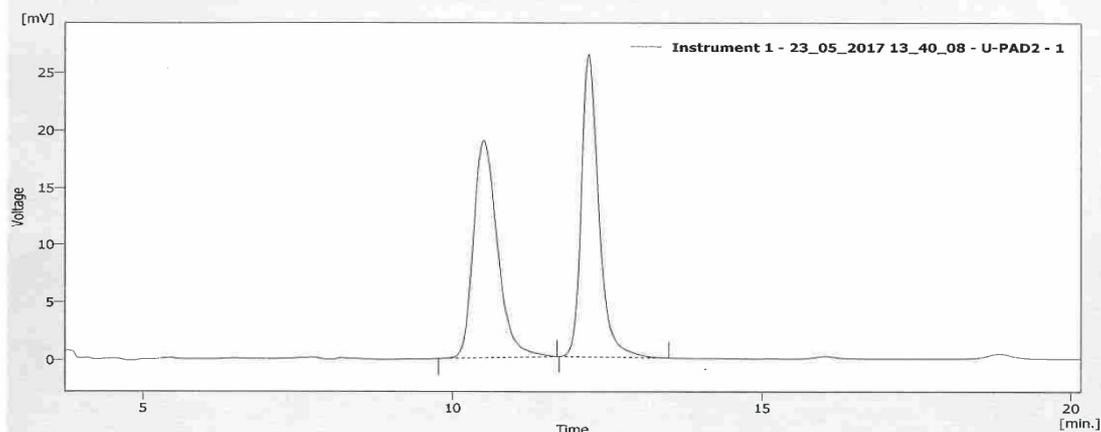
## Racemic product (purified):

23/05/2017 14:18

Chromatogram C:\Clarity\Work2\DATA\Instrument 1 - 23\_05\_2017 13\_40\_08.PRM

Page 1 of 1

**Clarity - Chromatography SW**  
DataApex 2006  
www.dataapex.com



Result Table (Uncal - Instrument 1 - 23\_05\_2017 13\_40\_08 - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	10.496	511.238	18.969	49.9	41.8	0.41	
2	12.200	513.296	26.392	50.1	58.2	0.28	
	Total	1024.534	45.360	100.0	100.0		

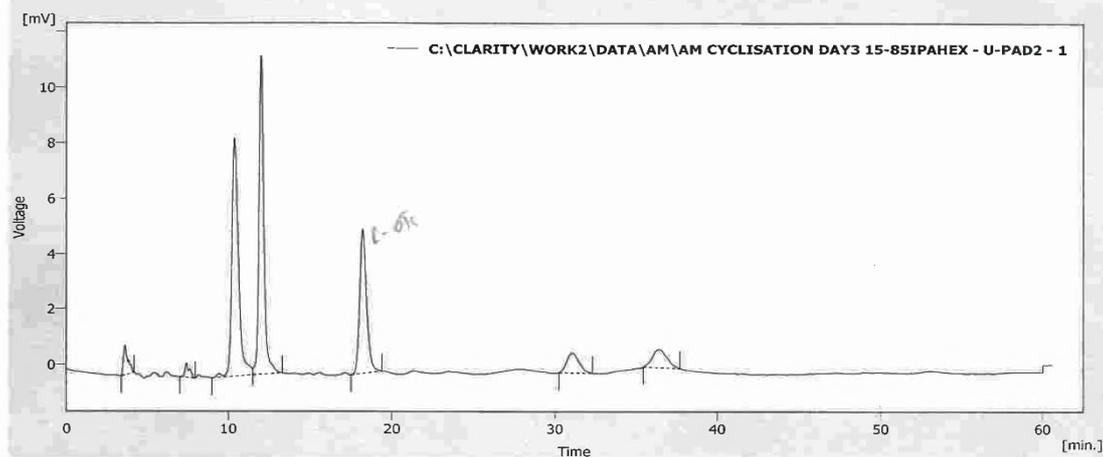
**Cyclisation on day 3** (crude mixture; miniworkup using EtOAc/NaHCO<sub>3</sub>): Products are at 10.4 and 12.0 min., peak at 18.2 min is ditosylate. Uncyclised is at 31.1 min. Unknown impurity at 36.4 min. uv detector and therefore non-quantitative.

26/05/2017 12:28

Chromatogram C:\CLARITY\WORK2\DATA\AM\AM CYCLISATION DAY3 15-85IPAHEX.PRM

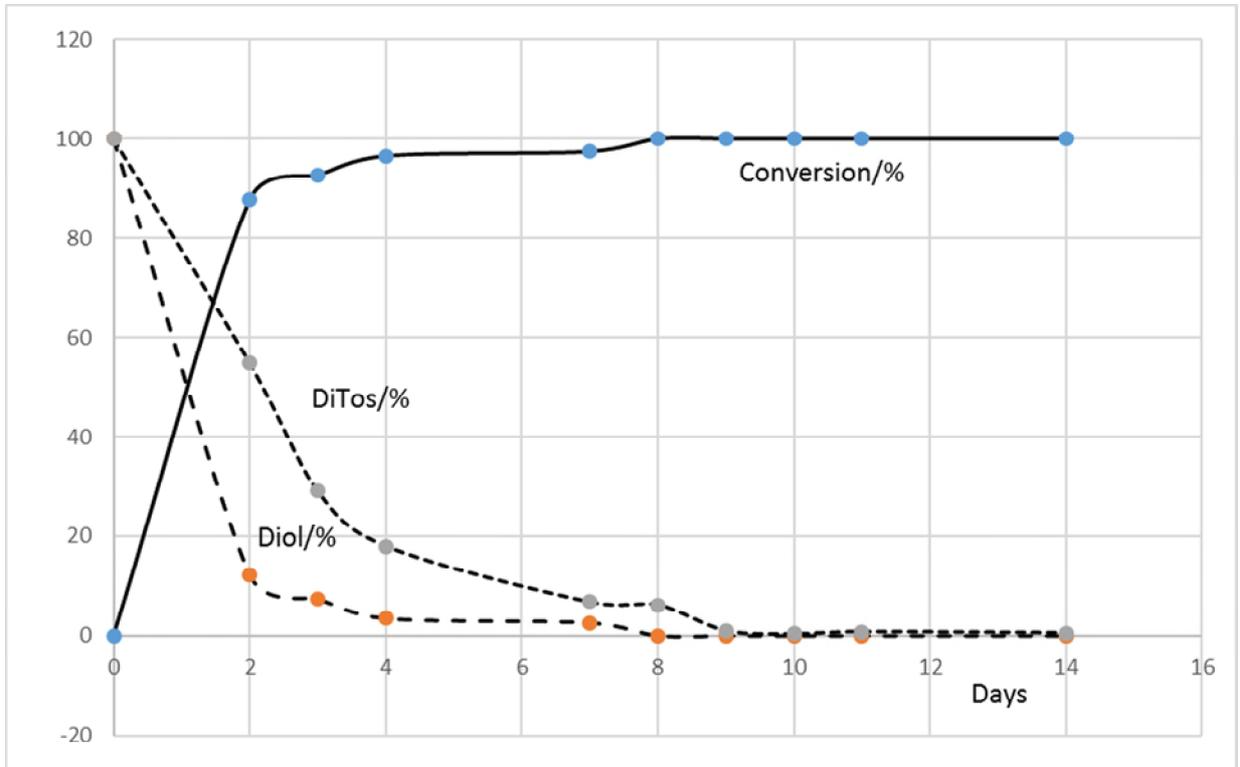
Page 1 of 1

**Clarity - Chromatography SW**  
DataApex 2006  
www.dataapex.com



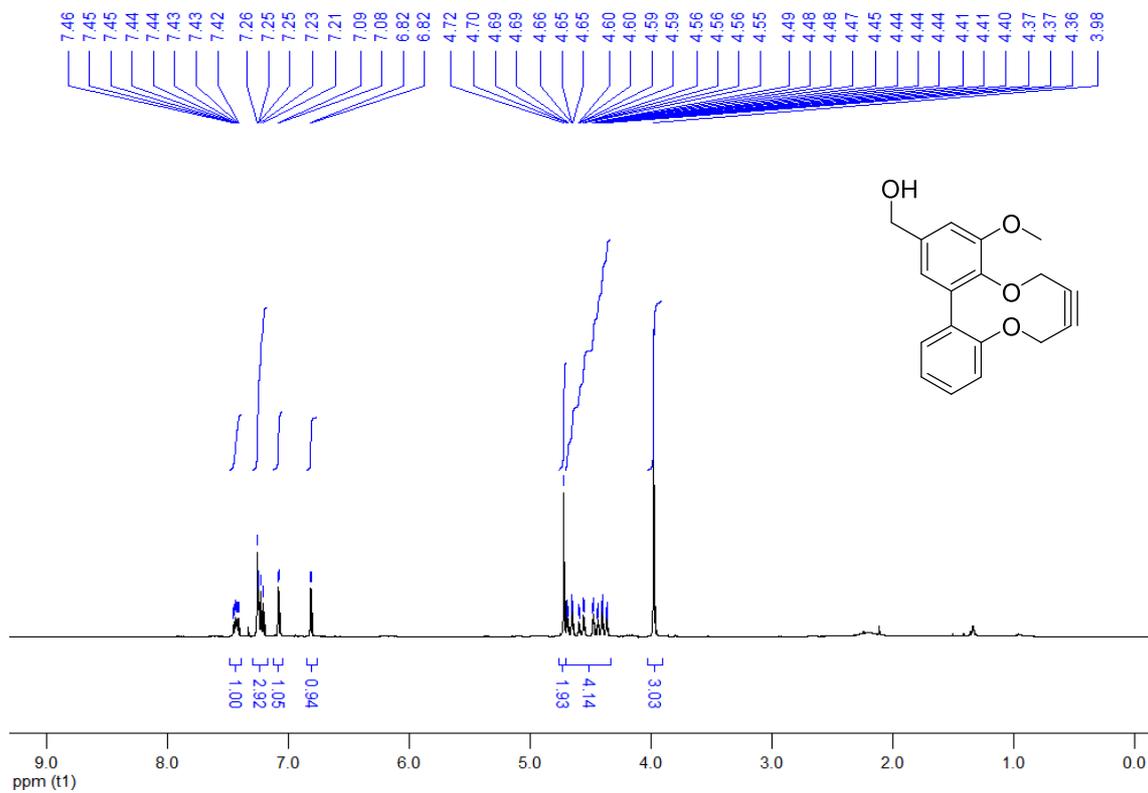
Result Table (Uncal - C:\CLARITY\WORK2\DATA\AM\AM CYCLISATION DAY3 15-85IPAHEX - U-PAD2 - 1)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]	Compound Name
1	3.620	20.778	1.056	2.8	3.7	0.28	
2	7.428	10.141	0.513	1.4	1.8	0.20	
3	10.376	245.835	8.574	33.2	30.4	0.41	
4	12.012	232.705	11.484	31.5	40.7	0.29	
5	18.216	150.888	5.216	20.4	18.5	0.44	
6	31.096	38.174	0.720	5.2	2.6	0.83	
7	36.404	41.106	0.650	5.6	2.3	1.01	
	Total	739.627	28.214	100.0	100.0		

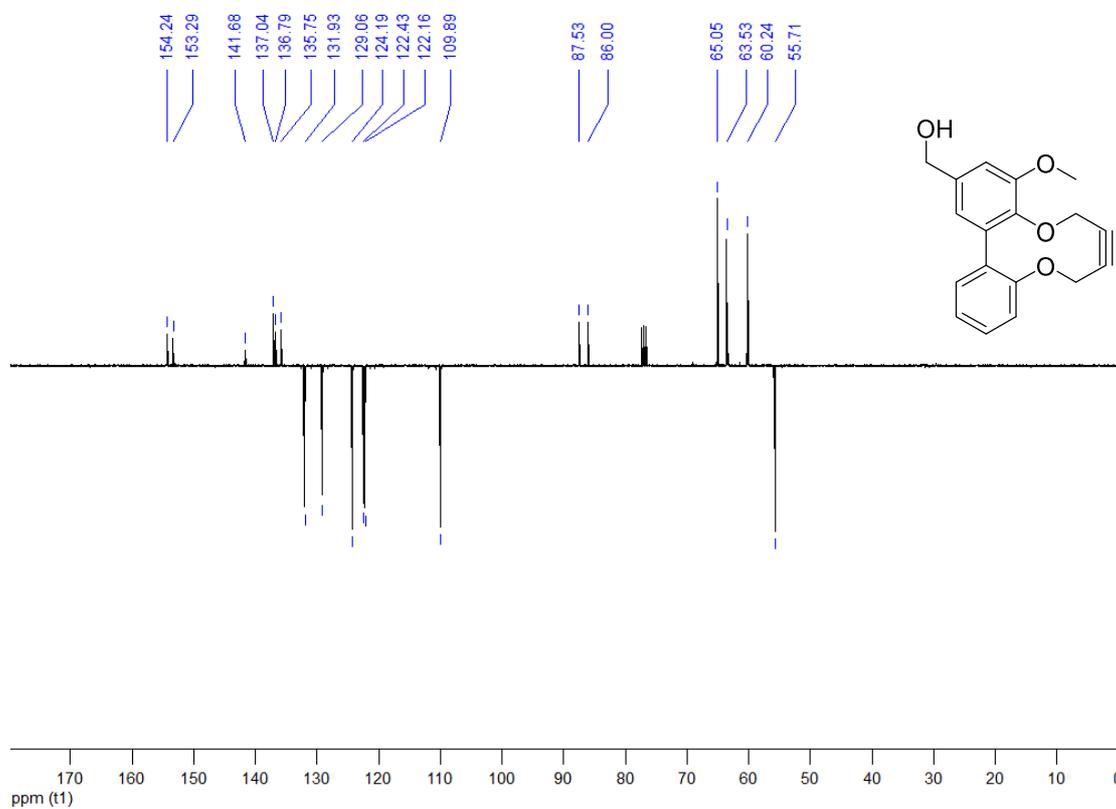


## Alkyne alcohol 19

$\delta_H$  (400 MHz,  $CDCl_3$ )



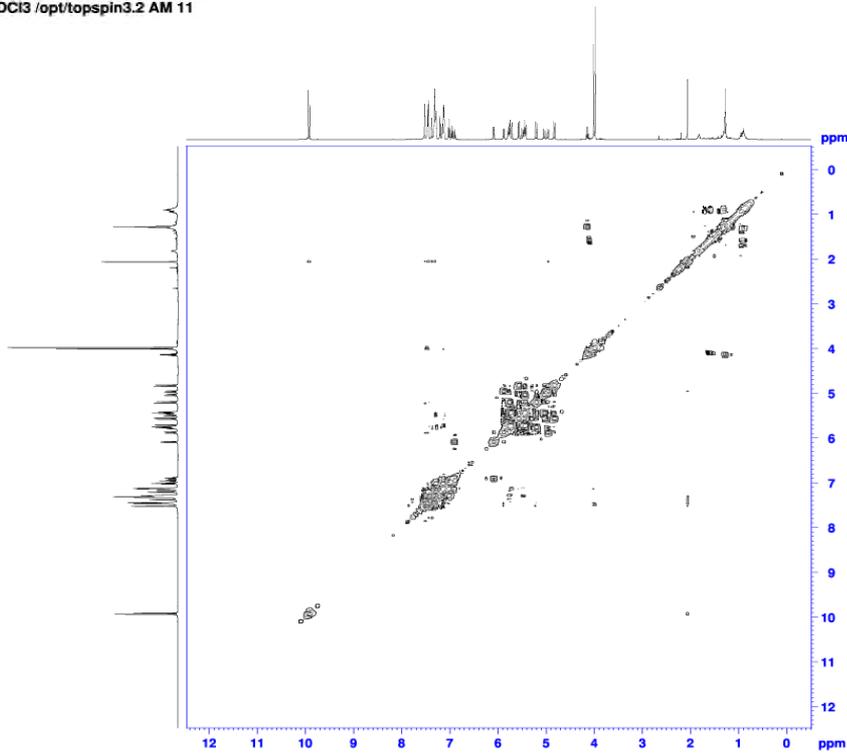
$\delta_C$  (100 MHz,  $CDCl_3$ )





# COSY:

Chemist Anish Mistry  
 AM-63 pure  
 COSY.w CDC13 /opt/topspin3.2 AM 11



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EXPNO    11
PROCNO   1

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PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        2
DS        4
SWH       6493.506 Hz
FIDRES   0.1576960 sec
AQ        0.1576960 sec
RG        312.85
DM        77.000 usec
DE        0.0000000 sec
TE        298.2 K
DD        0.0000000 sec
D1        1.9871203 sec
D11       0.0300000 sec
D12       0.0000000 sec
D13       0.0000000 sec
D14       0.0000000 sec
INO       0.00015380 sec

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NUC1     13C
P0        9.10 usec
P1        9.10 usec
P17       2500.00 usec
PL1       13.00000000 W
PL12      1.3394997 W

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GPA1     10.00 %
P16      1000.00 usec

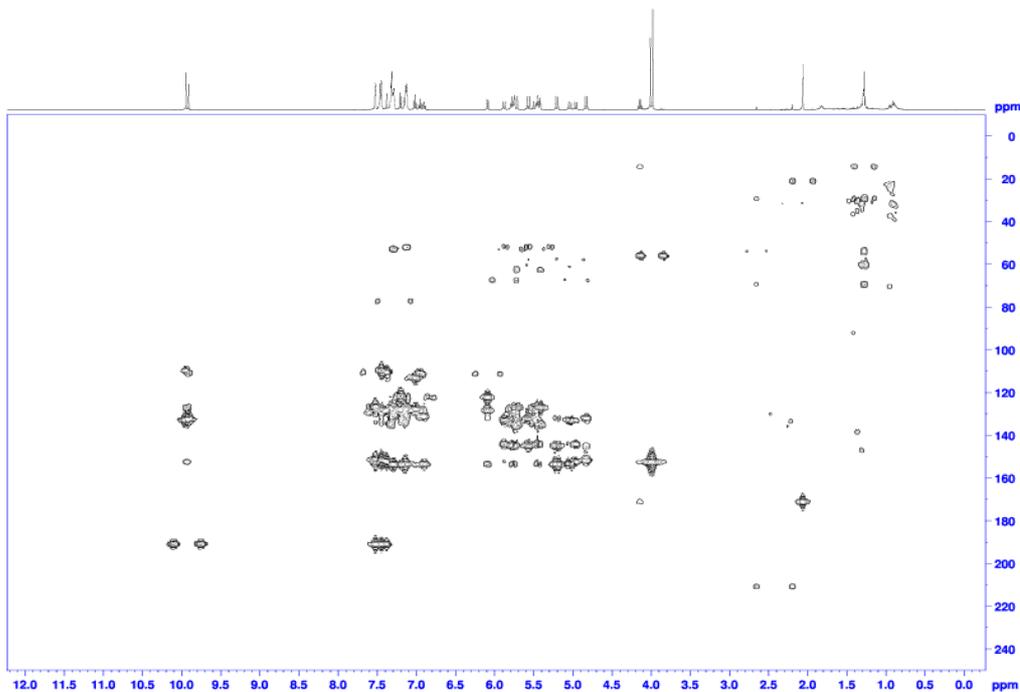
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FREQ0    13.00000000 ppm

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SF        500.1300000 MHz
SF0       0
GB        0 Hz
GB0       0 Hz
PC        1.40

F1 - Processing parameters
SI        32768
SF        500.1300000 MHz
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GB0       0 Hz
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# HMBC:

Chemist Anish Mistry  
 AM-63 pure  
 HMBC.w CDC13 /opt/topspin3.2 AM 11



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EXPNO    13
PROCNO   1

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INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  hmcop1p2degf
TD        65536
SOLVENT  CDCl3
NS        2
DS        4
SWH       6200.000 Hz
FIDRES   0.1581016 sec
AQ        0.1581016 sec
RG        312.85
DM        80.000 usec
DE        0.0000000 sec
TE        298.2 K
DD        10.0000000 sec
D1        0.0000000 sec
D2        1.49380000 sec
D3        0.00000000 sec
D4        0.00000000 sec
D5        0.00000000 sec
D6        0.00000000 sec
D7        0.00000000 sec
D8        0.00000000 sec
D9        0.00000000 sec
D10       0.00000000 sec

----- CHANNEL f1 -----
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NUC1     13C
P0        9.10 usec
P1        9.10 usec
P17       13.00000000 W

----- CHANNEL f2 -----
SFO2     125.7728195 MHz
NUC2     1H
P2        9.30 usec
P22       24.00000000 W

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GPMAM[3] SMSQ10.100
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GPA3     10.00 %
GPA4     10.00 %
GPA5     10.00 %
GPA6     10.00 %
GPA7     10.00 %
GPA8     10.00 %
GPA9     10.00 %
GPA10    10.00 %
GPA11    10.00 %
GPA12    10.00 %
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GPA15    10.00 %
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GPA93    10.00 %
GPA94    10.00 %
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GPA98    10.00 %
GPA99    10.00 %
GPA100   10.00 %

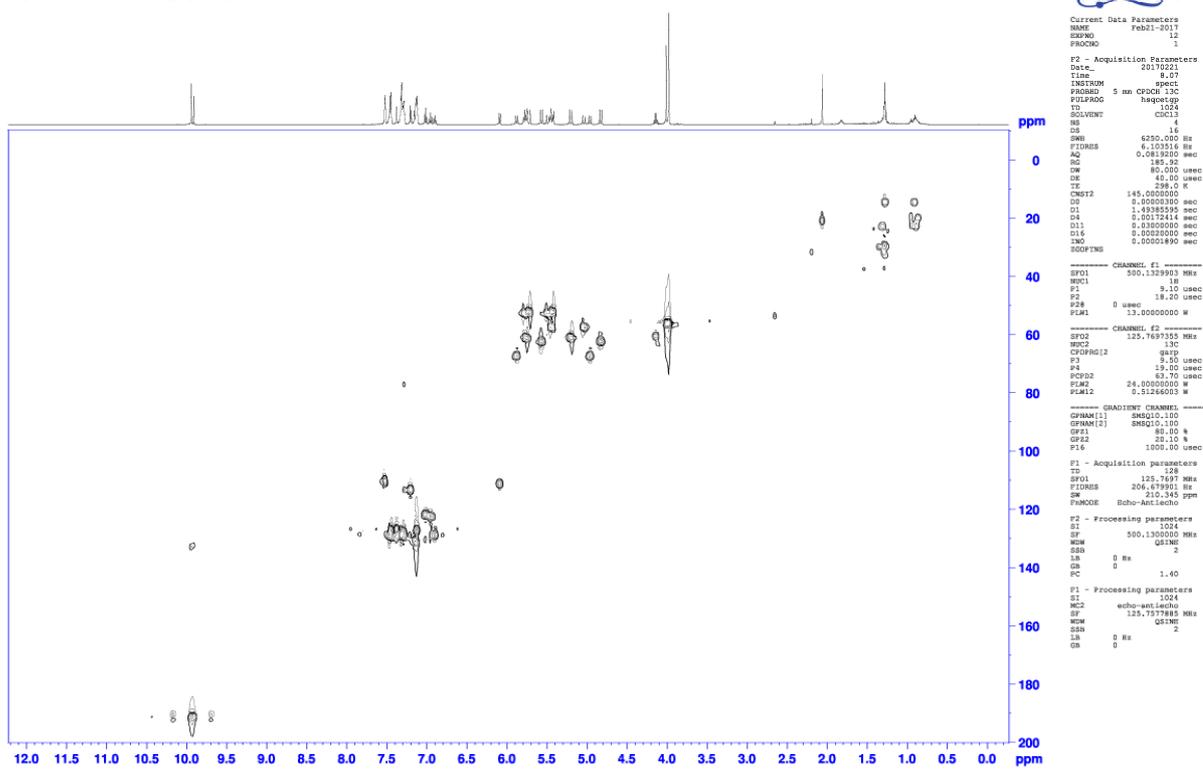
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FREQ0    125.7728195 ppm

F2 - Processing parameters
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SF        500.1300000 MHz
SF0       0
GB        0 Hz
GB0       0 Hz
PC        1.40

F1 - Processing parameters
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SF0       0
GB        0 Hz
GB0       0 Hz
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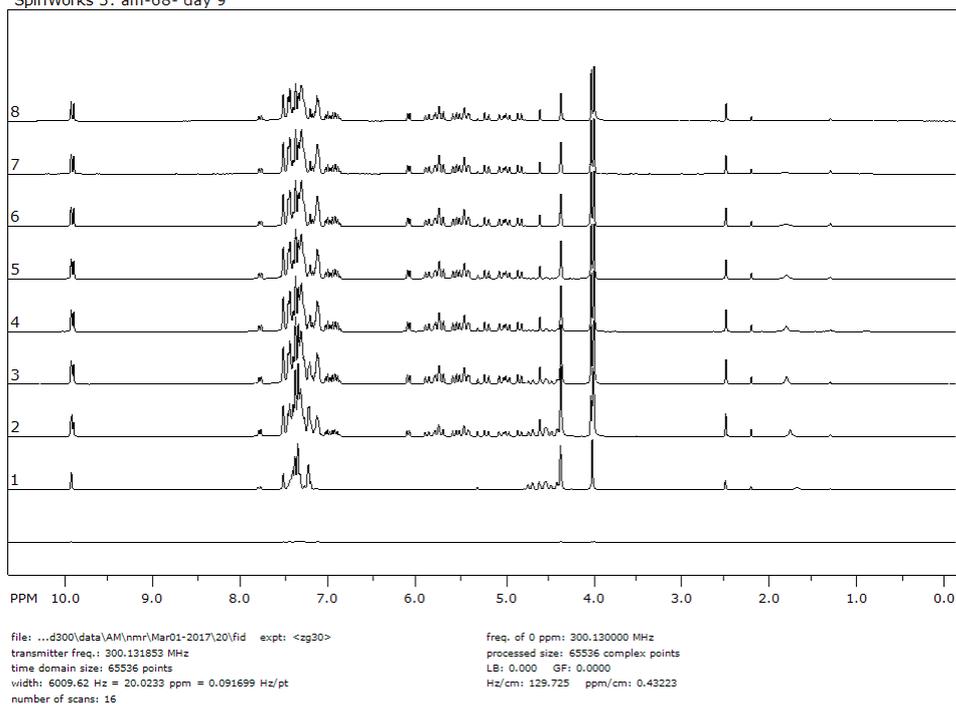
HSQC:

Chemist Anish Mistry  
AM-63 pure  
HSQC.w CDCI3 /opt/topspin3.2 AM 11

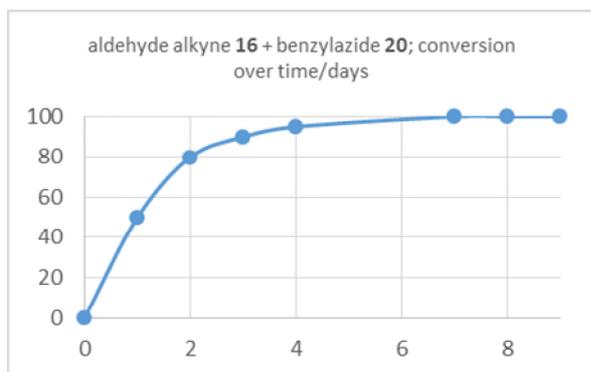


Conversion over time (AM68): Alkyne **16** (15 mg, 51.0  $\mu\text{mol}$ ) and benzyl azide **20** (6.4 mg, 51.0  $\mu\text{mol}$ ) were added together in deuterated chloroform (0.4 mL) and the reaction left at r.t.. The progression of the reaction was monitored daily by  $^1\text{H}$ -NMR. (0.128 M in both reagents).

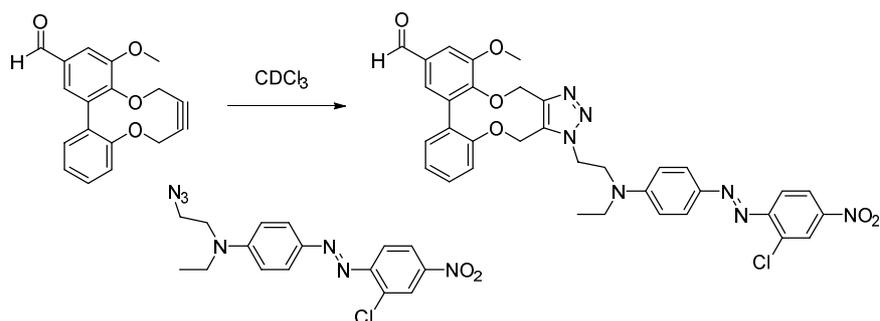
SpinWorks 3: am-68- day 9



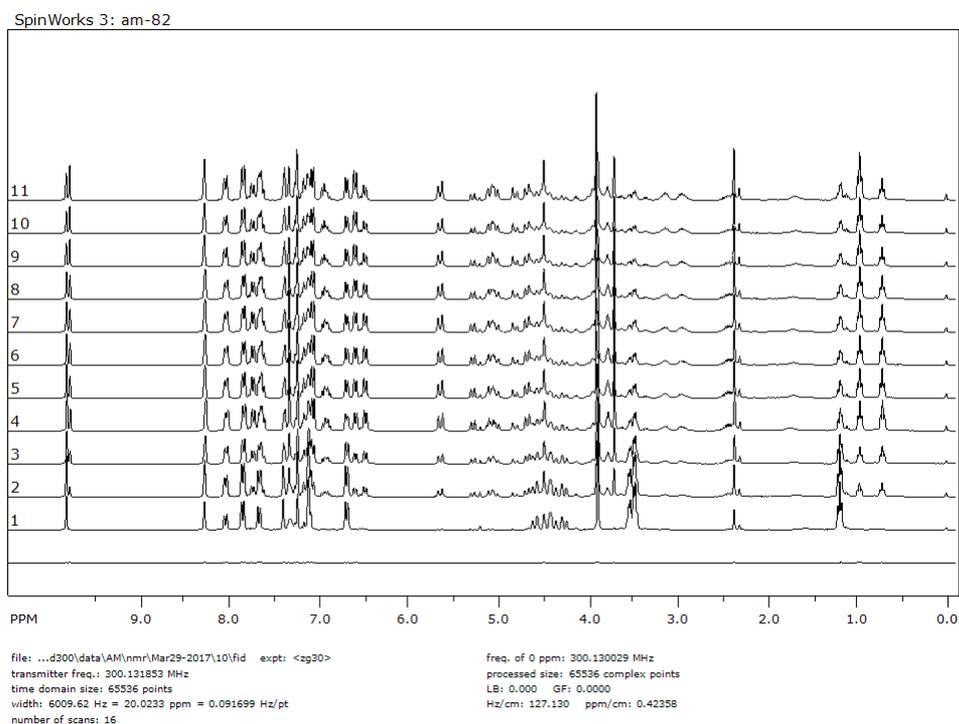
Characteristic peaks in the NMR used to define conversion;  $\delta_H$  (300 MHz,  $\text{CDCl}_3$ ) 9.82 (1H, s,  $\text{CHO}$ ), 7.10-6.90 (1H, m, ArH), 6.20-4.90 (6H, 6xAB systems for 6  $\text{CH}_2$  groups), 3.98 (s, 3H, isomer 1  $\text{OCH}_3$ ), 3.94 (s, 3H, isomer 2  $\text{OCH}_3$ ). The spectrum contains  $\text{PhCH}_2\text{N}_3$  peak at ca.  $\delta$  4.45. Conv approx.; 1) start, 0%, 2) day 1, 50%, 3) day 2, 80%, 4) day 3, 90%, 5) day 4, 95%, 6) day 7, 100%, 7) day 8, 100%, 8) day 9, 100%.



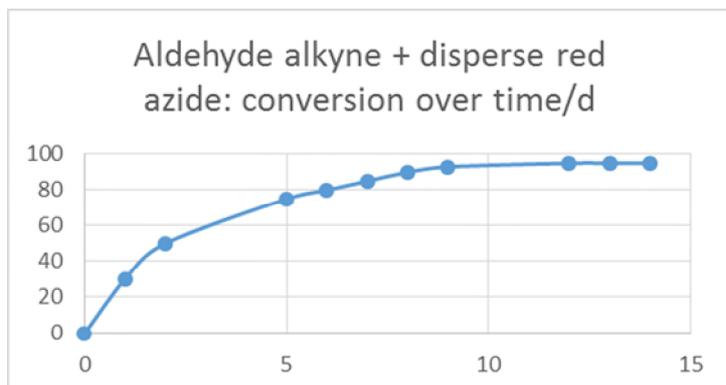
## Disperse red cycloadduct 23 (AM82).



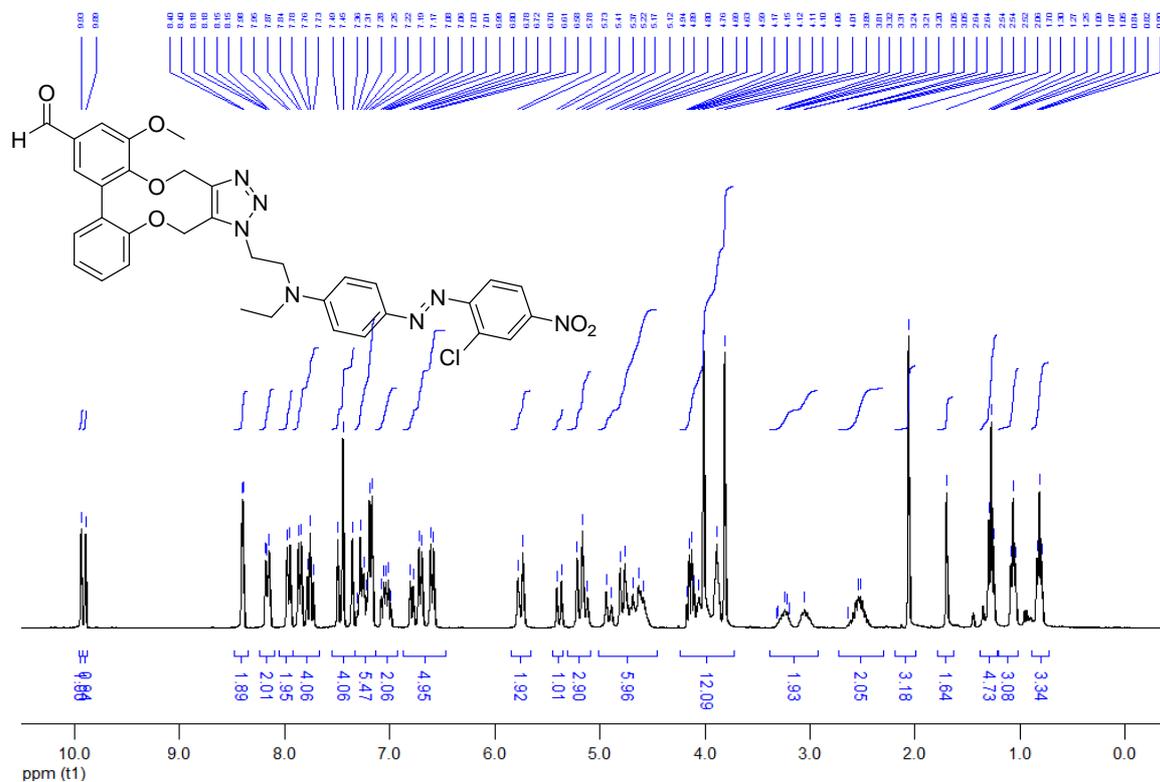
Stacked spectra over time intervals (defined below):



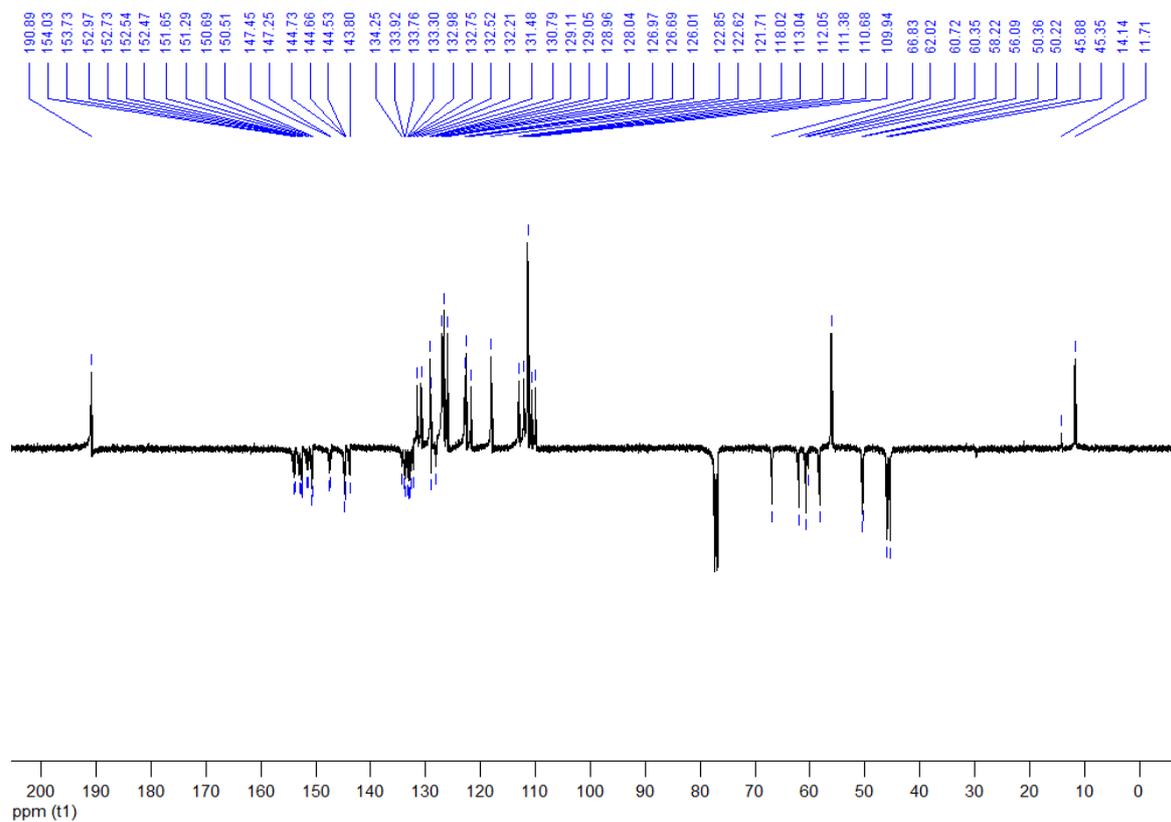
Characteristic peaks in the NMR used to define conversion:  $\delta_{\text{H}}$  (300 MHz, CDCl<sub>3</sub>); starting material OCH<sub>2</sub> peaks (4 H) at 4.60-4.15, product OCH<sub>2</sub> peaks (4 H) at 5.70-4.70. Conv approx.: 1) start, 0%, 2) day, 30%, 3) day 2, 50%, 4) day 5, 75%, 5) day 6, 80%, 6) day 7, 85%, 7) day 8, 90%, 8) day 9, 93%, 9-11), days 12,13,14, all 95%.



$\delta_H$  (300 MHz,  $CDCl_3$ ).

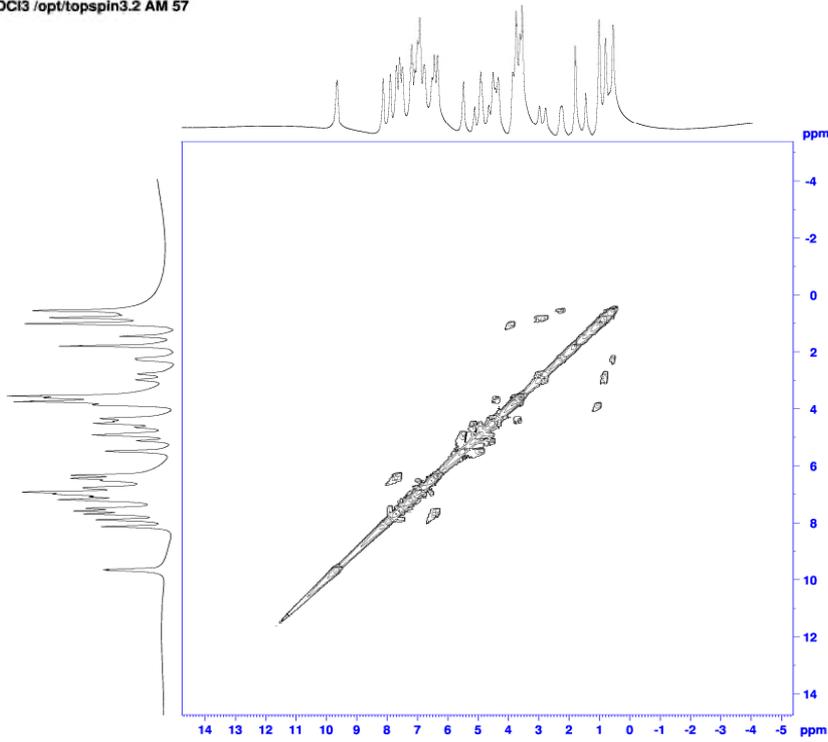


$\delta_C$  (126 MHz,  $CDCl_3$ ).



# COSY:

Chemist Anish Mistry  
AM-82 pure  
COSY.w CDCl3 /opt/topspin3.2 AM 57



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Current Data Parameters
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PROCNO   1

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PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        4
DS        2
SWH       10080.646 Hz
FIDRES   4.322189 Hz
AQ        0.1018898 sec
RG        452.5
DW        49.600 usec
DE        45.00 usec
TE        298.0 K
DQ        0.0000305 sec
D1        2.04710388 sec
D11       0.00000000 sec
D12       0.00002000 sec
D13       0.00000400 sec
D16       0.00000000 sec
DSD       0.00000000 sec

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NUC1     13
P1        9.10 usec
P11       9.10 usec
P17       2500.00 usec
PULSE    13.00000000 W
PLW10    1.59249997 W

----- GRADIENT CHANNEL -----
GPM1[1]  SMQ10.100
GP1[1]   10.00 %
P1[1]    1000.00 usec

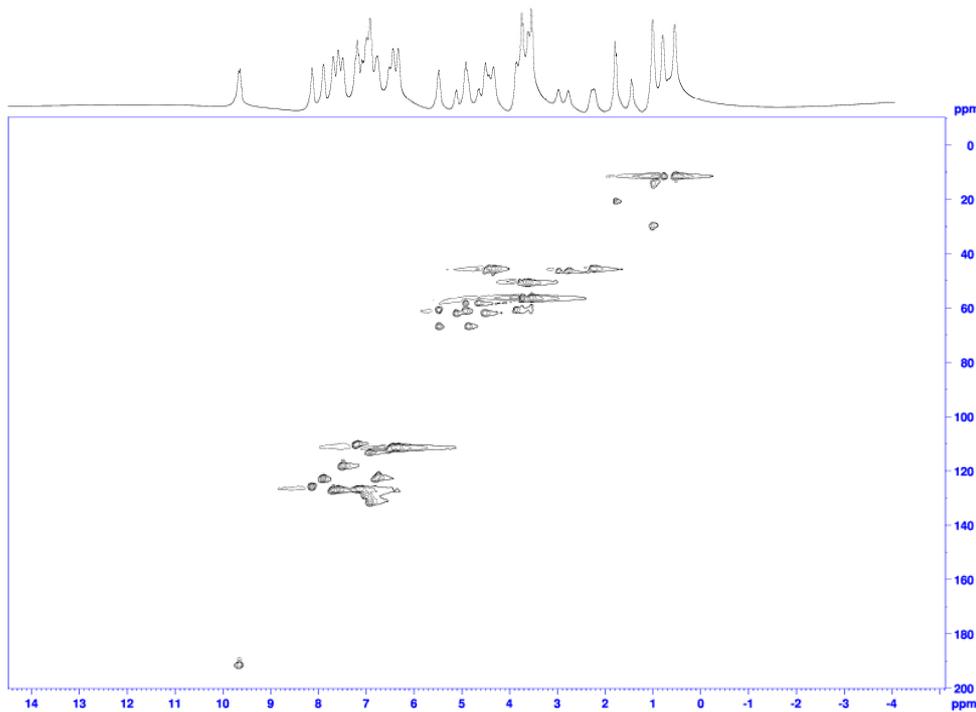
F1 - Acquisition parameters
TD        128
SFO1     500.1324680 MHz
FIDRES   78.396581 Hz
SW        20.113 ppm
FWDKDE   QF

F2 - Processing parameters
SI        32768
SF        500.1301244 MHz
WDW       QDRING
SSB       0 Hz
LB        0 Hz
GB        0 Hz
PC        1.40

F1 - Processing parameters
SI        1024
SF        500.1301244 MHz
WDW       QDRING
SSB       0 Hz
LB        0 Hz
GB        0 Hz
    
```

# HSQC:

HSQC.w CDCl3 /opt/topspin3.2 AM 57



```

Current Data Parameters
NAME      Mar31-2017
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20170402
Time     14.20
INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        4
DS        2
SWH       9803.952 Hz
FIDRES   9.276162 Hz
AQ        0.0222240 sec
RG        380.02
DW        51.000 usec
DE        50.00 usec
TE        298.0 K
DQ        0.00000000 sec
D1        1.8355194 sec
D11       0.00000000 sec
D12       0.00000000 sec
D13       0.00000000 sec
D16       0.00000000 sec
DSD       0.00000000 sec
DSDPRG2  HSEPTHS

----- CHANNEL f1 -----
SFO1     500.1324680 MHz
NUC1     13
P1        9.10 usec
P11       9.10 usec
P17       2500.00 usec
PULSE    13.00000000 W
PLW10    1.59249997 W

----- CHANNEL f2 -----
SFO2     125.7619500 MHz
INSTRUM  spect
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        4
DS        2
SWH       9803.952 Hz
FIDRES   9.276162 Hz
AQ        0.0222240 sec
RG        380.02
DW        51.000 usec
DE        50.00 usec
TE        298.0 K
DQ        0.00000000 sec
D1        1.8355194 sec
D11       0.00000000 sec
D12       0.00000000 sec
D13       0.00000000 sec
D16       0.00000000 sec
DSD       0.00000000 sec
DSDPRG2  HSEPTHS

----- GRADIENT CHANNEL -----
GPM1[1]  SMQ10.100
GPM2[2]  SMQ10.100
GP1[1]   10.00 %
GP2[2]   10.00 %
P1[1]    1000.00 usec
P1[2]    1000.00 usec

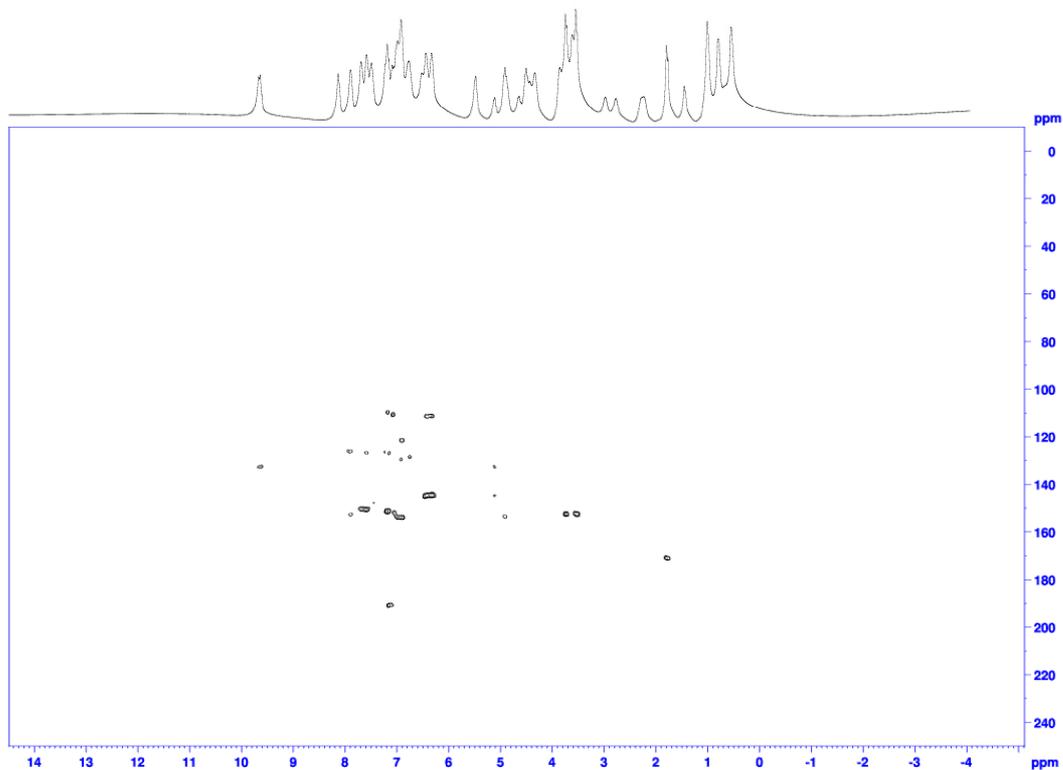
F1 - Acquisition parameters
TD        128
SFO1     125.7619500 MHz
FIDRES   206.074901 Hz
SW        22.21250 ppm
FWDKDE   Echo-Am1iecho

F2 - Processing parameters
SI        32768
SF        500.1301244 MHz
WDW       QDRING
SSB       0 Hz
LB        0 Hz
GB        0 Hz
PC        1.40

F1 - Processing parameters
SI        1024
SF        125.7617880 MHz
WDW       QDRING
SSB       0 Hz
LB        0 Hz
GB        0 Hz
    
```

HMBC:

HMBC.w CDCI3 /opt/topspin3.2 AM 57



```
Current Data Parameters
NAME      Msc1-2017
EXPNO    13
PROCNO   1

F2 - Acquisition Parameters
Date_    20170402
Time     14.41
INSTRUM  spect
PROBHD   5 mm CPDQX 13C
PULPROG  mbocgplpdcqr
TD        1024
SOLVENT  CDCl3
NS        8
DS        4
SWH       9803.922 Hz
FIDRES    9.574142 Hz
AQ        0.0522240 sec
RG         185.50
DE         91.000
DK         49.00 usec
TE        298.0
CMT13    145.0000000
DQ        10.0000000
DI        0.0000350 sec
D1        1.5235114 sec
D2        0.00148828 sec
D3        0.0000000 sec
D4        0.0000000 sec
D5        0.0000000 sec
D6        0.0000000 sec
D7        0.0000000 sec
D8        0.0000000 sec
D9        0.0000000 sec
D10       0.0000000 sec
D11       0.0000000 sec
D12       0.0000000 sec
D13       0.0000000 sec
D14       0.0000000 sec
D15       0.0000000 sec
D16       0.0000000 sec
D17       0.0000000 sec
D18       0.0000000 sec
D19       0.0000000 sec
D20       0.0000000 sec
D21       0.0000000 sec
D22       0.0000000 sec
D23       0.0000000 sec
D24       0.0000000 sec
D25       0.0000000 sec
D26       0.0000000 sec
D27       0.0000000 sec
D28       0.0000000 sec
D29       0.0000000 sec
D30       0.0000000 sec
D31       0.0000000 sec
D32       0.0000000 sec
D33       0.0000000 sec
D34       0.0000000 sec
D35       0.0000000 sec
D36       0.0000000 sec
D37       0.0000000 sec
D38       0.0000000 sec
D39       0.0000000 sec
D40       0.0000000 sec
D41       0.0000000 sec
D42       0.0000000 sec
D43       0.0000000 sec
D44       0.0000000 sec
D45       0.0000000 sec
D46       0.0000000 sec
D47       0.0000000 sec
D48       0.0000000 sec
D49       0.0000000 sec
D50       0.0000000 sec
D51       0.0000000 sec
D52       0.0000000 sec
D53       0.0000000 sec
D54       0.0000000 sec
D55       0.0000000 sec
D56       0.0000000 sec
D57       0.0000000 sec
D58       0.0000000 sec
D59       0.0000000 sec
D60       0.0000000 sec
D61       0.0000000 sec
D62       0.0000000 sec
D63       0.0000000 sec
D64       0.0000000 sec
D65       0.0000000 sec
D66       0.0000000 sec
D67       0.0000000 sec
D68       0.0000000 sec
D69       0.0000000 sec
D70       0.0000000 sec
D71       0.0000000 sec
D72       0.0000000 sec
D73       0.0000000 sec
D74       0.0000000 sec
D75       0.0000000 sec
D76       0.0000000 sec
D77       0.0000000 sec
D78       0.0000000 sec
D79       0.0000000 sec
D80       0.0000000 sec
D81       0.0000000 sec
D82       0.0000000 sec
D83       0.0000000 sec
D84       0.0000000 sec
D85       0.0000000 sec
D86       0.0000000 sec
D87       0.0000000 sec
D88       0.0000000 sec
D89       0.0000000 sec
D90       0.0000000 sec
D91       0.0000000 sec
D92       0.0000000 sec
D93       0.0000000 sec
D94       0.0000000 sec
D95       0.0000000 sec
D96       0.0000000 sec
D97       0.0000000 sec
D98       0.0000000 sec
D99       0.0000000 sec
D100      0.0000000 sec

----- CHANNEL f1 -----
SFO1     500.1324645 MHz
NUC1     13C
P1        9.10 usec
P2        18.20 usec
PL1       13.00000000 dB
PL2       0.00000000 dB

----- CHANNEL f2 -----
SFO2     125.772795 MHz
NUC2     13C
P3        9.10 usec
P4        18.20 usec
PL3       13.00000000 dB
PL4       0.00000000 dB

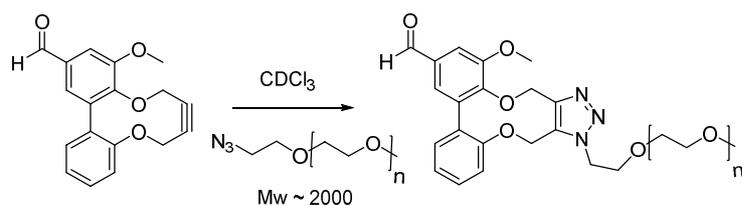
----- GRABINQ CHANNEL -----
CPGRAM[1] SINEQ10.100
CPGRAM[2] SINEQ10.100
CPGRAM[3] SINEQ10.100
GRP1      50.00 Hz
GRP2      50.00 Hz
GRP3      48.10 Hz
P16       1000.10 usec

F1 - Acquisition parameters
TD        1024
SFO1     500.1324645 MHz
FIDRES    9.574142 Hz
AQ        0.0522240 sec
RG         185.50
DE         91.000
DK         49.00 usec
TE        298.0
CMT13    145.0000000
DQ        10.0000000
DI        0.0000350 sec
D1        1.5235114 sec
D2        0.00148828 sec
D3        0.0000000 sec
D4        0.0000000 sec
D5        0.0000000 sec
D6        0.0000000 sec
D7        0.0000000 sec
D8        0.0000000 sec
D9        0.0000000 sec
D10       0.0000000 sec
D11       0.0000000 sec
D12       0.0000000 sec
D13       0.0000000 sec
D14       0.0000000 sec
D15       0.0000000 sec
D16       0.0000000 sec
D17       0.0000000 sec
D18       0.0000000 sec
D19       0.0000000 sec
D20       0.0000000 sec
D21       0.0000000 sec
D22       0.0000000 sec
D23       0.0000000 sec
D24       0.0000000 sec
D25       0.0000000 sec
D26       0.0000000 sec
D27       0.0000000 sec
D28       0.0000000 sec
D29       0.0000000 sec
D30       0.0000000 sec
D31       0.0000000 sec
D32       0.0000000 sec
D33       0.0000000 sec
D34       0.0000000 sec
D35       0.0000000 sec
D36       0.0000000 sec
D37       0.0000000 sec
D38       0.0000000 sec
D39       0.0000000 sec
D40       0.0000000 sec
D41       0.0000000 sec
D42       0.0000000 sec
D43       0.0000000 sec
D44       0.0000000 sec
D45       0.0000000 sec
D46       0.0000000 sec
D47       0.0000000 sec
D48       0.0000000 sec
D49       0.0000000 sec
D50       0.0000000 sec
D51       0.0000000 sec
D52       0.0000000 sec
D53       0.0000000 sec
D54       0.0000000 sec
D55       0.0000000 sec
D56       0.0000000 sec
D57       0.0000000 sec
D58       0.0000000 sec
D59       0.0000000 sec
D60       0.0000000 sec
D61       0.0000000 sec
D62       0.0000000 sec
D63       0.0000000 sec
D64       0.0000000 sec
D65       0.0000000 sec
D66       0.0000000 sec
D67       0.0000000 sec
D68       0.0000000 sec
D69       0.0000000 sec
D70       0.0000000 sec
D71       0.0000000 sec
D72       0.0000000 sec
D73       0.0000000 sec
D74       0.0000000 sec
D75       0.0000000 sec
D76       0.0000000 sec
D77       0.0000000 sec
D78       0.0000000 sec
D79       0.0000000 sec
D80       0.0000000 sec
D81       0.0000000 sec
D82       0.0000000 sec
D83       0.0000000 sec
D84       0.0000000 sec
D85       0.0000000 sec
D86       0.0000000 sec
D87       0.0000000 sec
D88       0.0000000 sec
D89       0.0000000 sec
D90       0.0000000 sec
D91       0.0000000 sec
D92       0.0000000 sec
D93       0.0000000 sec
D94       0.0000000 sec
D95       0.0000000 sec
D96       0.0000000 sec
D97       0.0000000 sec
D98       0.0000000 sec
D99       0.0000000 sec
D100      0.0000000 sec

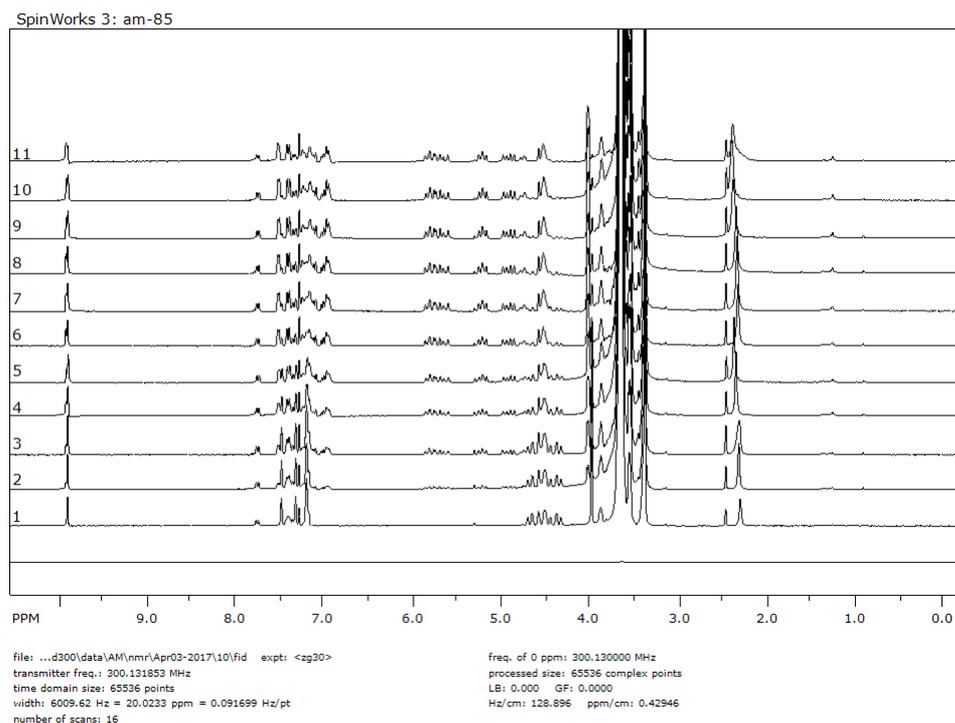
F2 - Processing parameters
SI        32768
SF        500.1301246 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
SF        125.772795 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
```

## Cycloadduct of PEG-2000 23. AM85.

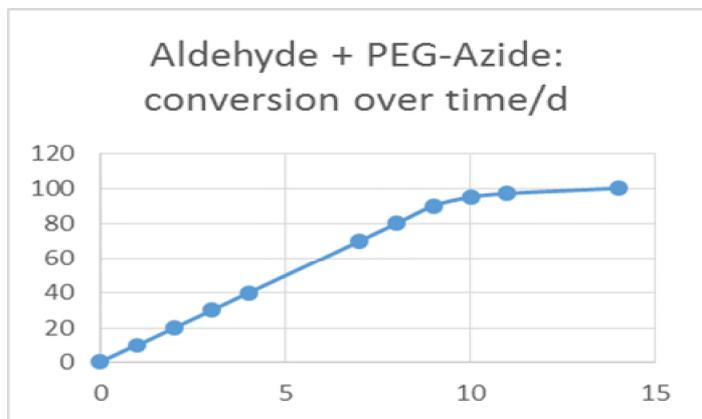
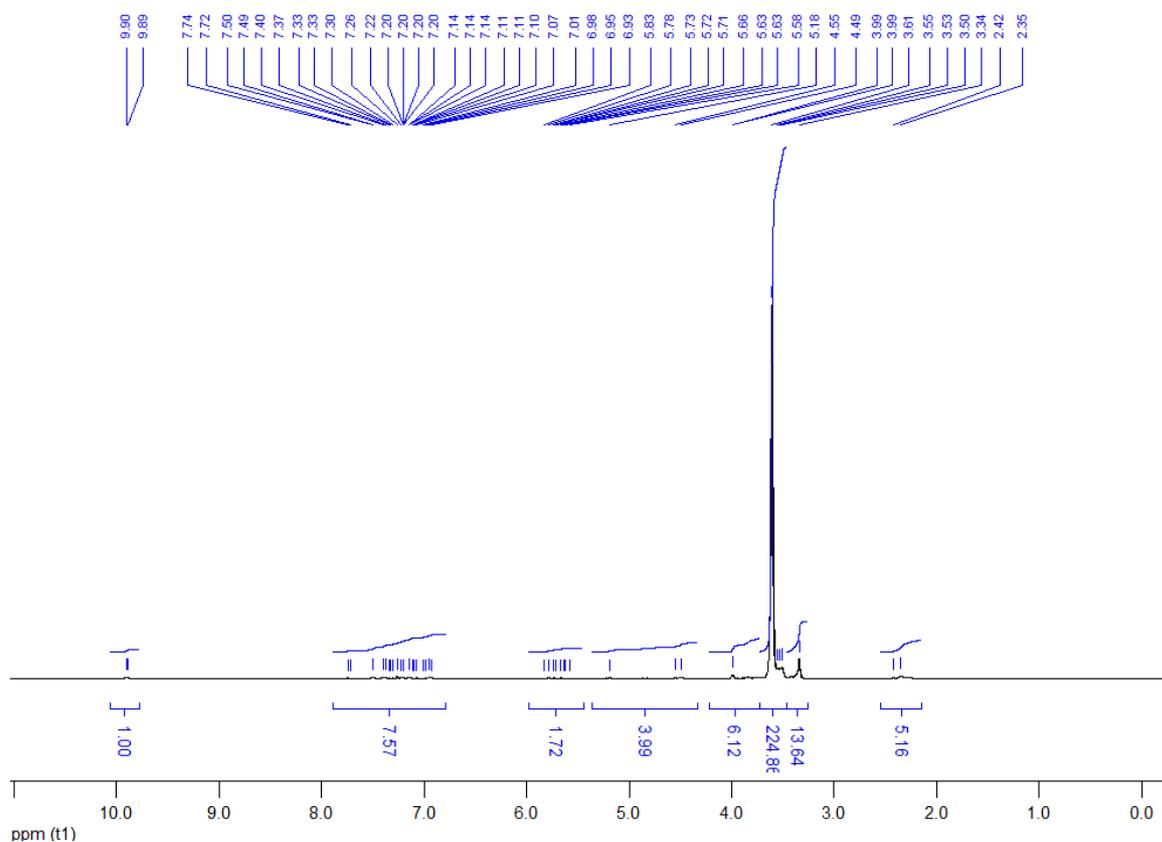


$^1\text{H}$  NMR followed over time:

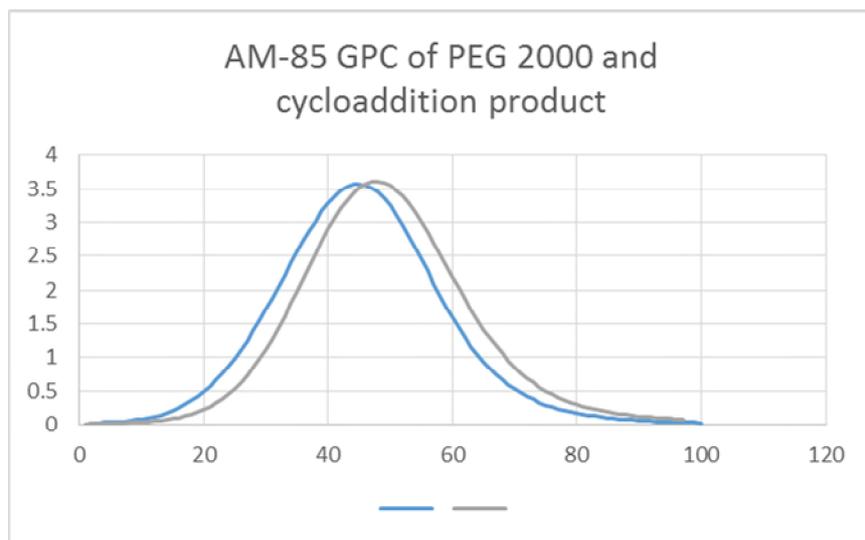


Conv approx.: 1) start, 0%, 2) day 1, 10%, 3) day 2, 20%, 4) day 3, 30%, 5) day 4, 40%, 6) day 7, 70%, 7) day 8, 80%, 8) day 9, 90%, 9) day 10, 95%, 10) day 11, 97%, 11) day 14, 100%.

Day 14, full NMR spectrum:



GPC data is from the reaction above.



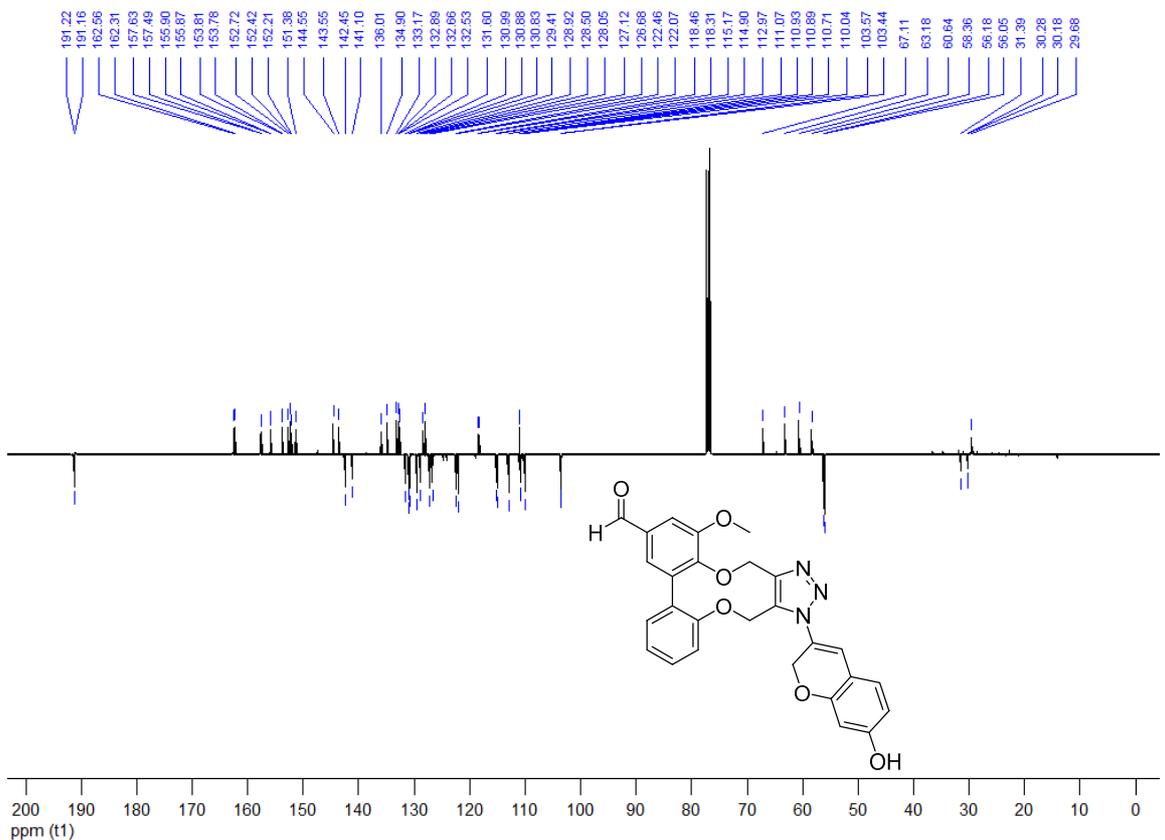
Blue is the starting material PEG-azide. Grey is AM-85 product of cycloaddition. Agilent 390LC MDS instrument equipped with differential refractive index (DRI), viscometry (VS) and dual angle light scatter (LS) detectors, with full details in main paper.

$M_n$ (g/mol)	$M_w$ (g/mol)	PDi
2100	2300	1.08

The PDi is near to 1 which is ideal (sharper peak). The molecular weight is 2305 which is very close to that of which we require (2294).

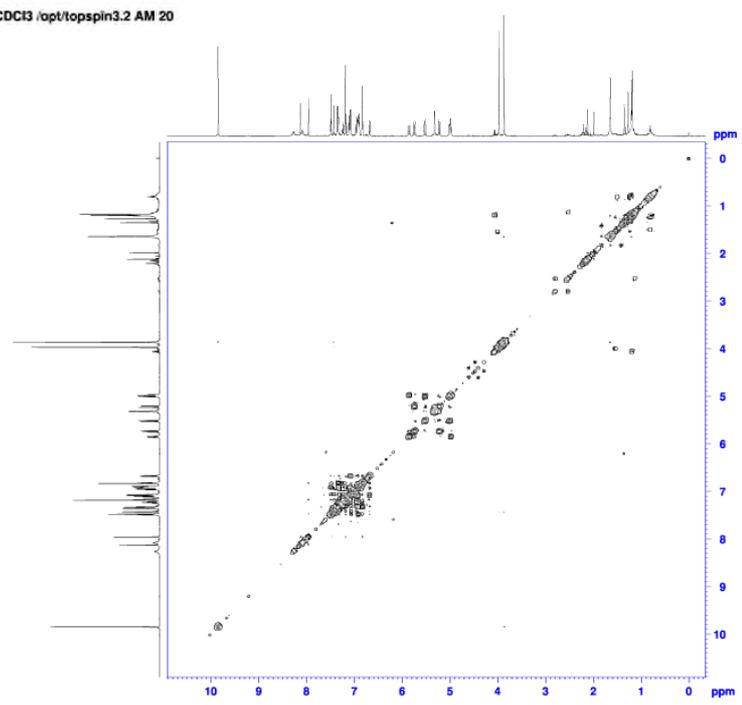


$\delta_C$  (126 MHz,  $CDCl_3$ ).



COSY:

Chemist Anish Mistry  
AM-88  
COSY.w  $CDCl_3$  /opt/topspin3.2 AM 20

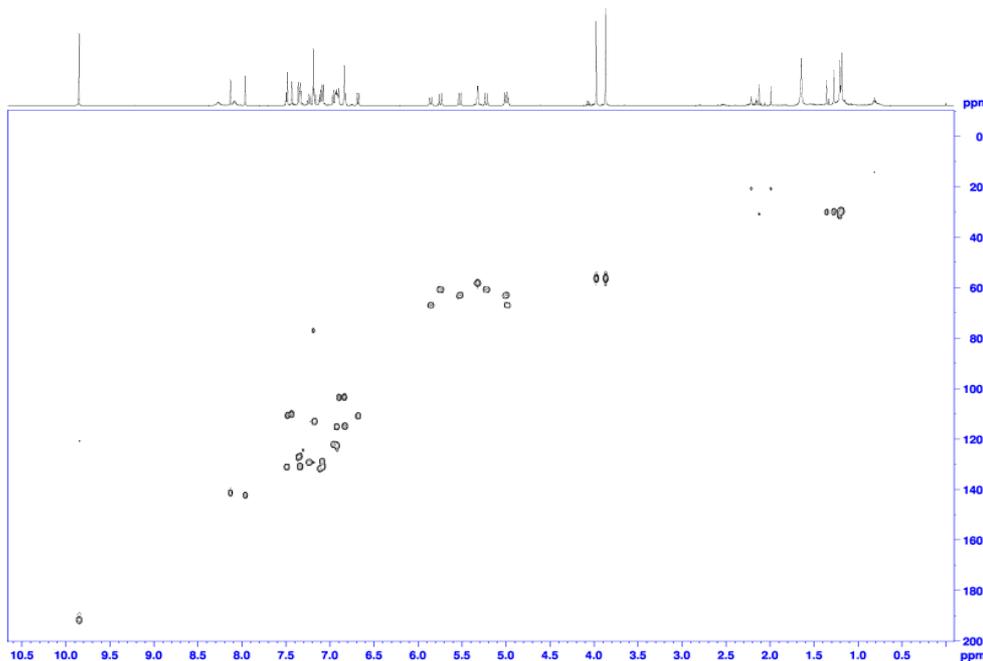


```

Current Data Parameters
NAME      Apr06-2011
EXPNO    11
PROCNO   1
----- Acquisition Parameters
Date_    20170407
Time     11:45
INSTRUM spect
PROBHD   5 mm CPICH 13C
PULPROG zgpg30
TD        65536
SOLVENT  CDCl3
NS        2
DS        4
SWH       5639.431 Hz
F2RES     2.748331 Hz
AQ        0.318824 sec
RG         65.07
DM        88.800 usec
DE        65.00 usec
TE        298.0 K
DQ        0.0000300 sec
DI1       1.96108794 sec
DI2       0.00000000 sec
DI3       0.00000000 sec
TI4       0.00000000 sec
RG        0.0001790 sec
----- CHANNEL f1 -----
NUC1      13C
NUC2      1H
P1        8.10 usec
P12       8.10 usec
P17       2500.00 usec
PL1       13.00000000 W
PL12      1.32489951 W
----- GRADIENT CHANNEL -----
SPRAME1   SMO10.100
GR1       10.00 %
P16       1000.00 usec
F1 - Acquisition parameters
NUC1      13C
NUC2      1H
P1        8.10 usec
P12       8.10 usec
P17       2500.00 usec
PL1       13.00000000 W
PL12      1.32489951 W
F2 - Processing parameters
SI        1024
SF        500.1300473 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.40
F1 - Processing parameters
SI        1024
SF        500.1300473 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.40
  
```

# HSQC:

Chemist Anish Mistry  
AM-86  
HSQC.w CDCI3 /opt/topspin3.2 AM 20



```

Current Data Parameters
NAME      Ag06-2011
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    2015021
Time     1.40
INSTRUM spect
PROBHD   5 mm CPDPR 13
PULPROG zgpg30
TD       1024
SOLVENT  CDCl3
DS       4
SWH      1574.244 Hz
FIDRES   0.125776 Hz
AQ       0.000330 sec
RG       185.00
DM       91.000 usec
DE       0.000000 sec
TE       300.2 K
CMT02    145.000000 usec
D1       1.4804397 usec
D8       0.0000000 usec
D11      0.0000000 usec
D16      0.0000000 usec
IND      0.00001890 sec

===== CHANNEL f1 =====
SFO1    500.1304000 MHz
NUC1    13C
P1      12.00 usec
SFO2    0 usec
P2      0 usec
P3      13.0000000 MHz

===== CHANNEL f2 =====
SFO3    125.7677800 MHz
NUC3    13C
CPDPRG2 zgpg30
SFO4    125.7677800 MHz
P3      12.00 usec
P4      12.00 usec
P5      24.0000000 MHz
P6      0.012400000 MHz

===== GRABBER CHANNEL =====
CPMAM11 8MHz10.100
CPMAM12 8MHz10.100
CPMAM13 8MHz10.100
CPMAM14 8MHz10.100
CPMAM15 1000.00 usec

F1 - Acquisition parameters
SFO1    500.1304000 MHz
P1      12.00 usec
FIDRES   0.125776 Hz
AQ       0.000330 sec
RG       185.00
DM       91.000 usec
DE       0.000000 sec
TE       300.2 K
CMT02    145.000000 usec
D1       1.4804397 usec
D8       0.0000000 usec
D11      0.0000000 usec
D16      0.0000000 usec
IND      0.00001890 sec

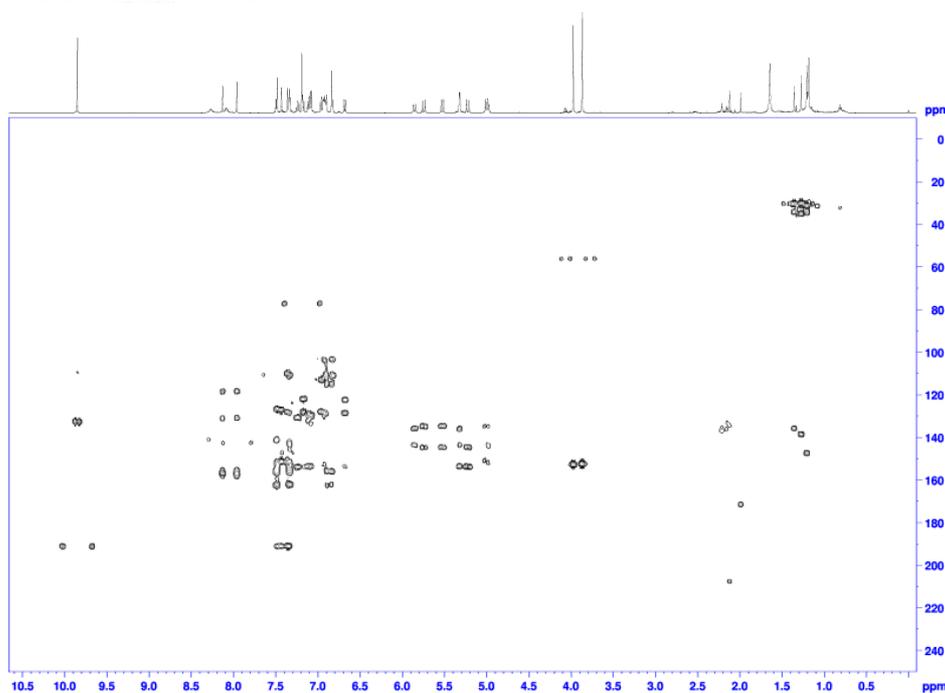
F2 - Processing parameters
SI      32768
SF      500.1304000 MHz
WDW     EM
SSB     0
GB      0
PC      1.40

F1 - Processing parameters
SI      32768
SF      500.1304000 MHz
WDW     EM
SSB     0
GB      0
PC      1.40

F3 - Processing parameters
SI      32768
SF      125.7677800 MHz
WDW     EM
SSB     0
GB      0
PC      1.40
    
```

# HMBC:

Chemist Anish Mistry  
AM-86  
HMBC.w CDCI3 /opt/topspin3.2 AM 20



```

Current Data Parameters
NAME      Ag06-2011
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    2015021
Time     2.02
INSTRUM spect
PROBHD   5 mm CPDPR 13C
PULPROG zgpg30
TD       1024
SOLVENT  CDCl3
DS       4
SWH      1574.244 Hz
FIDRES   0.125776 Hz
AQ       0.000330 sec
RG       185.00
DM       91.000 usec
DE       0.000000 sec
TE       300.2 K
CMT02    145.000000 usec
D1       1.4804397 usec
D8       0.0000000 usec
D11      0.0000000 usec
D16      0.0000000 usec
IND      0.00001890 sec

===== CHANNEL f1 =====
SFO1    500.1304000 MHz
NUC1    13C
P1      12.00 usec
SFO2    0 usec
P2      0 usec
P3      13.0000000 MHz

===== CHANNEL f2 =====
SFO3    125.7677800 MHz
NUC3    13C
CPDPRG2 zgpg30
SFO4    125.7677800 MHz
P3      12.00 usec
P4      12.00 usec
P5      24.0000000 MHz
P6      0.012400000 MHz

===== GRABBER CHANNEL =====
CPMAM11 8MHz10.100
CPMAM12 8MHz10.100
CPMAM13 8MHz10.100
CPMAM14 8MHz10.100
CPMAM15 1000.00 usec

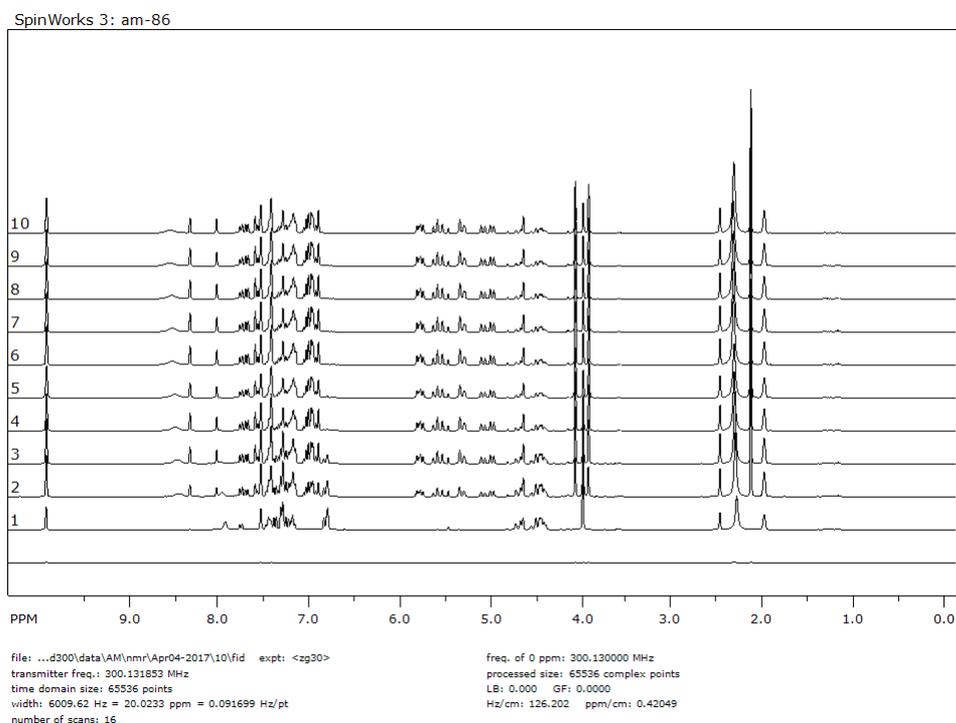
F1 - Acquisition parameters
SFO1    500.1304000 MHz
P1      12.00 usec
FIDRES   0.125776 Hz
AQ       0.000330 sec
RG       185.00
DM       91.000 usec
DE       0.000000 sec
TE       300.2 K
CMT02    145.000000 usec
D1       1.4804397 usec
D8       0.0000000 usec
D11      0.0000000 usec
D16      0.0000000 usec
IND      0.00001890 sec

F2 - Processing parameters
SI      32768
SF      500.1304000 MHz
WDW     EM
SSB     0
GB      0
PC      1.40

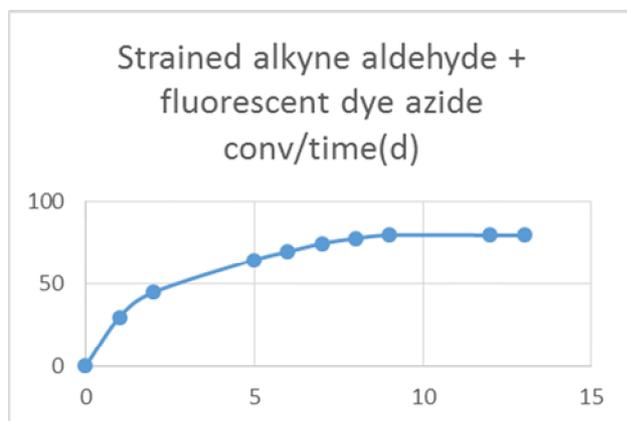
F1 - Processing parameters
SI      32768
SF      500.1304000 MHz
WDW     EM
SSB     0
GB      0
PC      1.40

F3 - Processing parameters
SI      32768
SF      125.7677800 MHz
WDW     EM
SSB     0
GB      0
PC      1.40
    
```

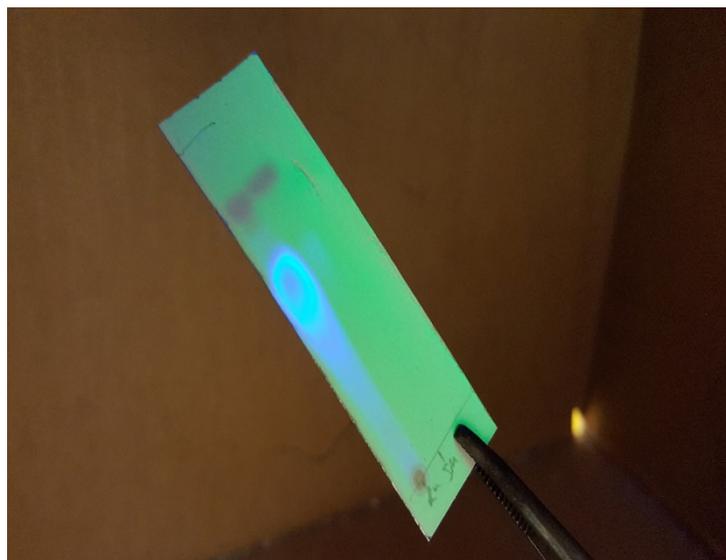
The conversion was followed over time during the reaction. Characteristic peaks of product were observed as follows:



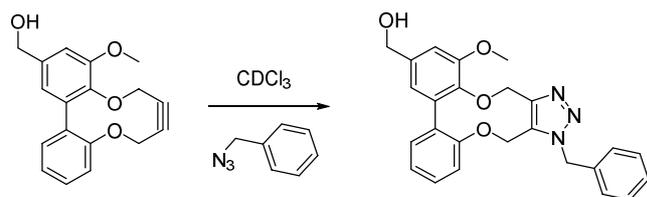
$\delta_H$  (400 MHz,  $CDCl_3$ ) 9.84+9.82 (1H, s x 2, *CHO*), 8.40-6.80 (m, ArHs..several distinctive, from Dye), 5.80-5.00 (4H, 2 x  $OCH_2$  in product), 4.65-4.35 ((4H, 2 x  $OCH_2$  in strained alkyne), 4.10 (3H, s,  $OCH_3$  - shifts). Conv approx.; 1) start, 0%, 2) day 1, 30%, 3) day 2, 45%, 4) day 5, 65%, 5) day 6, 70%, 6) day 7, 75%, 7) day 8, 75%, 8) day 9, 78%, 9) day 12, 80%, 10) day 13, 80%.



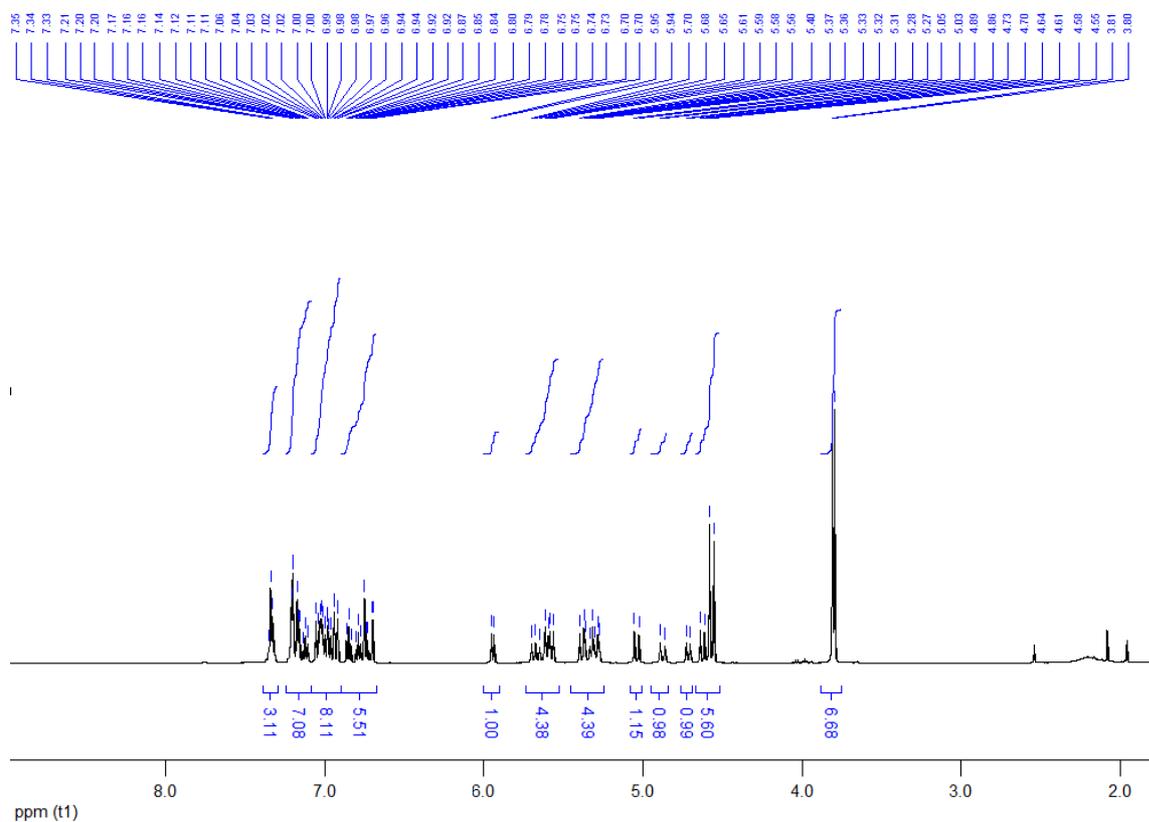
TLC of the fluorescent product, irradiated at 254 nm:



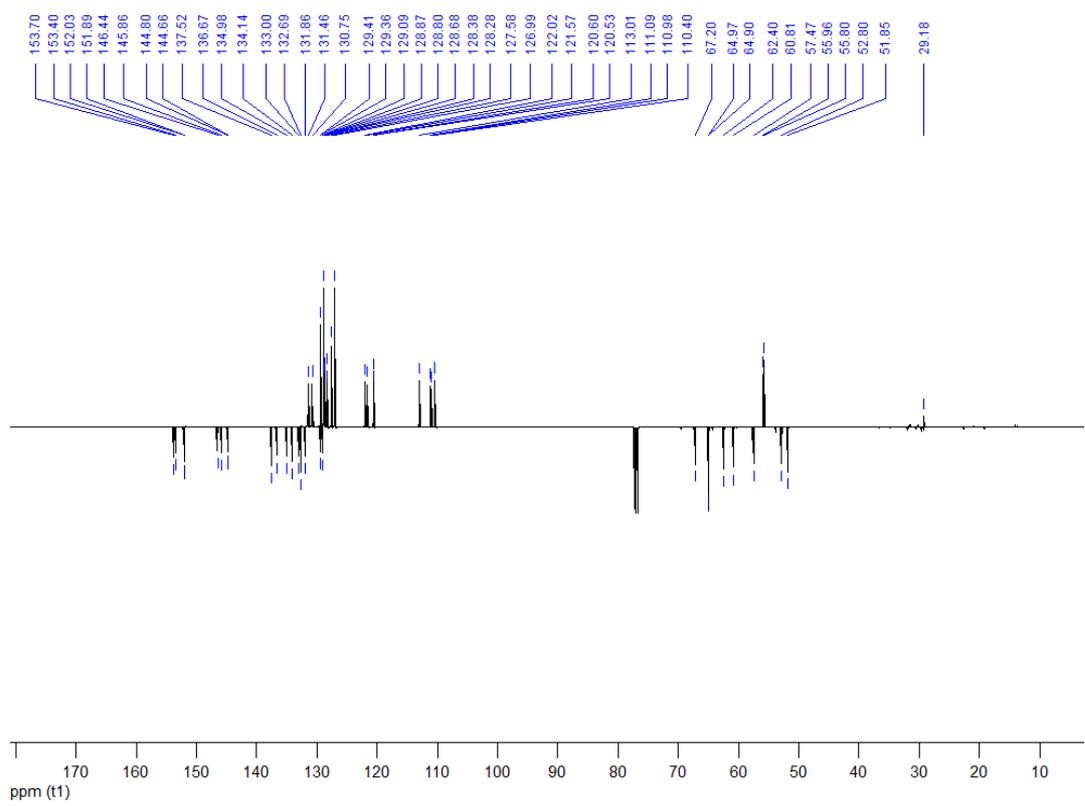
**Alcohol 19/benzyl azide Cycloadduct 28. AM60.**



$\delta_{\text{H}}$  (500 MHz,  $\text{CDCl}_3$ ).

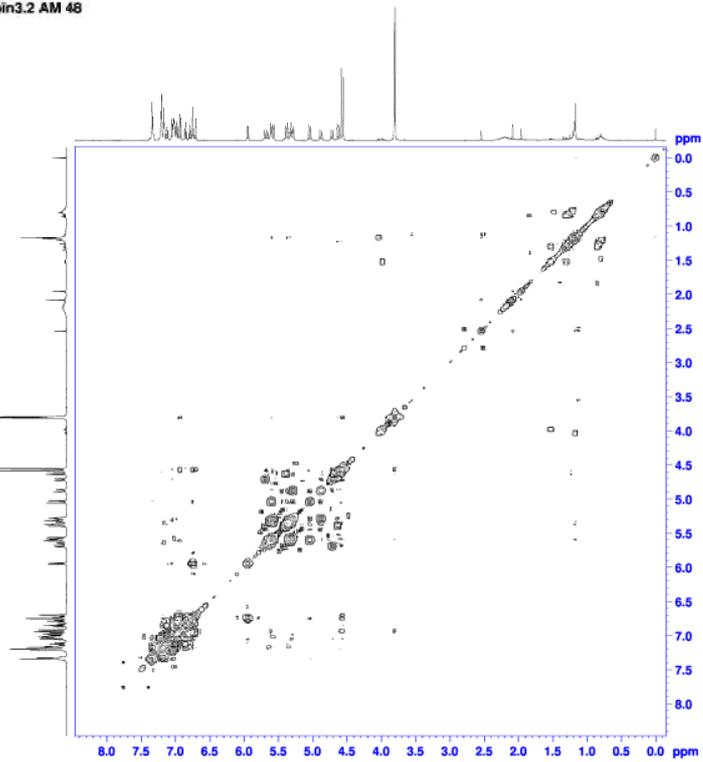


$\delta_C$  (126 MHz, CDCl<sub>3</sub>).



COSY:

Chemist Anish Mistry  
AM-60 pure  
COSY.w CDCl<sub>3</sub> /opt/topspin3.2 AM 48



```

Current Data Parameters
NAME      F0608-2013
EXPNO    11
PROCNO   1

F2 - Acquisition Parameters
Date_    20170208
Time     16.10
INSTRUM  spect
PROBHD   5 mm CPDCl 13C
P1PRG1   waltz16
SOLVENT  CDCl3
NS       1
DS       1
SWH      4310.343 Hz
F2FREQS  2.104561 Hz
AQ       0.2375680 sec
RG       17.41
DQ       116.0000 usec
TE       298.0 K
DE       0.0000200 sec
D1       1.89964793 sec
D11      0.0300000 sec
D12      0.0000200 sec
D13      0.0000000 sec
D14      0.0000000 sec
D15      0.0000000 sec
D16      0.0000000 sec
D17      0.0000000 sec
D18      0.0000000 sec
D19      0.0000000 sec
D20      0.0000000 sec
D21      0.0000000 sec
D22      0.0000000 sec
D23      0.0000000 sec
D24      0.0000000 sec
D25      0.0000000 sec
D26      0.0000000 sec
D27      0.0000000 sec
D28      0.0000000 sec
D29      0.0000000 sec
D30      0.0000000 sec
D31      0.0000000 sec
D32      0.0000000 sec
D33      0.0000000 sec
D34      0.0000000 sec
D35      0.0000000 sec
D36      0.0000000 sec
D37      0.0000000 sec
D38      0.0000000 sec
D39      0.0000000 sec
D40      0.0000000 sec
D41      0.0000000 sec
D42      0.0000000 sec
D43      0.0000000 sec
D44      0.0000000 sec
D45      0.0000000 sec
D46      0.0000000 sec
D47      0.0000000 sec
D48      0.0000000 sec
D49      0.0000000 sec
D50      0.0000000 sec
D51      0.0000000 sec
D52      0.0000000 sec
D53      0.0000000 sec
D54      0.0000000 sec
D55      0.0000000 sec
D56      0.0000000 sec
D57      0.0000000 sec
D58      0.0000000 sec
D59      0.0000000 sec
D60      0.0000000 sec
D61      0.0000000 sec
D62      0.0000000 sec
D63      0.0000000 sec
D64      0.0000000 sec
D65      0.0000000 sec
D66      0.0000000 sec
D67      0.0000000 sec
D68      0.0000000 sec
D69      0.0000000 sec
D70      0.0000000 sec
D71      0.0000000 sec
D72      0.0000000 sec
D73      0.0000000 sec
D74      0.0000000 sec
D75      0.0000000 sec
D76      0.0000000 sec
D77      0.0000000 sec
D78      0.0000000 sec
D79      0.0000000 sec
D80      0.0000000 sec
D81      0.0000000 sec
D82      0.0000000 sec
D83      0.0000000 sec
D84      0.0000000 sec
D85      0.0000000 sec
D86      0.0000000 sec
D87      0.0000000 sec
D88      0.0000000 sec
D89      0.0000000 sec
D90      0.0000000 sec
D91      0.0000000 sec
D92      0.0000000 sec
D93      0.0000000 sec
D94      0.0000000 sec
D95      0.0000000 sec
D96      0.0000000 sec
D97      0.0000000 sec
D98      0.0000000 sec
D99      0.0000000 sec
D100     0.0000000 sec

----- CHANNEL f1 -----
SFO1    500.1321341 MHz
NUC1    13C
P1      9.10 usec
PL1     2500.00 usec
PL12    13.00000000 W
PL14    1.33548893 W

----- GRADIENT CHANNEL -----
SFRAN[1] SMOG10.100
US1     10.00 %
P16     1000.00 usec

F1 - Acquisition parameters
TS      128
SFO1    500.1321 MHz
F2FREQS 31.703635 Hz
SW      8.426 ppm
FREQDIX CF

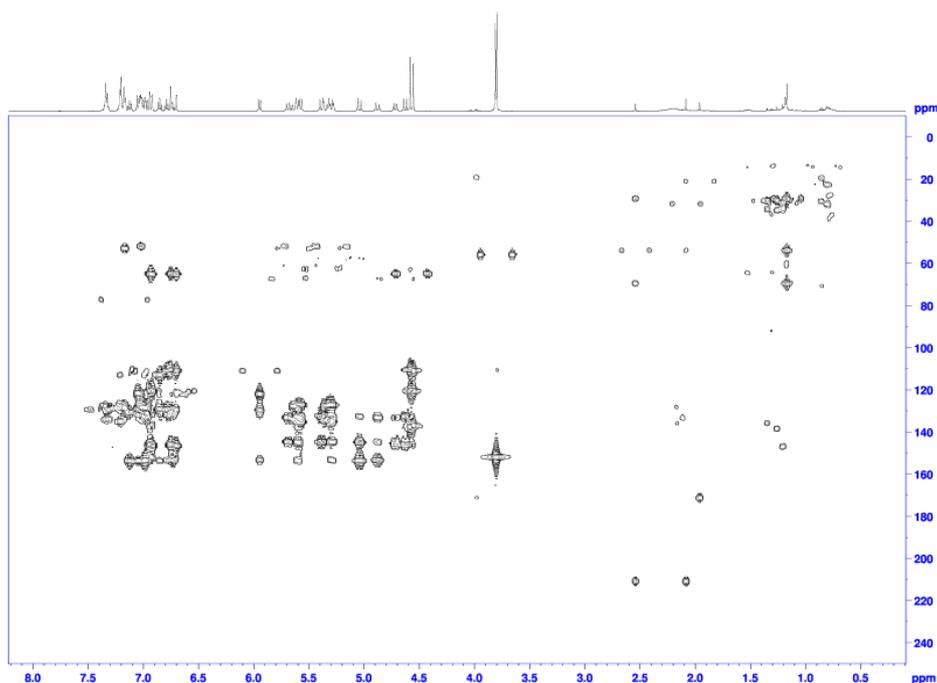
F2 - Processing parameters
SI      1024
SF      500.1300549 MHz
WDW     0
SSB     0
LB      0 Hz
GB      0
PC      1.40

F1 - Processing parameters
SI      1024
SF      500.1300549 MHz
WDW     0
SSB     0
LB      0 Hz
GB      0

```

# HMBC:

HMBC.w CDC13 /opt/topspin3.2 AM 48



```

Current Data Parameters
NAME: 4808-2017
EXPNO: 13
PROCNO: 1

F2 - Acquisition Parameters
Date_: 2017038
Time: 16.31
INSTRUM: spect
PROBHD: 5 mm CPDCH 13C
PULPROG: zgpg30
TD: 1024
SOLVENT: CDCl3
NS: 8
DS: 16
SWH: 4003.241 Hz
FIDRES: 3.009766 Hz
AQ: 0.1219520 sec
RG: 185.00
SQ: 131.000 usec
TE: 300.2 K
CMT2: 145.000000 sec
D1: 0.0000000 sec
D11: 1.44982398 sec
D2: 0.0000000 sec
D4: 0.0000000 sec
D15: 0.0000000 sec
INVD: 0.0001830 sec

----- CHANNEL f1 -----
NUC1: 500.1321341 MHz
P1: 18
P2: 9.10 usec
P3: 18.20 usec
PL1: 33.0000000 W

----- CHANNEL f2 -----
NUC2: 13C
P3: 9.30 usec
P4: 24.0000000 W

----- GRADIENT CHANNEL -----
GPMAX[1]: 8M02.100
GPMAX[2]: 8M02.100
GPMAX[3]: 8M02.100
GPI1: 30.00 W
GPI2: 30.00 W
GPI3: 40.00 W
P15: 1000.00 usec

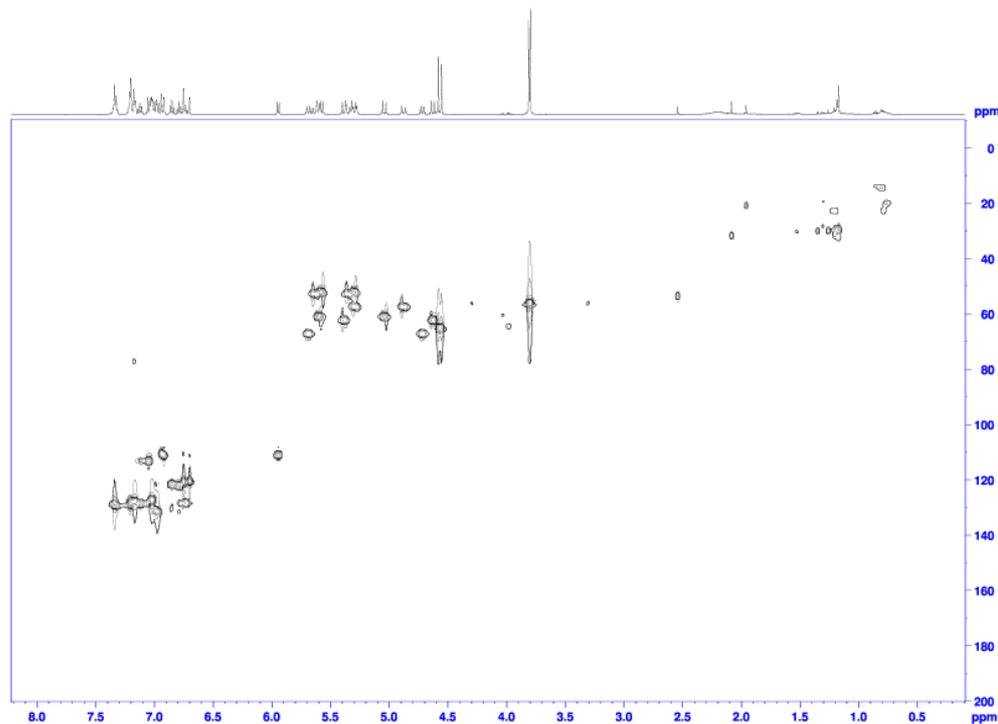
F1 - Acquisition parameters
TD: 138
SF: 125.767785 MHz
F2RES: 230.410413 Hz
SW: 210.431 ppm
FWD000: Echo-AntiEcho

F2 - Processing parameters
SI: 32768
SF: 500.1321341 MHz
WDW: EM
SSB: 0
GB: 0 Hz
PC: 1.40

F1 - Processing parameters
SI: 1024
SF: 125.767785 MHz
WDW: EM
SSB: 0
GB: 0 Hz
PC: 0
    
```

# HSQC:

Chemist Anish Mistry  
AM-60 pure  
HSQC.w CDC13 /opt/topspin3.2 AM 48



```

Current Data Parameters
NAME: 4808-2017
EXPNO: 13
PROCNO: 1

F2 - Acquisition Parameters
Date_: 2017038
Time: 16.16
INSTRUM: spect
PROBHD: 5 mm CPDCH 13C
PULPROG: zgpg30
TD: 1024
SOLVENT: CDCl3
NS: 8
DS: 16
SWH: 4003.241 Hz
FIDRES: 3.009766 Hz
AQ: 0.1219520 sec
RG: 185.00
SQ: 131.000 usec
TE: 300.2 K
CMT2: 145.000000 sec
D1: 0.0000000 sec
D11: 1.44982398 sec
D2: 0.0000000 sec
D4: 0.0000000 sec
D15: 0.0000000 sec
INVD: 0.0001830 sec

----- CHANNEL f1 -----
NUC1: 500.1321341 MHz
P1: 18
P2: 9.10 usec
P3: 18.20 usec
PL1: 33.0000000 W

----- CHANNEL f2 -----
NUC2: 13C
P3: 9.30 usec
P4: 24.0000000 W

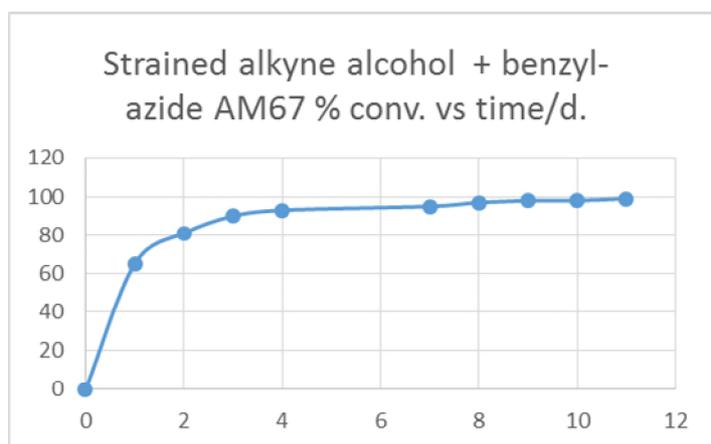
----- GRADIENT CHANNEL -----
GPMAX[1]: 8M02.100
GPMAX[2]: 8M02.100
GPMAX[3]: 8M02.100
GPI1: 30.00 W
GPI2: 30.00 W
GPI3: 40.00 W
P15: 1000.00 usec

F1 - Acquisition parameters
TD: 138
SF: 125.767785 MHz
F2RES: 230.410413 Hz
SW: 210.431 ppm
FWD000: Echo-AntiEcho

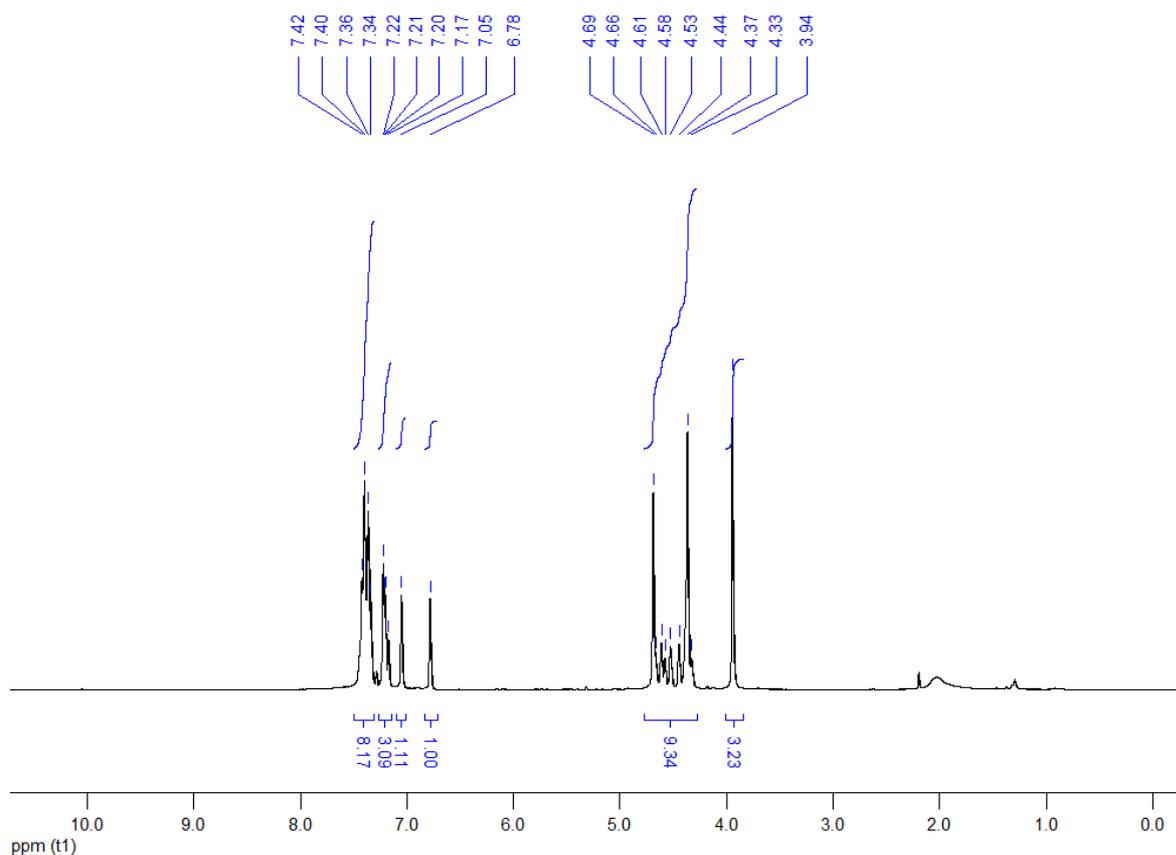
F2 - Processing parameters
SI: 32768
SF: 500.1321341 MHz
WDW: EM
SSB: 0
GB: 0 Hz
PC: 1.40

F1 - Processing parameters
SI: 1024
SF: 125.767785 MHz
WDW: EM
SSB: 0
GB: 0 Hz
PC: 0
    
```

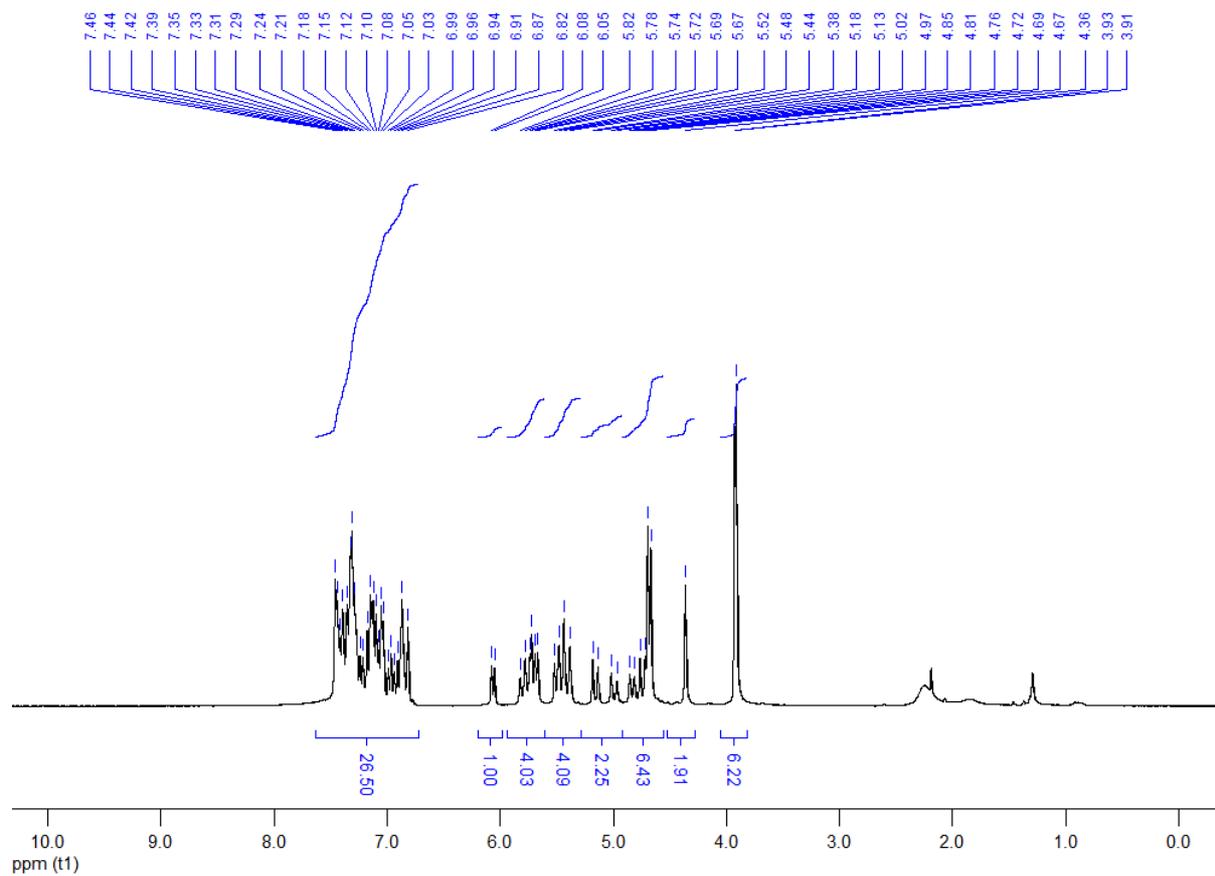
AM67 Is conversion over time; some error likely due to overlaps but start and finish NMRs show extent of conversion. Characteristic peaks were the OCH<sub>2</sub> and NCH<sub>2</sub> peaks of the product and the OCH<sub>2</sub> peaks of the strained alkyne. Day 1; 65%, day 2; 81%, day 3; 90%, day 4; 93%, day 7; 95%, day 8; 97%, day 9; 98%, day 10; 98%, day 11; 99% conversion.



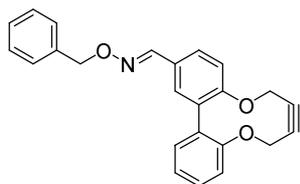
NMR at start (day 0):  $\delta_H$  (300 MHz, CDCl<sub>3</sub>).



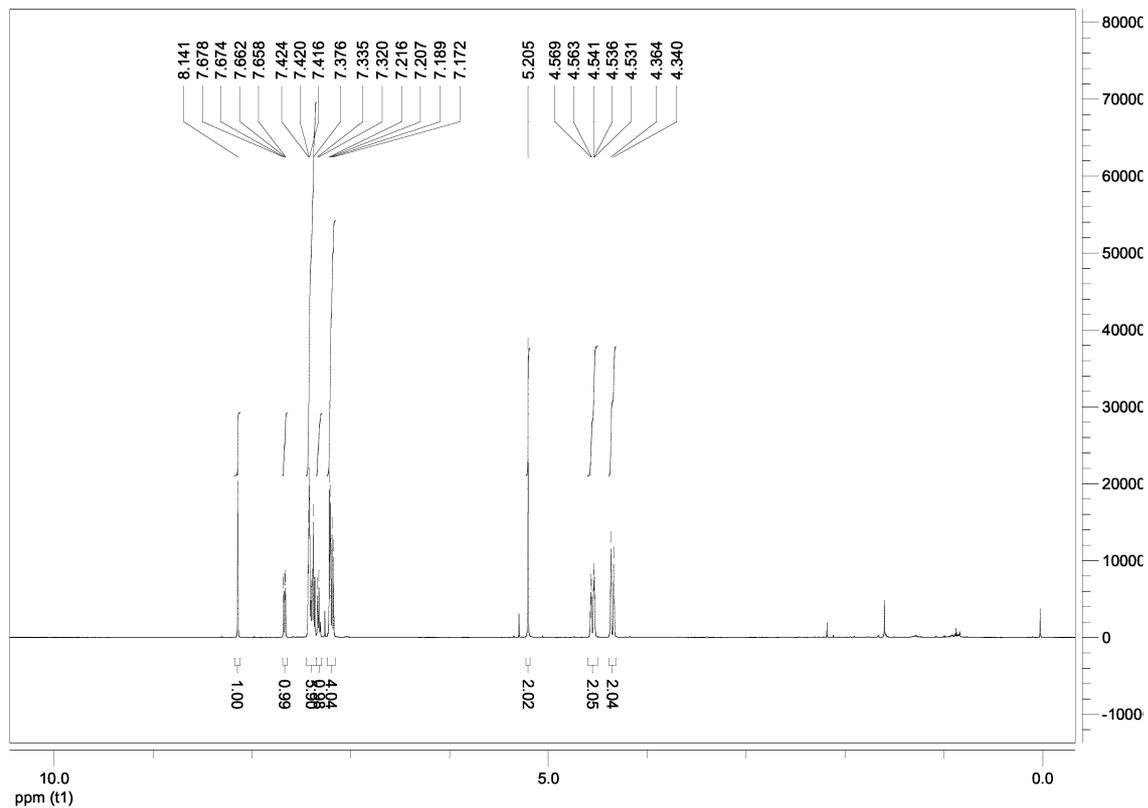
NMR at end (day 11); ca. 99% conversion:  $\delta_H$  (300 MHz,  $CDCl_3$ ).



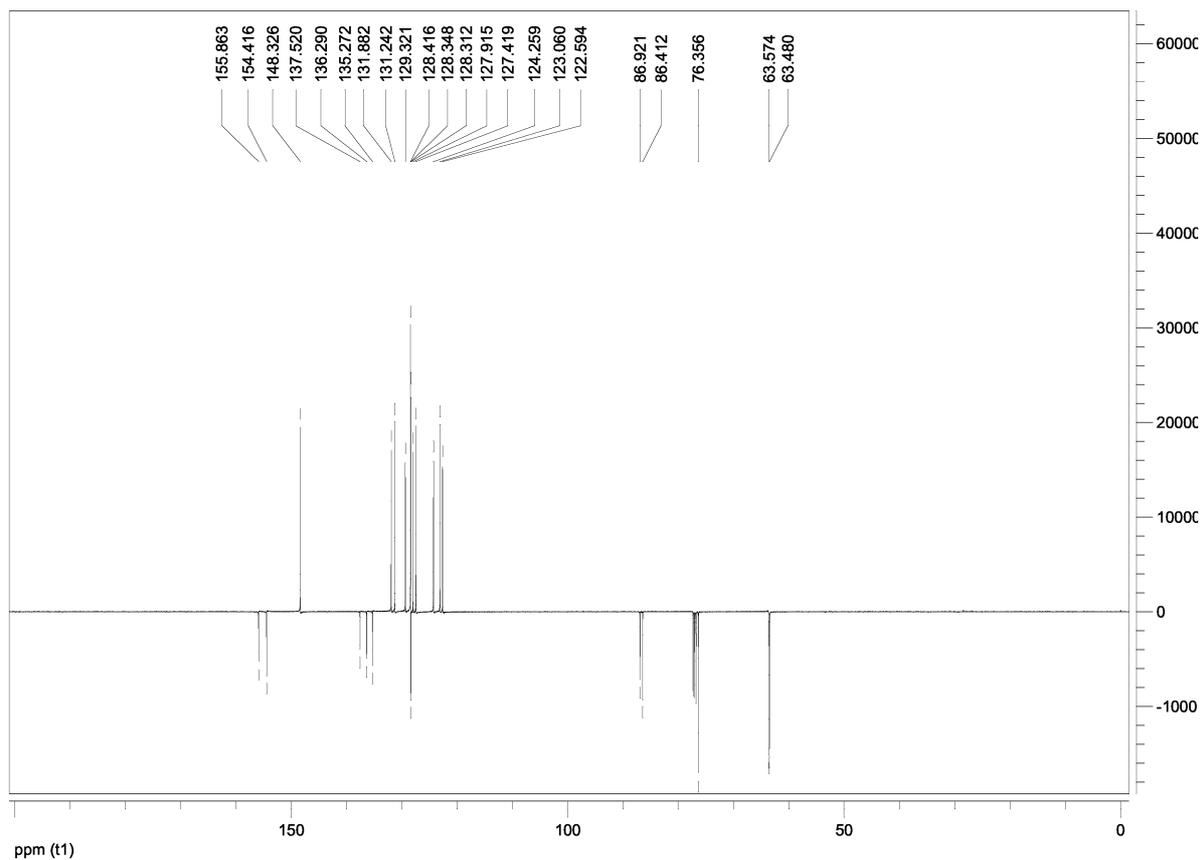
**Oxime ether 29 SF271.**



$\delta_H$  (500 MHz,  $CDCl_3$ ).



$\delta_C$  (126 MHz, CDCl<sub>3</sub>).



COSY.

Chemist Sam Forshaw  
SF271  
COSY.w CDCl<sub>3</sub> /opt/topspin3.2 SF1 57



```

Current Data Parameters
NAME: Mar15-2018
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters
Date_ 20180315
Time 23.22
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1
DS 8
SWH 4973.475 Hz
FIDRES 2.428404 Hz
AQ 0.2058933 sec
RG 37.48
DM 100.533 usec
DE 40.00 usec
TE 298.0 K
D0 0.0000000 sec
D1 1.53448398 sec
D11 0.0000000 sec
D12 0.0000000 sec
D13 0.0000000 sec
D16 0.0000000 sec
IND 0.00020140 sec

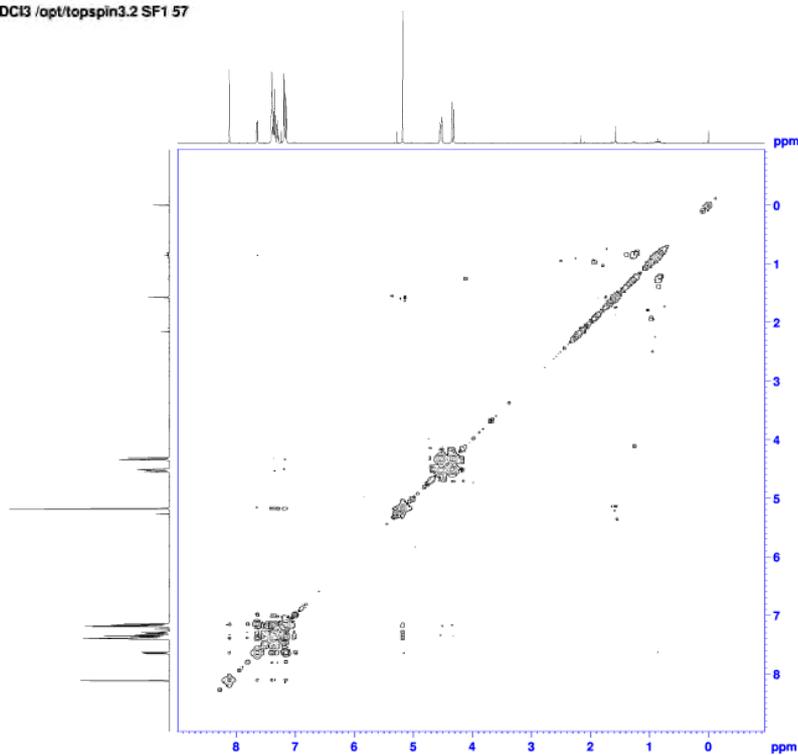
----- CHANNEL f1 -----
NUC1 13C
P1 9.10 usec
P14 2500.00 usec
PL1 13.00000000 W
PL10 1.5243993 W

----- GRADIENT CHANNEL -----
GPMAX[1] 25000.100
USE1 10.00 %
P14 1000.00 usec

F1 - Acquisition parameters
TD 65536
SFO1 500.132 MHz
FIDRES 24.795662 Hz
SW 9.928 PPM
F0BASE0 0

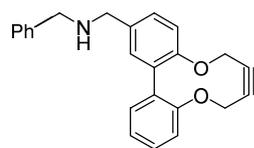
F2 - Processing parameters
SI 32768
SF 500.1302423 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 32768
SF 500.1302423 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.40
    
```

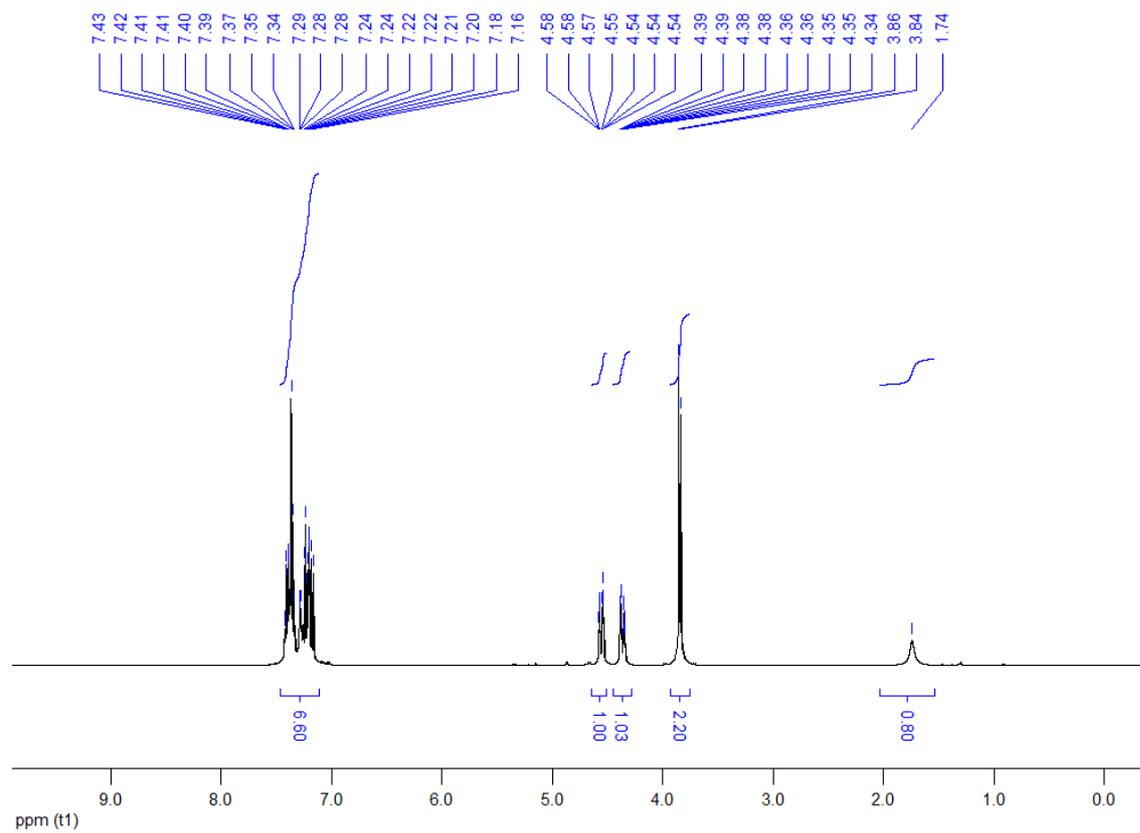




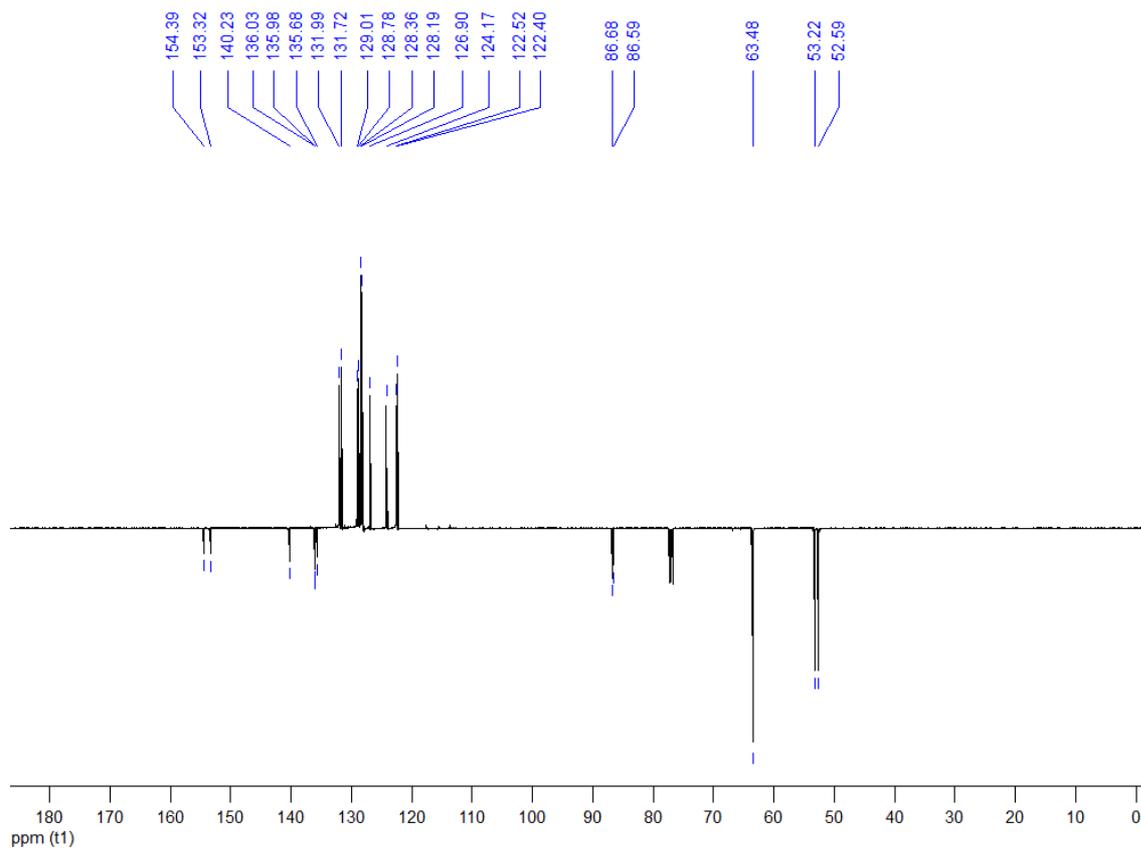
### Benzyl amine derivative 30. AM66.



$\delta_H$  (500 MHz,  $CDCl_3$ ).

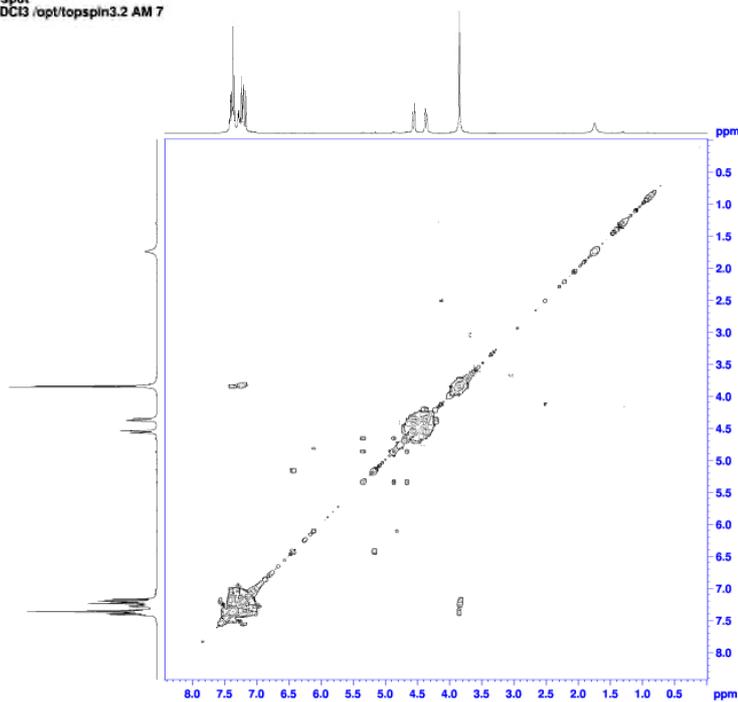


$\delta_C$  (126 MHz, CDCl<sub>3</sub>).



COSY:

Chemist Arish Mistry  
AM-68-1st spot  
COSY.w CDCl<sub>3</sub> /opt/topspin3.2 AM 7



```

Current Data Parameters
NAME      Pub00-2017
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20170220
Time     12.36
INSTRUM  spect
PROBHD   5 mm CPDQX 1.3C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1
DS        8
SWH       4722.973 Hz
FIDRES    2.061889 Hz
AQ        0.242433 sec
RG         42.9
SWH       133.400 usec
DE        40.00 usec
TE        298.2 K
D0        0.0000000 sec
D1        5.8930402 sec
D11       0.0300000 sec
D12       0.0000000 sec
D13       0.0000000 sec
D14       0.0000000 sec
D15       0.0000000 sec
D16       0.0000000 sec
D17       0.0000000 sec
D18       0.0000000 sec
D19       0.0000000 sec
D20       0.0000000 sec

===== CHANNEL f1 =====
NUC1      13C
P1        9.10 usec
PL1       0 dB
PC1       13.0000000 W
P1M10    1.5924999 W
P1M10    1.5924999 W

===== GRABINQ CHANNEL =====
SFO1      500.1321053 Mhz
NUC1      13C
P1        9.10 usec
PL1       0 dB
PC1       13.0000000 W
P1M10    1.5924999 W
P1M10    1.5924999 W

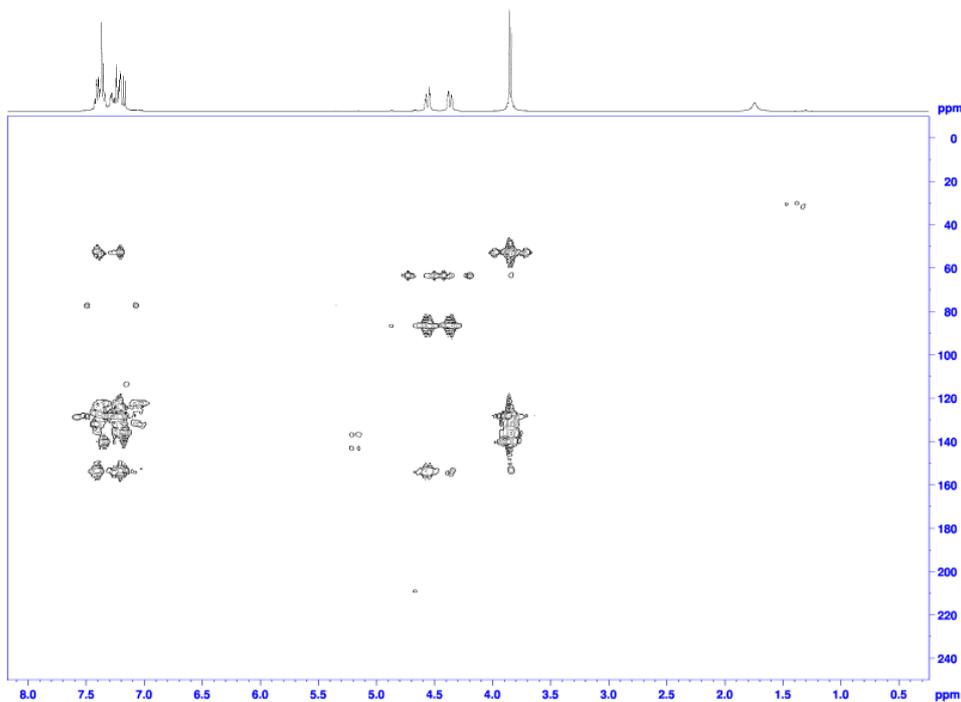
F1 - Acquisition parameters
IS        1624
SFO1      500.1321 Mhz
FIDRES    32.844134 Hz
SWH       8.417 ppm
FOCUS     0 F

F2 - Processing parameters
SI        1624
SF        500.1300000 Mhz
WDW       0
SSB       0 Hz
LB        0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1624
SFO1      500.1300000 Mhz
WDW       0
SSB       0 Hz
LB        0 Hz
GB        0
PC        1.40
    
```

# HMBC:

Chemist Anish Mistry  
AM-65-1st spot  
HMBC.w CDC13 /opt/topspin3.2 AM 7



```
Current Data Parameters
NAME      F6020-2011
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20110223
Time     18.57
INSTRUM  spect
PROBHD   5 mm CPDCH 13
PULPROG  zgpg30
TD        1024
SOLVENT  CDCl3
NS        16
DS        4
SWH       3962.154 Hz
FIDRES    0.187548 Hz
AQ        0.132024 sec
RG         382
WDW       EM
SSB       0
LB        134.000 usec
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec

----- CHANNEL f1 -----
SFO1     500.1321053 MHz
NUC1     13
P1        9.10 usec
P2        18.20 usec
PL1       0.00000000 dB
PL2       13.00000000 dB

----- CHANNEL f2 -----
SFO2     125.7679125 MHz
NUC2     13C
P3        9.10 usec
P4        18.20 usec
PL3       0.00000000 dB
PL4       13.00000000 dB

----- CHANNEL CHANNEL -----
CPDPRG1  smgq10.100
CPDPRG2  smgq10.100
CPDPRG3  smgq10.100
CPDPRG4  smgq10.100
CPDPRG5  smgq10.100
CPDPRG6  smgq10.100
P16      1000.00 usec

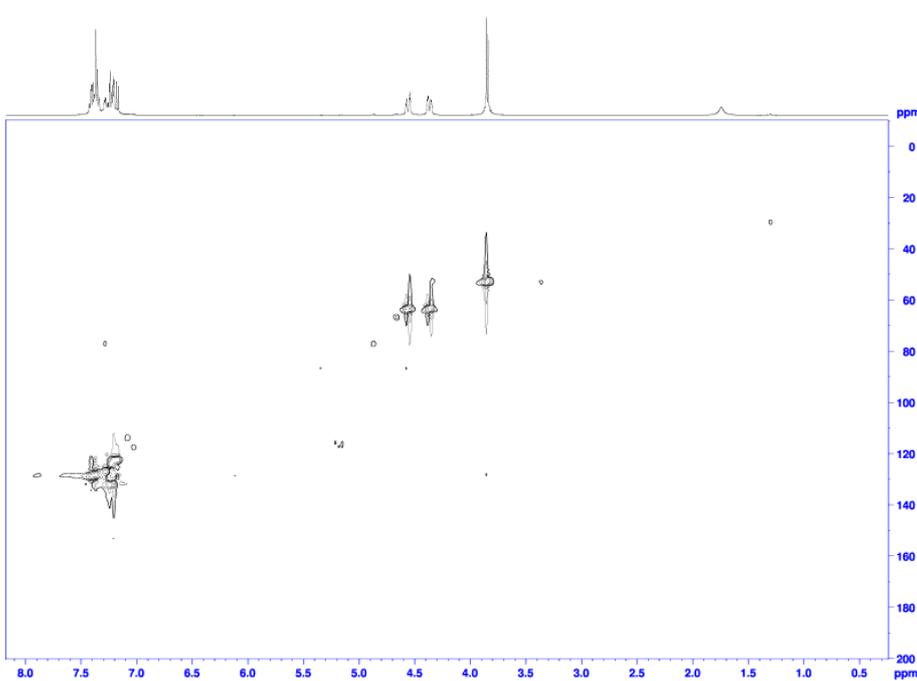
F1 - Acquisition parameters
SI        32768
SF        500.1321053 MHz
WDW       EM
SSB       0
LB        0 Hz
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec

F2 - Processing parameters
SI        32768
SF        500.1321053 MHz
WDW       EM
SSB       0
LB        0 Hz
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec

F1 - Processing parameters
SI        32768
SF        125.7679125 MHz
WDW       EM
SSB       0
LB        0 Hz
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec
```

# HSQC:

HSQC.w CDC13 /opt/topspin3.2 AM 7



```
Current Data Parameters
NAME      F6020-2011
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20110223
Time     18.52
INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  zgpg30
TD        1024
SOLVENT  CDCl3
NS        16
DS        4
SWH       3962.154 Hz
FIDRES    0.187548 Hz
AQ        0.132024 sec
RG         382
WDW       EM
SSB       0
LB        134.000 usec
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec

----- CHANNEL f1 -----
SFO1     500.1321053 MHz
NUC1     13
P1        9.10 usec
P2        18.20 usec
PL1       0.00000000 dB
PL2       13.00000000 dB

----- CHANNEL f2 -----
SFO2     125.7679125 MHz
NUC2     13C
P3        9.10 usec
P4        18.20 usec
PL3       0.00000000 dB
PL4       13.00000000 dB

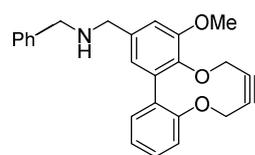
----- CHANNEL CHANNEL -----
CPDPRG1  smgq10.100
CPDPRG2  smgq10.100
CPDPRG3  smgq10.100
CPDPRG4  smgq10.100
CPDPRG5  smgq10.100
CPDPRG6  smgq10.100
P16      1000.00 usec

F1 - Acquisition parameters
SI        32768
SF        500.1321053 MHz
WDW       EM
SSB       0
LB        0 Hz
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec

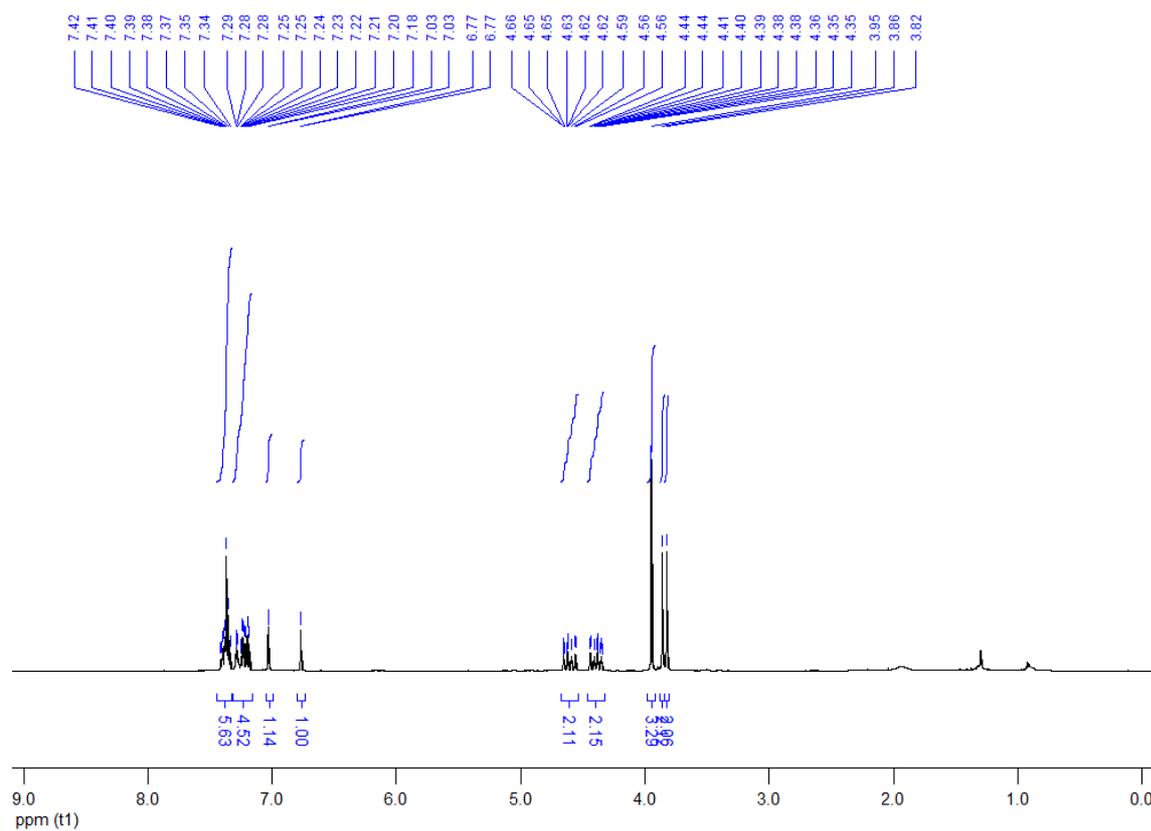
F2 - Processing parameters
SI        32768
SF        500.1321053 MHz
WDW       EM
SSB       0
LB        0 Hz
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec

F1 - Processing parameters
SI        32768
SF        125.7679125 MHz
WDW       EM
SSB       0
LB        0 Hz
GB         0
TE        298.2 K
CMT2      149.000000
DE        0.00000000
DI        1.44675130 usec
DQ        0.00000000 usec
DR        0.00000000 usec
DL        0.00000000 usec
DU        0.00000000 usec
DV        0.00000000 usec
IN0       0.0001330 usec
```

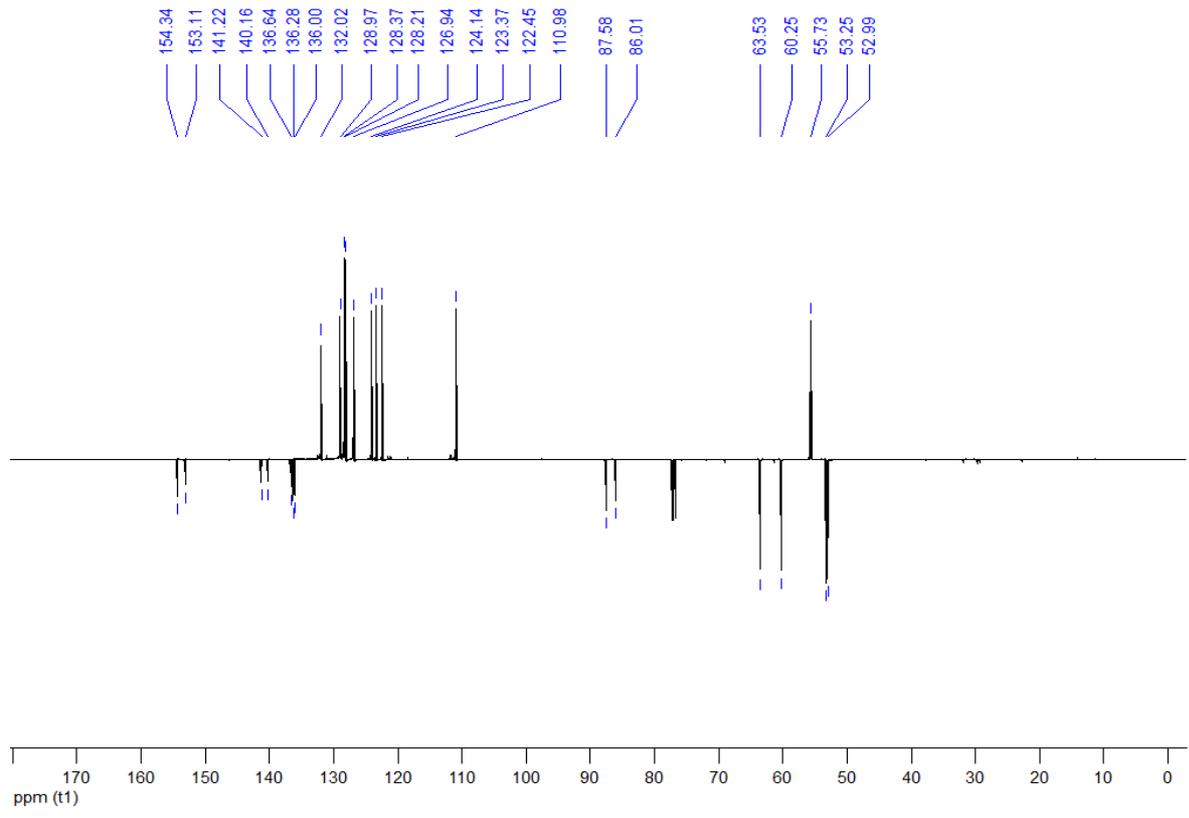
### Benzyl amine derivative 31. AM70



$\delta_H$  (500 MHz,  $CDCl_3$ ).

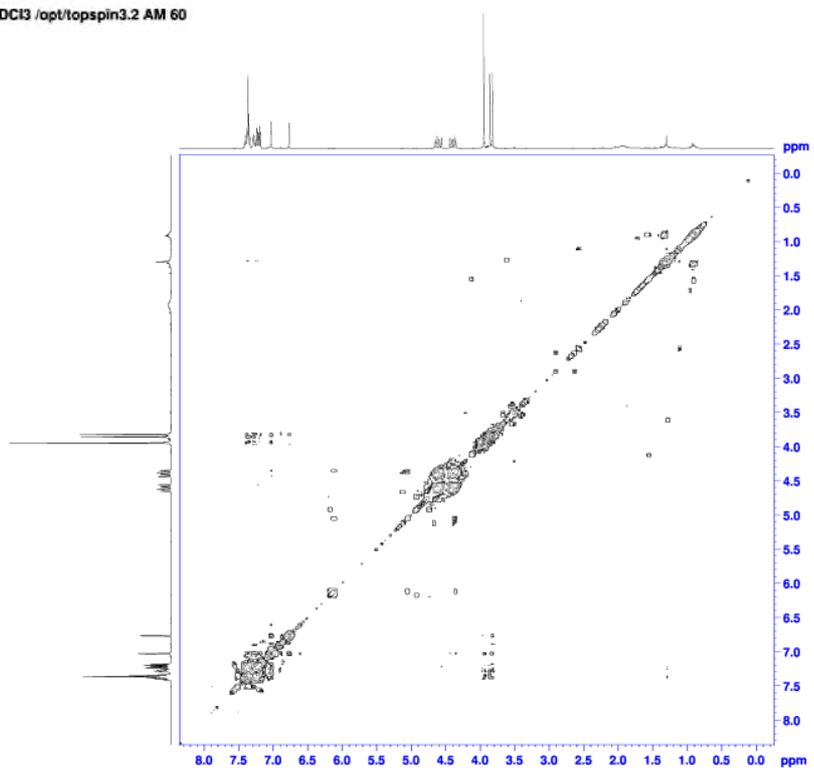


$\delta_C$  (126 MHz,  $CDCl_3$ ).



COSY:

Chemist Anish Mistry  
AM-70  
COSY.w  $CDCl_3$  /opt/topspin3.2 AM 60

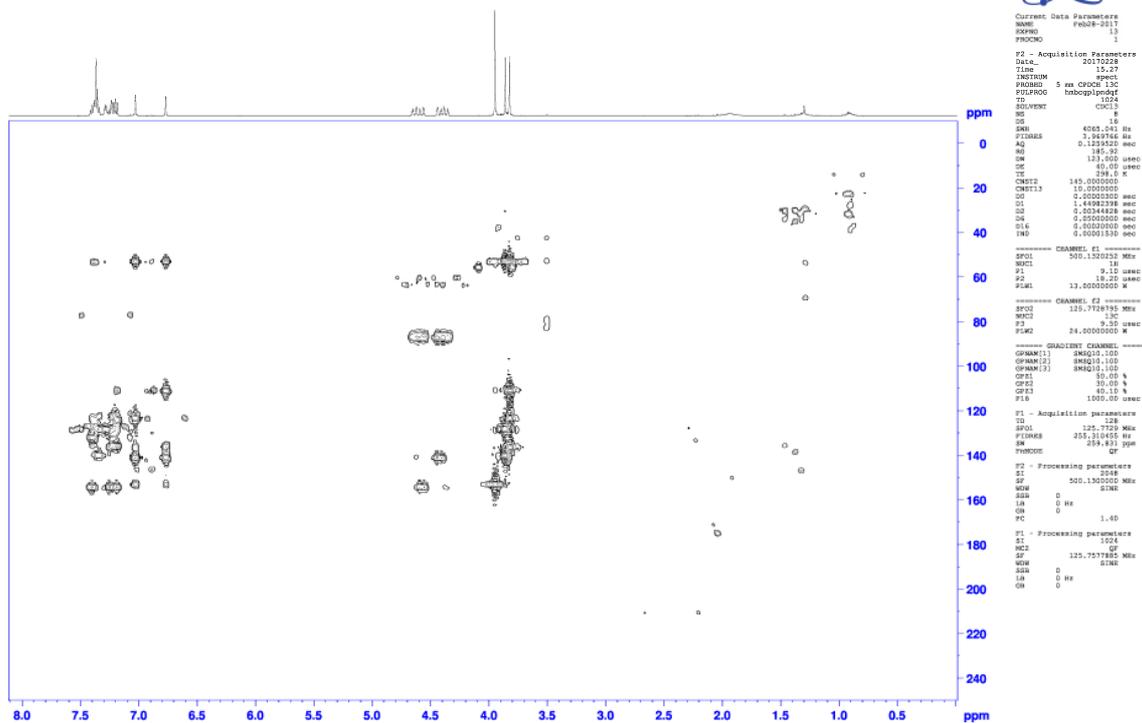


```

Current Data Parameters
NAME          Feb28-2017
EXPNO        11
PROCNO       1
F2 - Acquisition Parameters
Date_        20170228
Time         15.06
INSTRUM      spect
PROBHD       5 mm CPDPC130
PULPROG      zgpg30
SOLVENT      CDCl3
NS           5
DS           2
SWH          4310.345 Hz
FIDRES       2.104661 Hz
AQ           0.2375880 sec
RG           31.55
CW           116.000 usec
DE           40.00 usec
TE           298.0 K
D0           0.0000000 sec
D1           1.8984795 sec
D11          0.5000000 sec
D12          0.0000000 sec
D13          0.0000000 sec
D16          0.0000000 sec
ZWD          0.0003180 sec
----- CHANNEL f1 -----
SFO1        500.130252 MHz
NUC1         13
P1           9.10 usec
P1Y         2800.00 usec
PL1         13.0000000 W
PL12        1.5924999 W
----- GRADIENT CHANNEL -----
GPRM1[1]    SFOG01.100
GPR1        10.00 %
P16         1000.00 usec
F1 - Acquisition parameters
TD           500
SFO1        500.130
FIDRES       33.703625 Hz
SW           4.024 ppm
FAMODE       GP
F2 - Processing parameters
SI           3274
SF           500.1300000 MHz
WDW          GEMME
SSB          0
LB           0 Hz
GB           0
PC           1.40
F1 - Processing parameters
SI           3274
SF           500.1300000 MHz
WDW          GEMME
SSB          0
LB           0 Hz
GB           0
  
```

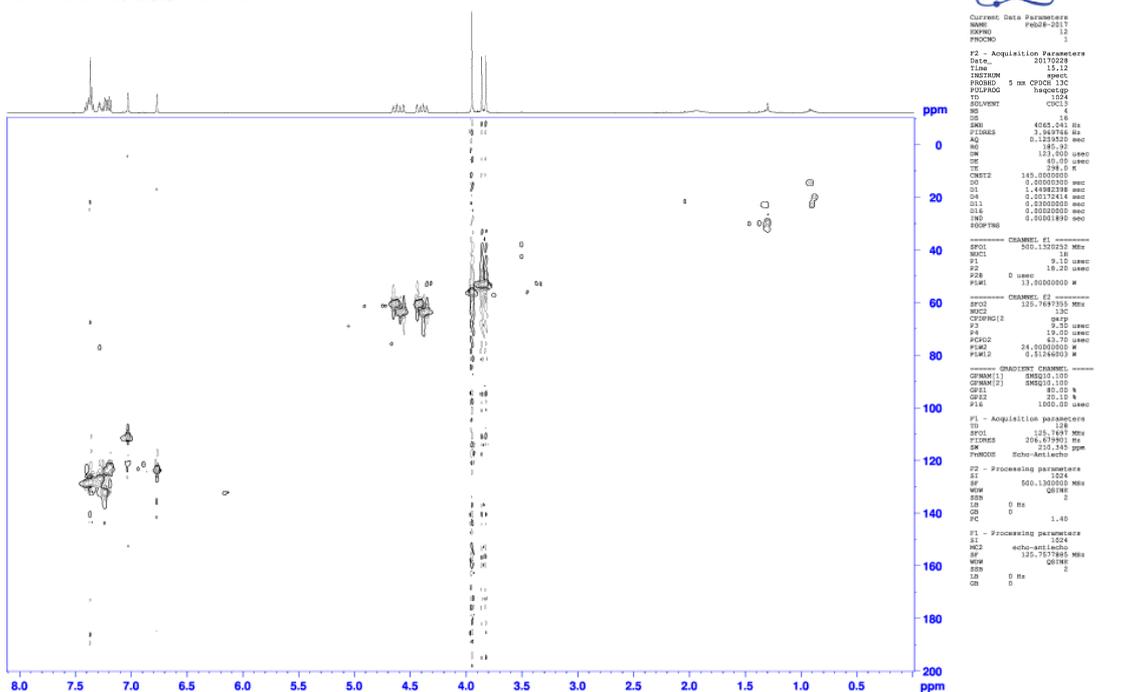
# HMBC:

Chemist Anish Mistry  
AM-70  
HMBC.w CDCI3 /opt/topspin3.2 AM 60

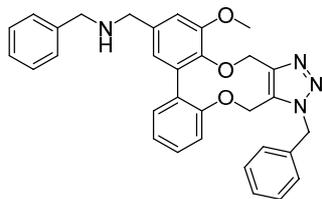


# HSQC:

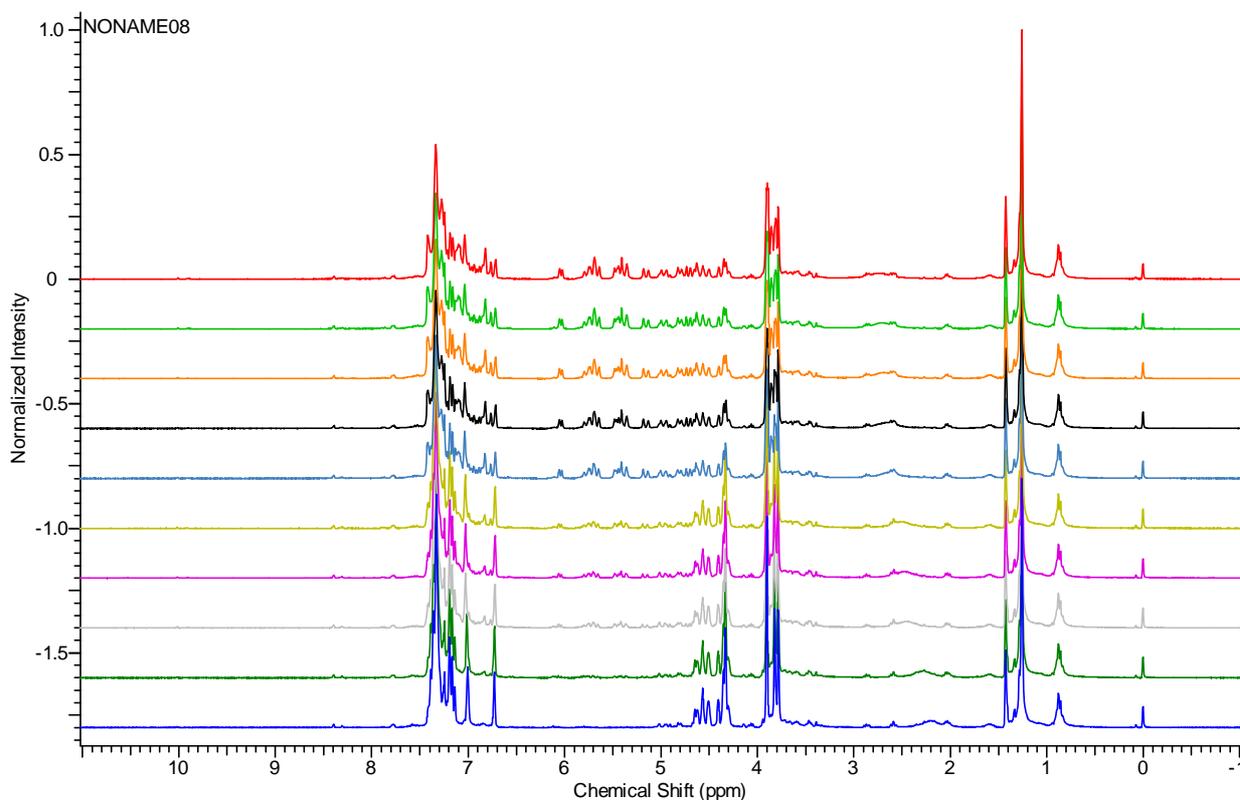
Chemist Anish Mistry  
AM-70  
HSQC.w CDCI3 /opt/topspin3.2 AM 60



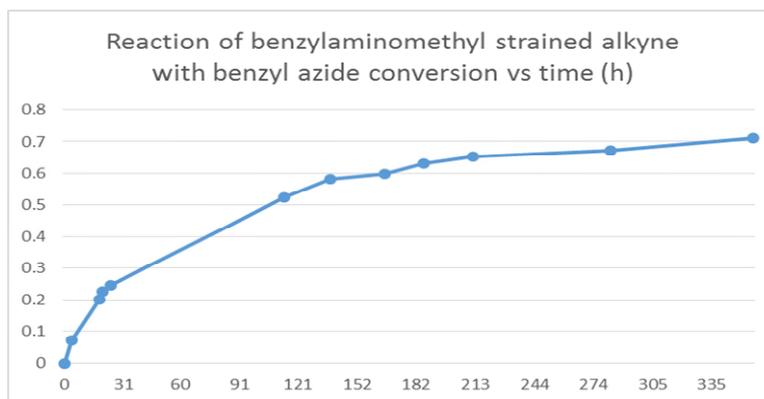
**Cycloaddition to give the compound below (mixture of isomers) SF224.**



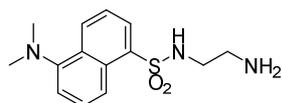
To a solution of alkyne **16** (16 mg, 0.041mmol, 1 eq) in  $\text{CDCl}_3$  (0.5 mL), benzylazide **20** (5.8 mg, 5.4  $\mu\text{L}$ , 0.051 mmol, 1.2 eq) was added and the reaction was followed by NMR. The product was not isolated.



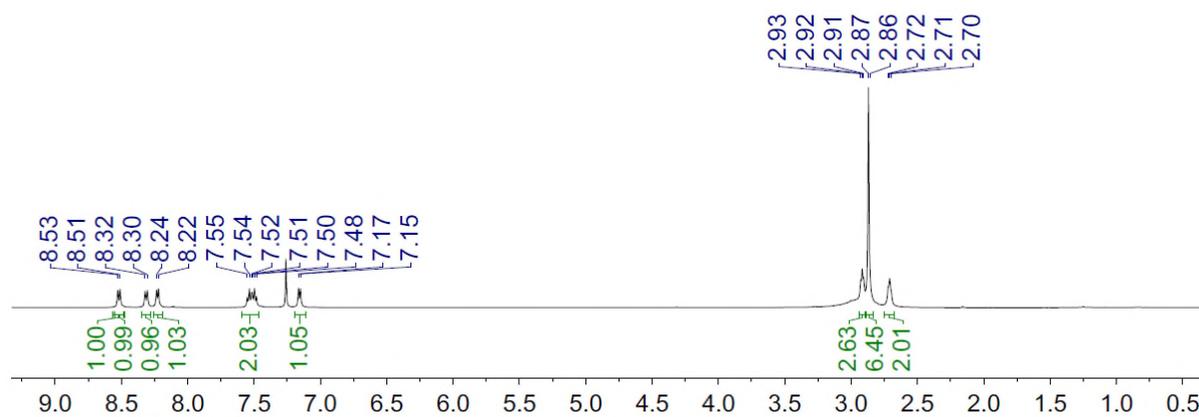
Times are 1) 0 h, 2) 4h , 3) 18 h, 4) 20.75 h, 5) 24 h, 6) 114.5 h, 7) 138.5 h, 8) 166.5 h, 9) 186.5 h, 10) 212 h, 13) 283 h.



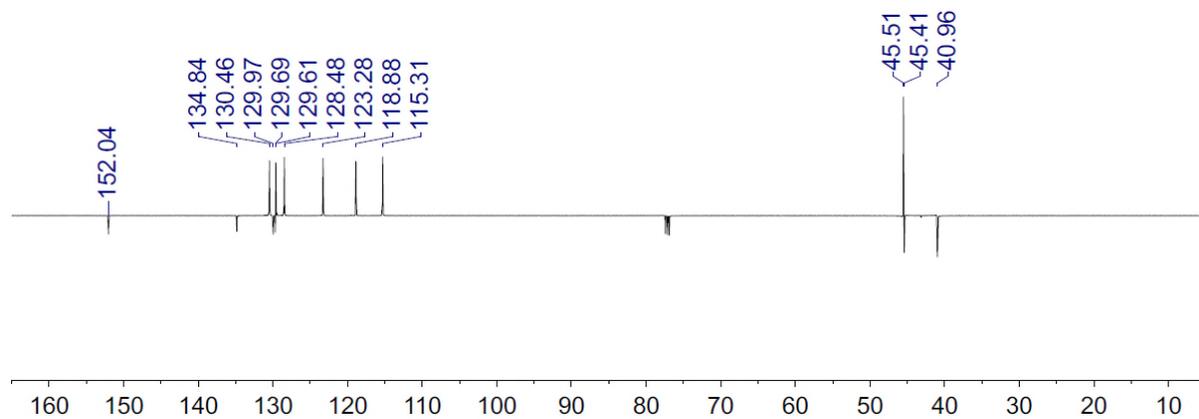
**Dansyl amine precursor 34 (RK-2-182):**



$\delta_H$  (500 MHz,  $CDCl_3$ ).

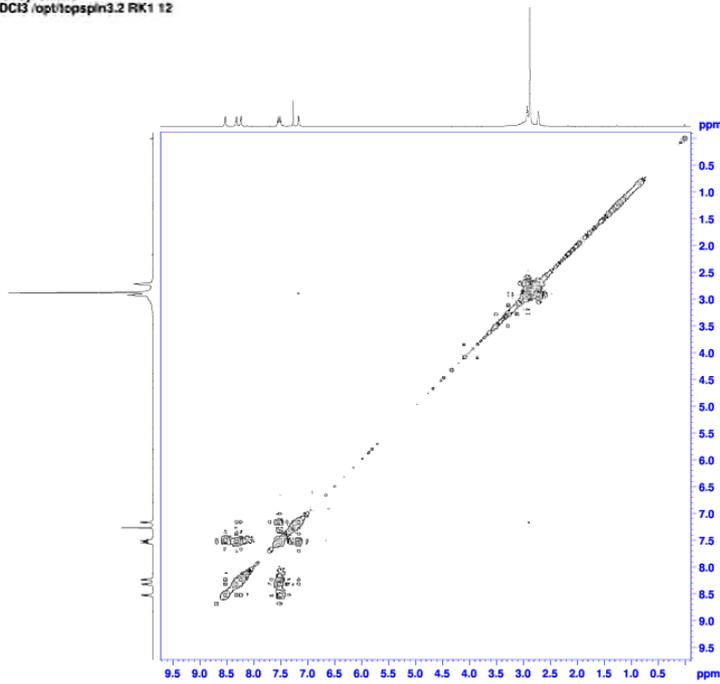


$\delta_C$  (126 MHz,  $CDCl_3$ ).



# COSY.

Chemist Richard Knighton  
 RK-2-135 dansyl amine  
 COSY.w CDCI3 /opt/topspin3.2 RK1 12



```
Current Data Parameters
NAME      Aug10-2011
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20110810
Time     8.09
INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        4
DS        4
SFO1     401.248 MHz
FIDRES   0.462858 Hz
AQ        0.1000765 sec
RG         7.41
DM         161.600 usec
DE         40.00 usec
TE        298.2 K
D0         0.0000000 sec
D1         1.9381596 sec
D11        0.0000000 sec
D12        0.0000000 sec
D13        0.0000000 sec
D14        0.0000000 sec
D15        0.0000000 sec
D16        0.0000000 sec
D17        0.0000000 sec
D18        0.0000000 sec
D19        0.0000000 sec

===== CHANNEL f1 =====
SFO1     500.1324140 MHz
NUC1      13
P1         9.10 usec
P11        18.20 usec
P12        2500.00 usec
P13        13.00000000 W
P14        1.39489997 W
PL1WD0

===== GRABF2 CHANDEL =====
SFO2     101.6250000 MHz
P2         9.10 usec
P21        18.20 usec

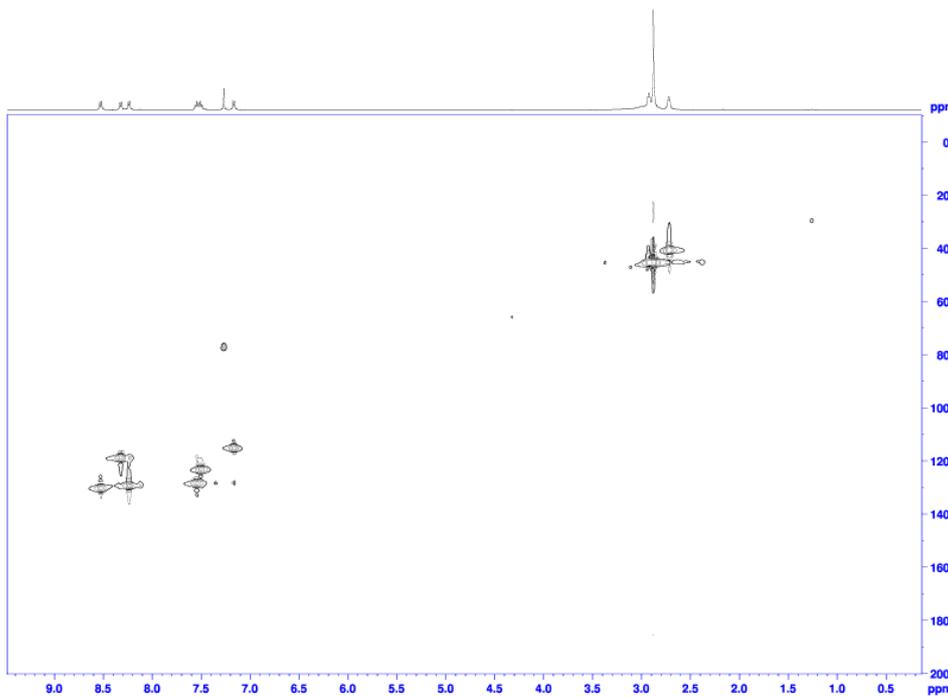
F1 - Acquisition parameters
TD        65536
SFO1     500.1324140 MHz
FIDRES   0.462858 Hz
AQ        0.1000765 sec
RG         7.41
DM         161.600 usec
DE         40.00 usec
TE        298.2 K
D0         0.0000000 sec
D1         1.9381596 sec
D11        0.0000000 sec
D12        0.0000000 sec
D13        0.0000000 sec
D14        0.0000000 sec
D15        0.0000000 sec
D16        0.0000000 sec
D17        0.0000000 sec
D18        0.0000000 sec
D19        0.0000000 sec

F1 - Processing parameters
SI        32768
SF        500.1300000 MHz
WDW       EM
SSB       0 Hz
GB         0
PC         1.40

F2 - Processing parameters
SI        16384
SF        500.1300000 MHz
WDW       EM
SSB       0 Hz
GB         0
PC         1.40
```

# HMQC.

Chemist Richard Knighton  
 RK-2-135 dansyl amine  
 HSQC.w CDCI3 /opt/topspin3.2 RK1 12



```
Current Data Parameters
NAME      Aug10-2011
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20110810
Time     8.14
INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        4
DS        4
SFO1     401.248 MHz
FIDRES   0.462858 Hz
AQ        0.1000765 sec
RG         7.41
DM         161.600 usec
DE         40.00 usec
TE        298.2 K
D0         0.0000000 sec
D1         1.9381596 sec
D11        0.0000000 sec
D12        0.0000000 sec
D13        0.0000000 sec
D14        0.0000000 sec
D15        0.0000000 sec
D16        0.0000000 sec
D17        0.0000000 sec
D18        0.0000000 sec
D19        0.0000000 sec

===== CHANNEL f1 =====
SFO1     500.1324140 MHz
NUC1      13
P1         9.10 usec
P11        18.20 usec
P12        2500.00 usec
P13        13.00000000 W
P14        1.39489997 W
PL1WD0

===== CHANNEL f2 =====
SFO2     101.6250000 MHz
P2         9.10 usec
P21        18.20 usec
P22        0 usec
P23        13.00000000 W
PL2WD0

===== GRABF2 CHANDEL =====
SFO2     101.6250000 MHz
P2         9.10 usec
P21        18.20 usec

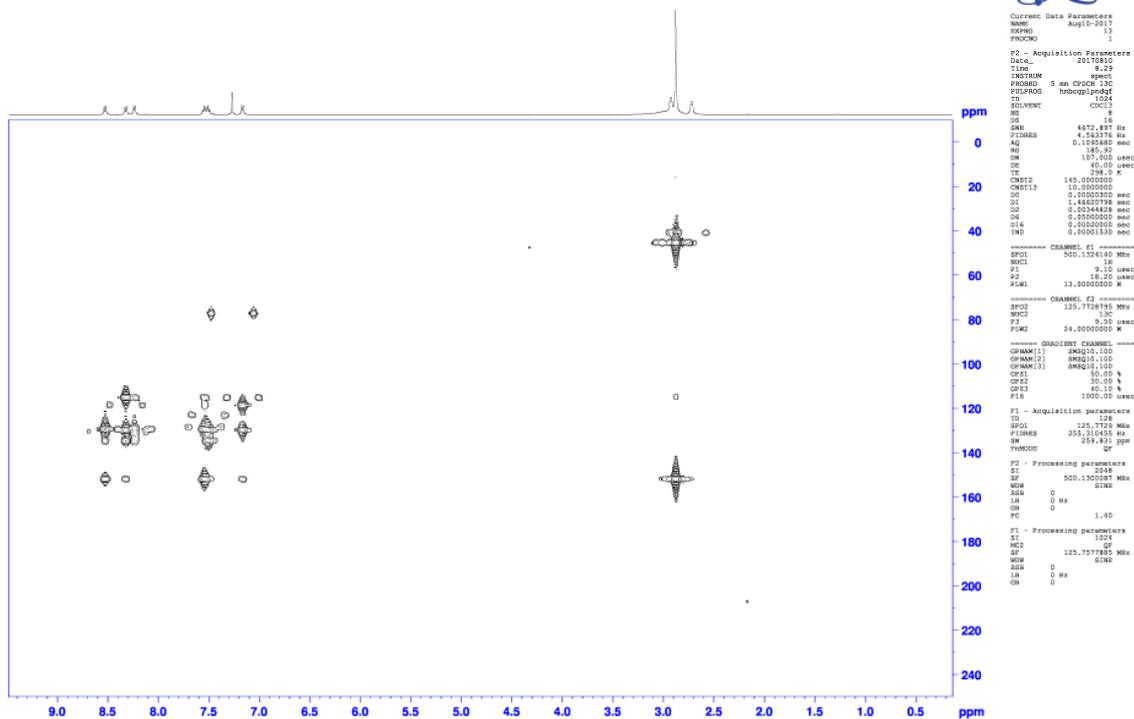
F1 - Acquisition parameters
TD        65536
SFO1     500.1300000 MHz
FIDRES   0.462858 Hz
AQ        0.1000765 sec
RG         7.41
DM         161.600 usec
DE         40.00 usec
TE        298.2 K
D0         0.0000000 sec
D1         1.9381596 sec
D11        0.0000000 sec
D12        0.0000000 sec
D13        0.0000000 sec
D14        0.0000000 sec
D15        0.0000000 sec
D16        0.0000000 sec
D17        0.0000000 sec
D18        0.0000000 sec
D19        0.0000000 sec

F1 - Processing parameters
SI        32768
SF        500.1300000 MHz
WDW       EM
SSB       0 Hz
GB         0
PC         1.40

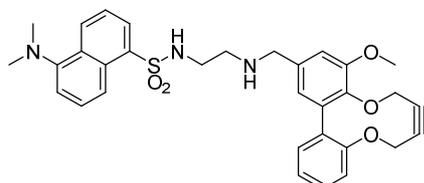
F2 - Processing parameters
SI        16384
SF        500.1300000 MHz
WDW       EM
SSB       0 Hz
GB         0
PC         1.40
```

HMBC.

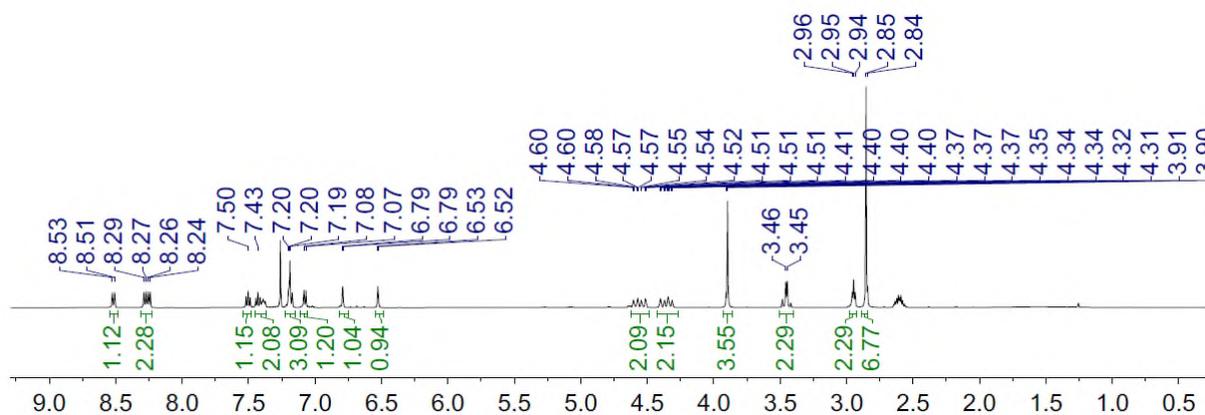
HMBC.w CDCl<sub>3</sub> /opt/topspin3.2 RK1 12  
nstyl amine



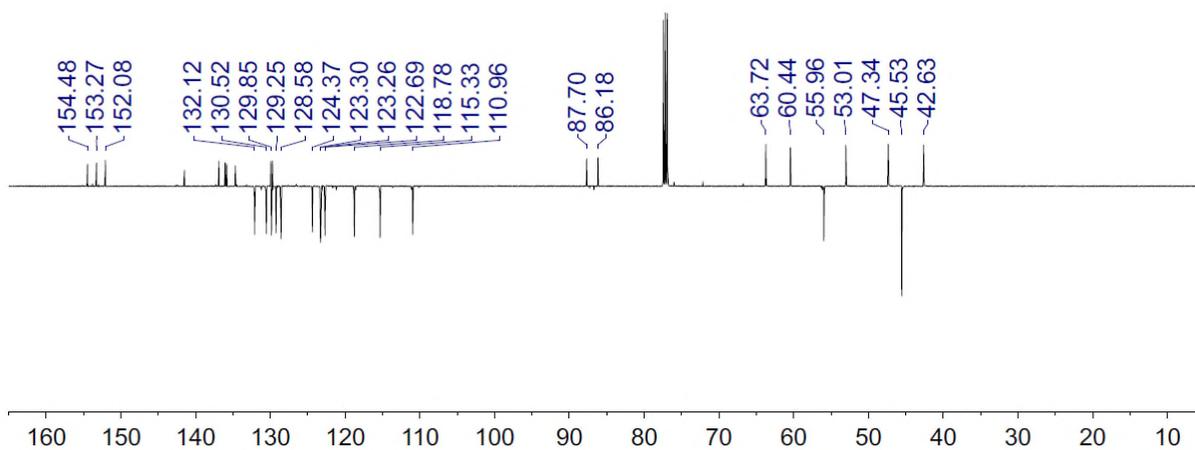
**Dansyl strained alkyne 35 (RK-2-184):**



$\delta_H$  (500 MHz, CDCl<sub>3</sub>).

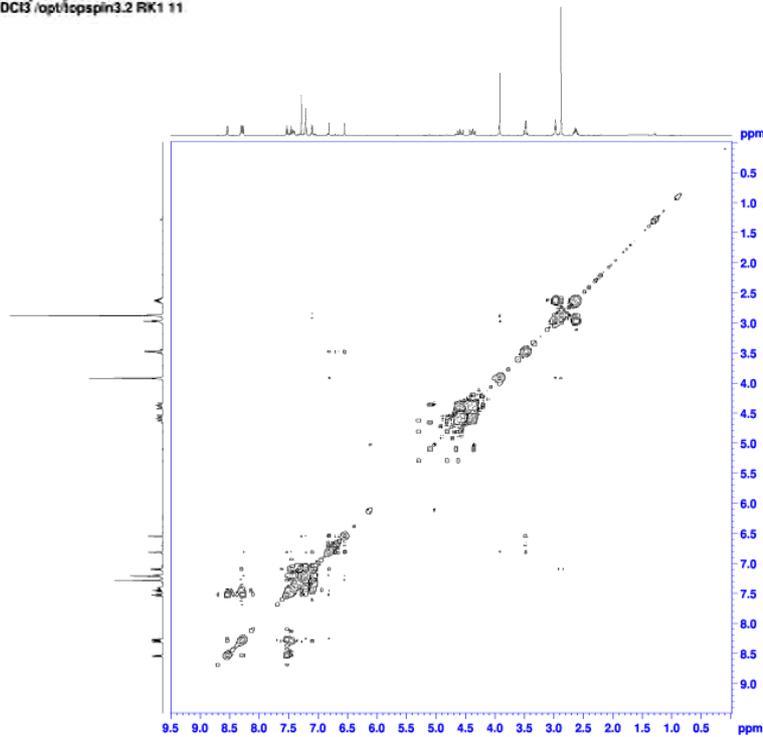


$\delta_C$  (126 MHz, CDCl<sub>3</sub>).



# COSY.

Chemist Richard Knighton  
 RK-2-185 dansyl alkylne  
 COSY.w CDC13 /opt/topspin3.2 RK1 11



```

Current Data Parameters
NAME      Jan03-2018
EXPNO    12
PROCNO    1

F2 - Acquisition Parameters
Date_     20180303
Time      16.07
INSTRUM   spect
PROBHD    5 mm CPQCH 13C
PULPROG   zgpg30
SOLVENT   CDCl3
NS         648
DS         4
SWH        4761.905 Hz
FIDRES    2.33149 Hz
AQ         0.2150400 sec
RG         60.07
DM         105.000 usec
DE         60.00 usec
TE         298.0 K
D1         1.9442400 sec
d11        0.0000000 sec
d12        0.0000000 sec
d16        0.0000000 sec
TNO       0.0002100 sec

----- CHANNEL f1 -----
SFO1      500.132709 MHz
NUC1      13C
P1         9.10 usec
P2         9.10 usec
P12       2900.00 usec
PL12      13.0000000 W
PL121     1.59249997 W

----- GRADIENT CHANNEL -----
GPM1[1]   PM100.100
SFO1      10.00 MHz
P14        1500.00 usec

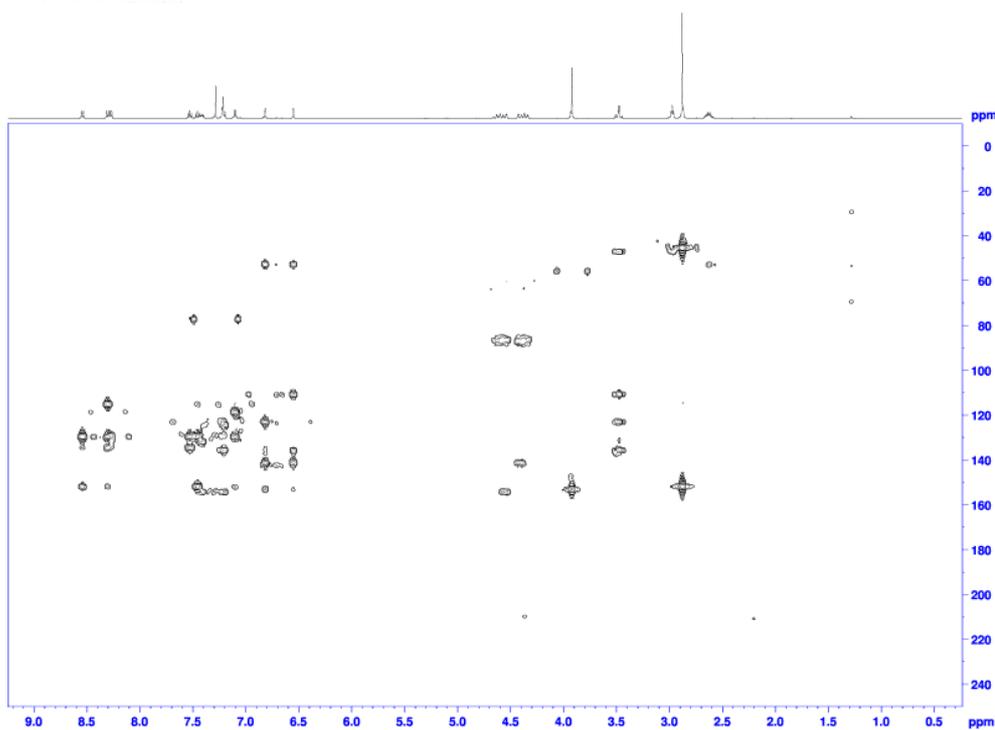
F1 - Acquisition parameters
SI         128
SF         500.1324 MHz
FIDRES    37.131453 Hz
AQ         9.903 ppm
F0MODE    QF

F2 - Processing parameters
SI         1024
SF         500.130000 MHz
WDW        GEM
SSB        0 Hz
LB         0 Hz
GB         1.40

F1 - Processing parameters
SI         1024
SF         500.130000 MHz
WDW        GEM
SSB        0 Hz
LB         0 Hz
GB         0
  
```

# HMBC.

Chemist Richard Knighton  
 RK-2-185 dansyl alkylne  
 HMBC.w CDC13 /opt/topspin3.2 RK1 11



```

Current Data Parameters
NAME      Jan03-2018
EXPNO    14
PROCNO    1

F2 - Acquisition Parameters
Date_     20180303
Time      16.28
INSTRUM   spect
PROBHD    5 mm CPQCH 13C
PULPROG   hmbpgp30
SOLVENT   CDCl3
NS         1224
DS         8
SWH        4504.204 Hz
FIDRES    4.38830 Hz
AQ         0.1136440 sec
RG         180.00
DM         111.000 usec
DE         60.00 usec
TE         298.0 K
D1         149.0000000 sec
D11        10.0000000 sec
D12        0.0000000 sec
D16        0.00344828 sec
D17        0.0000000 sec
D18        0.0000000 sec
TNO       0.0001100 sec

----- CHANNEL f1 -----
SFO1      500.132709 MHz
NUC1      13C
P1         9.10 usec
P2         18.20 usec
PL12      13.0000000 W

----- CHANNEL f2 -----
SFO2      125.7729195 MHz
NUC2      13C
P3         9.20 usec
PL32      14.0000000 W

----- GRADIENT CHANNEL -----
GPM1[1]   PM100.100
GPM1[2]   PM100.100
GPM1[3]   PM100.100
SFO1      10.00 MHz
SFO2      30.00 MHz
CP12      60.00 W
CP13      60.00 W
P16       1000.00 usec

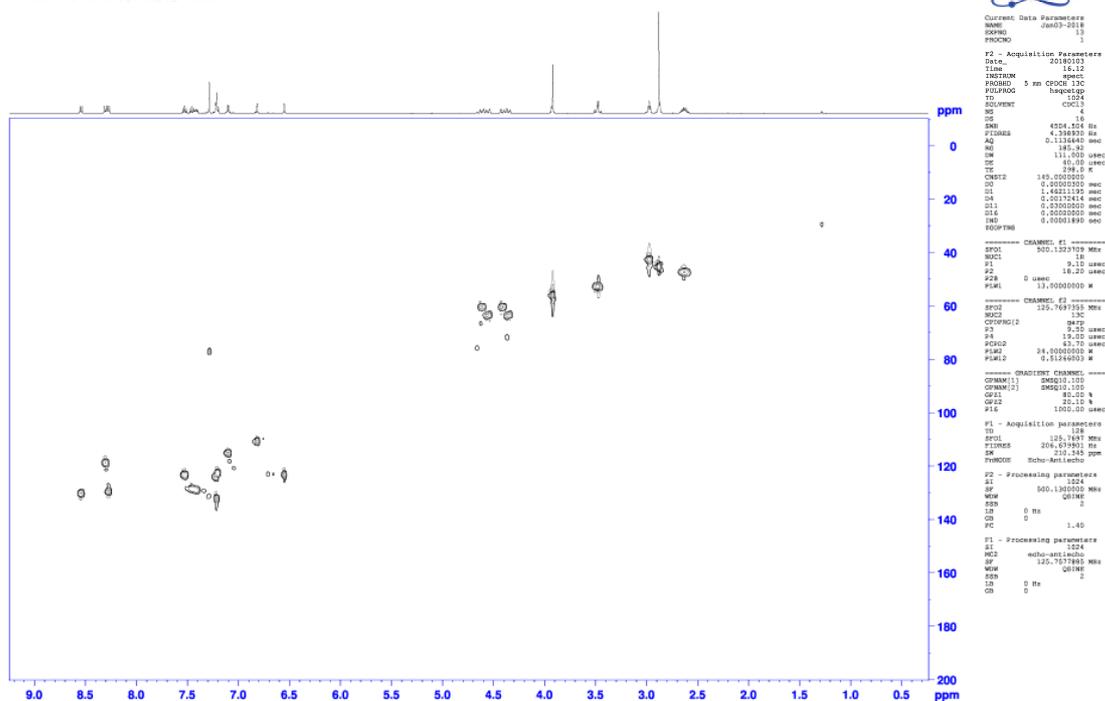
F1 - Acquisition parameters
SI         128
SF         500.1324 MHz
FIDRES    205.310455 Hz
AQ         299.491 ppm
F0MODE    QF

F2 - Processing parameters
SI         1024
SF         500.130000 MHz
WDW        GEM
SSB        0 Hz
LB         0 Hz
GB         1.40

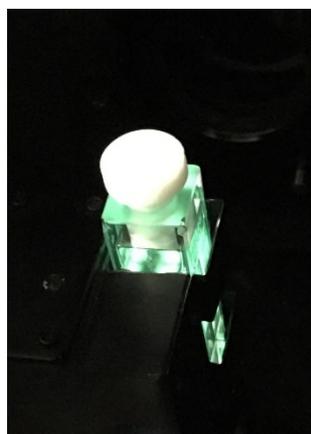
F1 - Processing parameters
SI         1024
SF         125.7577195 MHz
WDW        GEM
SSB        0 Hz
LB         0 Hz
GB         0
  
```

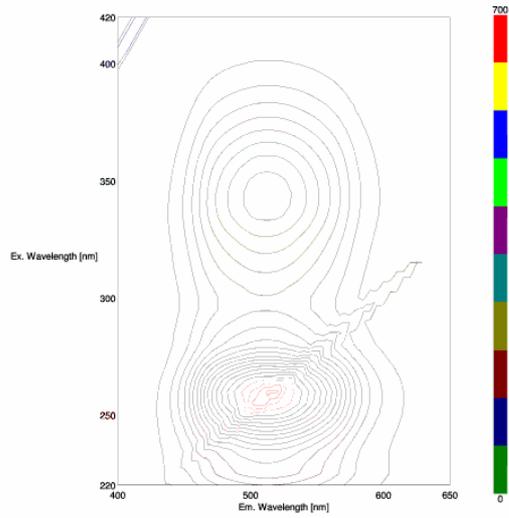
HMQC.

Chemist Richard Knighton  
RK-2-185 dansyl alkynes  
HSQC.w CDCl<sub>3</sub> /opt/topspin3.2 RK1 11

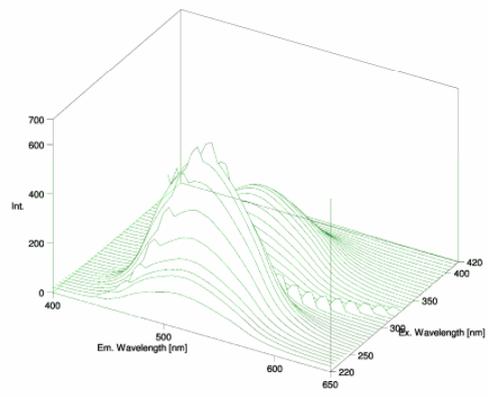


Fluorescence spectra and images.



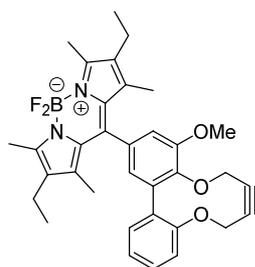


Date: 1/11/2007 14:37  
 File name: JSCA TEST 048263, ALKYLNE TEST#017.job  
 Model: PF-2020  
 Serial No.: C0708050  
 Measurement Mode: Excitation Spectrum  
 Slit width (nm): 3 nm  
 Slit width (nm): 3 nm  
 Resolution: 1 nm  
 Slit width: 3 nm  
 Measurement range: 400 - 650 nm  
 Data path: 000  
 Excitation Wavelength: 350 nm  
 Scanning Speed: 17 nm/min  
 Slit No.: 17  
 No. of Cycles: 1  
 Sample name: Chemsky - JCSFS  
 Operator:  
 Comment:

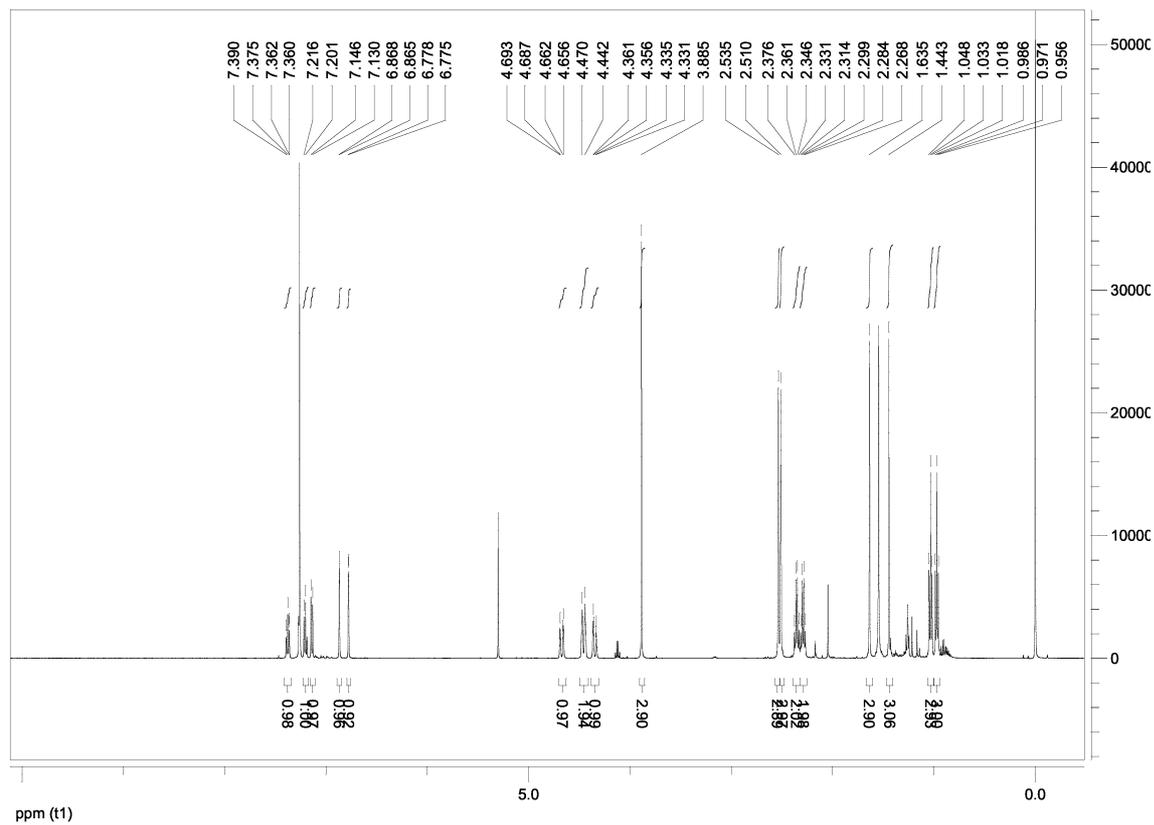


Date: 1/11/2007 14:37  
 File name: JSCA TEST 048263, ALKYLNE TEST#017.job  
 Model: PF-2020  
 Serial No.: C0708050  
 Measurement Mode: Excitation Spectrum  
 Slit width (nm): 3 nm  
 Slit width (nm): 3 nm  
 Resolution: 1 nm  
 Slit width: 3 nm  
 Measurement range: 400 - 650 nm  
 Data path: 000  
 Excitation Wavelength: 350 nm  
 Scanning Speed: 17 nm/min  
 Slit No.: 17  
 No. of Cycles: 1  
 Sample name: Chemsky - JCSFS  
 Operator:  
 Comment:

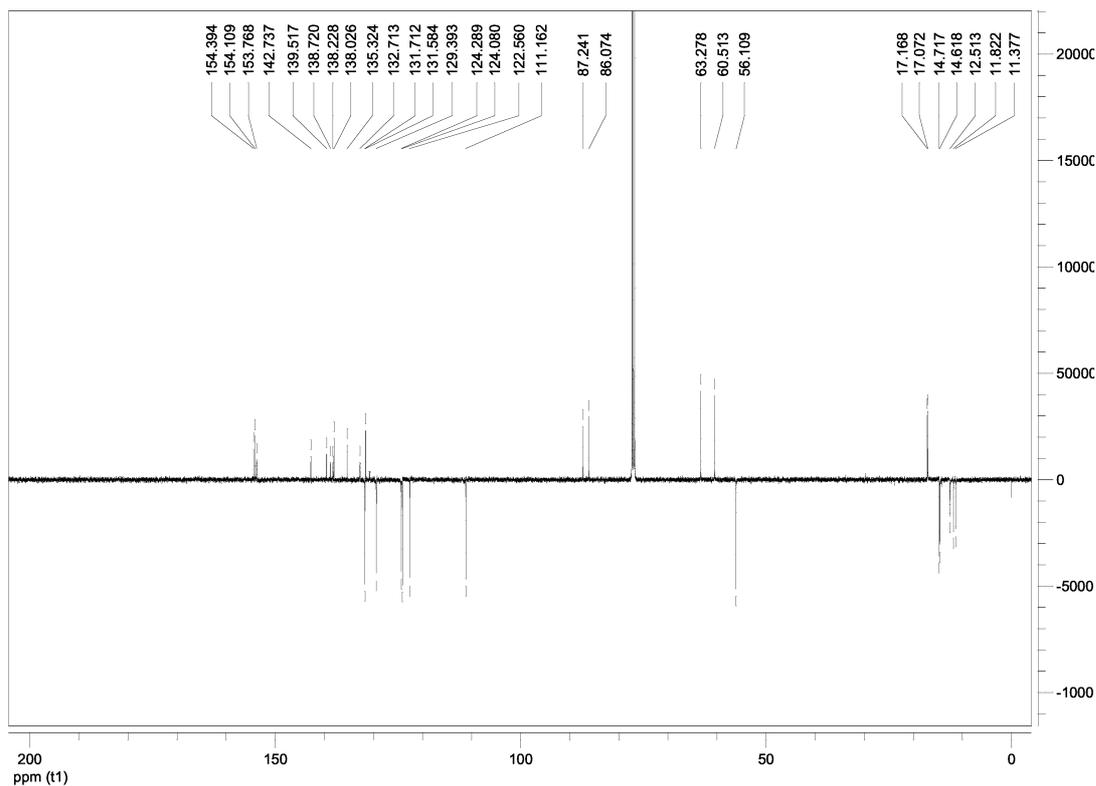
## 2,4-Dimethyl-3-ethyl BoDIPY Strained alkyne 36. SF232.



$\delta_H$  (500 MHz,  $CDCl_3$ ).

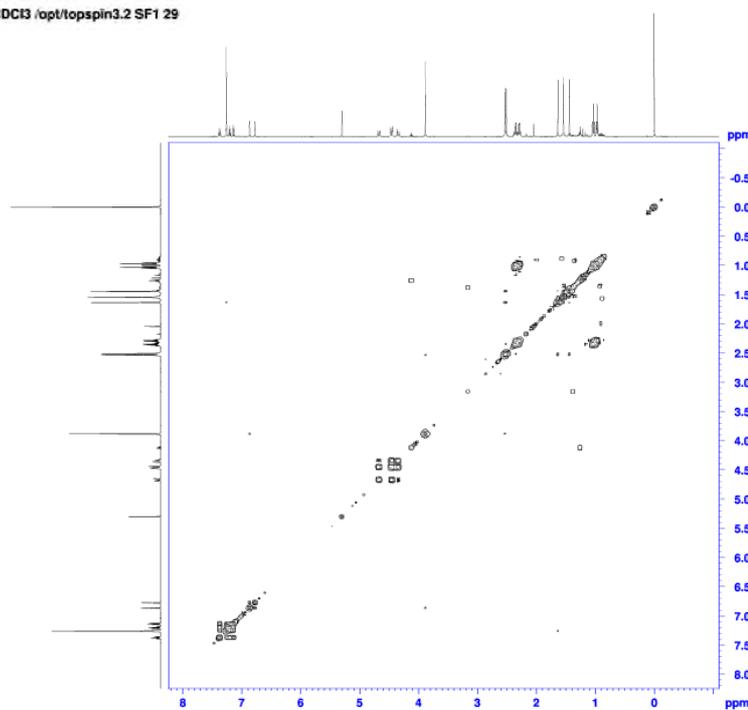


$\delta_C$  (126 MHz,  $CDCl_3$ ).



COSY:

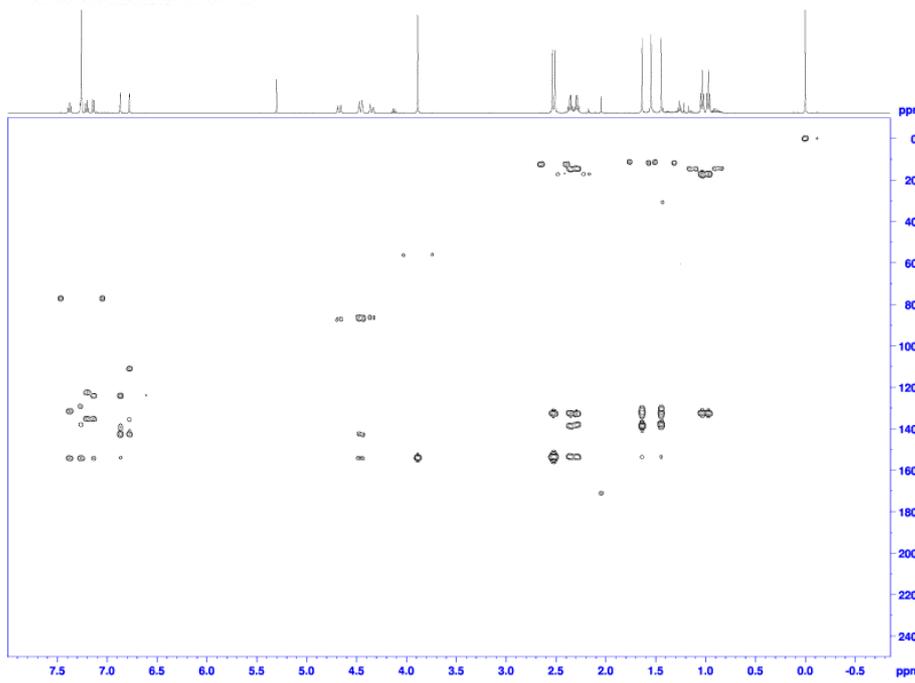
Chemist Sam Forshaw  
SF232  
COSY.w  $CDCl_3$  /opt/topspin3.2 SF1 29



```
Current Data Parameters
NAME      Dec08-2013
EXPNO    11
PROCNO   1
F2 - Acquisition Parameters
Date_    20121208
Time     20.43
INSTRUM  spect
PROBHD   5 mm QNP1H 13C
P1       12.00000000 sec
SFO      125.7611988 MHz
SOLVENT  CDCl3
NS       1
DS       8
SWH      4672.897 Hz
FIDRES   2.281488 Hz
AQ       0.218180 sec
RG        65.17
CW       157.000 usec
DE       40.00 usec
TE       298.2 K
D1       0.00000000 sec
D11      0.00000000 sec
D12      0.00000000 sec
D13      0.00000000 sec
D14      0.00000000 sec
D15      0.00000000 sec
D16      0.00000000 sec
D17      0.00000000 sec
D18      0.00000000 sec
D19      0.00000000 sec
D20      0.00000000 sec
----- CHANNEL f1 -----
NUC1     13C
P1       9.10 usec
P2       9.10 usec
P3       9.10 usec
P4       9.10 usec
P5       9.10 usec
P6       9.10 usec
P7       9.10 usec
P8       9.10 usec
P9       9.10 usec
P10      9.10 usec
P11      9.10 usec
P12      9.10 usec
P13      9.10 usec
P14      9.10 usec
P15      9.10 usec
P16      9.10 usec
P17      9.10 usec
P18      9.10 usec
P19      9.10 usec
P20      9.10 usec
P21      9.10 usec
P22      9.10 usec
P23      9.10 usec
P24      9.10 usec
P25      9.10 usec
P26      9.10 usec
P27      9.10 usec
P28      9.10 usec
P29      9.10 usec
P30      9.10 usec
P31      9.10 usec
P32      9.10 usec
P33      9.10 usec
P34      9.10 usec
P35      9.10 usec
P36      9.10 usec
P37      9.10 usec
P38      9.10 usec
P39      9.10 usec
P40      9.10 usec
P41      9.10 usec
P42      9.10 usec
P43      9.10 usec
P44      9.10 usec
P45      9.10 usec
P46      9.10 usec
P47      9.10 usec
P48      9.10 usec
P49      9.10 usec
P50      9.10 usec
P51      9.10 usec
P52      9.10 usec
P53      9.10 usec
P54      9.10 usec
P55      9.10 usec
P56      9.10 usec
P57      9.10 usec
P58      9.10 usec
P59      9.10 usec
P60      9.10 usec
P61      9.10 usec
P62      9.10 usec
P63      9.10 usec
P64      9.10 usec
P65      9.10 usec
P66      9.10 usec
P67      9.10 usec
P68      9.10 usec
P69      9.10 usec
P70      9.10 usec
P71      9.10 usec
P72      9.10 usec
P73      9.10 usec
P74      9.10 usec
P75      9.10 usec
P76      9.10 usec
P77      9.10 usec
P78      9.10 usec
P79      9.10 usec
P80      9.10 usec
P81      9.10 usec
P82      9.10 usec
P83      9.10 usec
P84      9.10 usec
P85      9.10 usec
P86      9.10 usec
P87      9.10 usec
P88      9.10 usec
P89      9.10 usec
P90      9.10 usec
P91      9.10 usec
P92      9.10 usec
P93      9.10 usec
P94      9.10 usec
P95      9.10 usec
P96      9.10 usec
P97      9.10 usec
P98      9.10 usec
P99      9.10 usec
P100     9.10 usec
----- GRABINTE CHANNEL -----
GRABINTE 2000000.00
SFO      125.7611988 MHz
SOLVENT  CDCl3
NS       1
DS       8
SWH      4672.897 Hz
FIDRES   2.281488 Hz
AQ       0.218180 sec
RG        65.17
CW       157.000 usec
DE       40.00 usec
TE       298.2 K
D1       0.00000000 sec
D11      0.00000000 sec
D12      0.00000000 sec
D13      0.00000000 sec
D14      0.00000000 sec
D15      0.00000000 sec
D16      0.00000000 sec
D17      0.00000000 sec
D18      0.00000000 sec
D19      0.00000000 sec
D20      0.00000000 sec
----- CHANNEL f2 -----
NUC2     1H
P1       9.10 usec
P2       9.10 usec
P3       9.10 usec
P4       9.10 usec
P5       9.10 usec
P6       9.10 usec
P7       9.10 usec
P8       9.10 usec
P9       9.10 usec
P10      9.10 usec
P11      9.10 usec
P12      9.10 usec
P13      9.10 usec
P14      9.10 usec
P15      9.10 usec
P16      9.10 usec
P17      9.10 usec
P18      9.10 usec
P19      9.10 usec
P20      9.10 usec
P21      9.10 usec
P22      9.10 usec
P23      9.10 usec
P24      9.10 usec
P25      9.10 usec
P26      9.10 usec
P27      9.10 usec
P28      9.10 usec
P29      9.10 usec
P30      9.10 usec
P31      9.10 usec
P32      9.10 usec
P33      9.10 usec
P34      9.10 usec
P35      9.10 usec
P36      9.10 usec
P37      9.10 usec
P38      9.10 usec
P39      9.10 usec
P40      9.10 usec
P41      9.10 usec
P42      9.10 usec
P43      9.10 usec
P44      9.10 usec
P45      9.10 usec
P46      9.10 usec
P47      9.10 usec
P48      9.10 usec
P49      9.10 usec
P50      9.10 usec
P51      9.10 usec
P52      9.10 usec
P53      9.10 usec
P54      9.10 usec
P55      9.10 usec
P56      9.10 usec
P57      9.10 usec
P58      9.10 usec
P59      9.10 usec
P60      9.10 usec
P61      9.10 usec
P62      9.10 usec
P63      9.10 usec
P64      9.10 usec
P65      9.10 usec
P66      9.10 usec
P67      9.10 usec
P68      9.10 usec
P69      9.10 usec
P70      9.10 usec
P71      9.10 usec
P72      9.10 usec
P73      9.10 usec
P74      9.10 usec
P75      9.10 usec
P76      9.10 usec
P77      9.10 usec
P78      9.10 usec
P79      9.10 usec
P80      9.10 usec
P81      9.10 usec
P82      9.10 usec
P83      9.10 usec
P84      9.10 usec
P85      9.10 usec
P86      9.10 usec
P87      9.10 usec
P88      9.10 usec
P89      9.10 usec
P90      9.10 usec
P91      9.10 usec
P92      9.10 usec
P93      9.10 usec
P94      9.10 usec
P95      9.10 usec
P96      9.10 usec
P97      9.10 usec
P98      9.10 usec
P99      9.10 usec
P100     9.10 usec
----- GRABINTE CHANNEL -----
GRABINTE 2000000.00
SFO      500.1317889 MHz
SOLVENT  CDCl3
NS       1
DS       8
SWH      4672.897 Hz
FIDRES   2.281488 Hz
AQ       0.218180 sec
RG        65.17
CW       157.000 usec
DE       40.00 usec
TE       298.2 K
D1       0.00000000 sec
D11      0.00000000 sec
D12      0.00000000 sec
D13      0.00000000 sec
D14      0.00000000 sec
D15      0.00000000 sec
D16      0.00000000 sec
D17      0.00000000 sec
D18      0.00000000 sec
D19      0.00000000 sec
D20      0.00000000 sec
----- CHANNEL f3 -----
NUC3     13C
P1       9.10 usec
P2       9.10 usec
P3       9.10 usec
P4       9.10 usec
P5       9.10 usec
P6       9.10 usec
P7       9.10 usec
P8       9.10 usec
P9       9.10 usec
P10      9.10 usec
P11      9.10 usec
P12      9.10 usec
P13      9.10 usec
P14      9.10 usec
P15      9.10 usec
P16      9.10 usec
P17      9.10 usec
P18      9.10 usec
P19      9.10 usec
P20      9.10 usec
P21      9.10 usec
P22      9.10 usec
P23      9.10 usec
P24      9.10 usec
P25      9.10 usec
P26      9.10 usec
P27      9.10 usec
P28      9.10 usec
P29      9.10 usec
P30      9.10 usec
P31      9.10 usec
P32      9.10 usec
P33      9.10 usec
P34      9.10 usec
P35      9.10 usec
P36      9.10 usec
P37      9.10 usec
P38      9.10 usec
P39      9.10 usec
P40      9.10 usec
P41      9.10 usec
P42      9.10 usec
P43      9.10 usec
P44      9.10 usec
P45      9.10 usec
P46      9.10 usec
P47      9.10 usec
P48      9.10 usec
P49      9.10 usec
P50      9.10 usec
P51      9.10 usec
P52      9.10 usec
P53      9.10 usec
P54      9.10 usec
P55      9.10 usec
P56      9.10 usec
P57      9.10 usec
P58      9.10 usec
P59      9.10 usec
P60      9.10 usec
P61      9.10 usec
P62      9.10 usec
P63      9.10 usec
P64      9.10 usec
P65      9.10 usec
P66      9.10 usec
P67      9.10 usec
P68      9.10 usec
P69      9.10 usec
P70      9.10 usec
P71      9.10 usec
P72      9.10 usec
P73      9.10 usec
P74      9.10 usec
P75      9.10 usec
P76      9.10 usec
P77      9.10 usec
P78      9.10 usec
P79      9.10 usec
P80      9.10 usec
P81      9.10 usec
P82      9.10 usec
P83      9.10 usec
P84      9.10 usec
P85      9.10 usec
P86      9.10 usec
P87      9.10 usec
P88      9.10 usec
P89      9.10 usec
P90      9.10 usec
P91      9.10 usec
P92      9.10 usec
P93      9.10 usec
P94      9.10 usec
P95      9.10 usec
P96      9.10 usec
P97      9.10 usec
P98      9.10 usec
P99      9.10 usec
P100     9.10 usec
----- GRABINTE CHANNEL -----
GRABINTE 2000000.00
SFO      500.1317889 MHz
SOLVENT  CDCl3
NS       1
DS       8
SWH      4672.897 Hz
FIDRES   2.281488 Hz
AQ       0.218180 sec
RG        65.17
CW       157.000 usec
DE       40.00 usec
TE       298.2 K
D1       0.00000000 sec
D11      0.00000000 sec
D12      0.00000000 sec
D13      0.00000000 sec
D14      0.00000000 sec
D15      0.00000000 sec
D16      0.00000000 sec
D17      0.00000000 sec
D18      0.00000000 sec
D19      0.00000000 sec
D20      0.00000000 sec
```

HMBC:

Chemist Sam Forshaw  
SF232  
HMBC.w CDCI3 /opt/topspin3.2 SF1 29

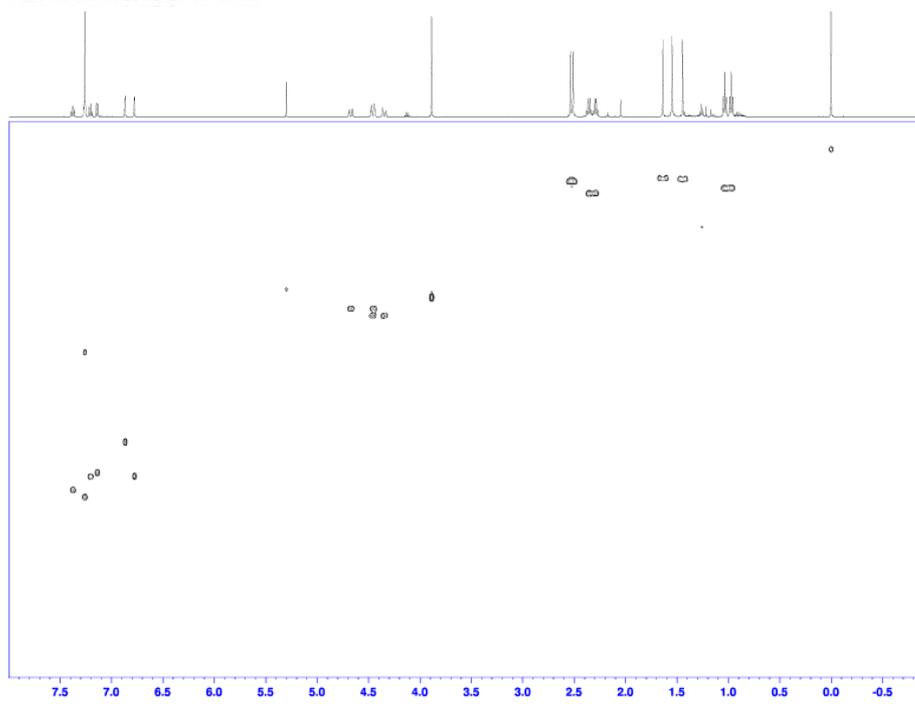


```

Current Data Parameters
NAME          Dec08-2011
EXPNO        13
PROCNO       1
----- Acquisition Parameters
Date_         2011029
Time         22:04
INSTRUM      spect
PROBHD       5 mm CPDQX 13C
PULPROG      hmcoplgpuf
TD           65536
SOLVENT      CDCl3
NS           6
DS           4
SWH           4624.770 Hz
F2RES        4.22120 Hz
AQ           0.1127720 sec
RG           137.00
WDW           EM
SS           133.000 usec
LB           60.00 usec
TE           298.2 K
CH212        145.000000
CH213        125.000000
DO           0.0000000 sec
DE           1.4000000 sec
DM           0.0017474 sec
DQ           0.0000000 sec
DI1          0.0000000 sec
DI2          0.0000000 sec
IND          0.0001930 sec
----- CHANNEL f1 -----
NUC1          13C
P1           9.10 usec
PC           13.0000000 W
----- CHANNEL f2 -----
NUC2          1H
P2           9.30 usec
PC           24.0000000 W
----- GRABBER CHANNEL -----
OPAMN11      SMIG1.100
OPAMN12      SMIG1.100
OPAMN13      SMIG1.100
OPF1         80.00 N
OPF2         80.00 N
OPF3         80.00 N
P15          1000.00 usec
P1 - Acquisition parameters
TD           65536
SF           125.762 MHz
FIDRES       206.47961 Hz
SFO          200.13143 MHz
PWRMODE      Echo-AntiEcho
P2 - Processing parameters
SI           32768
SF           500.1300125 MHz
WDW          EM
SS           0 Hz
LB           0 Hz
GB           0
PC           1.40
P1 - Processing parameters
SI           32768
SF           125.7577885 MHz
WDW          EM
SS           0 Hz
LB           0 Hz
GB           0
PC           0
  
```

HSQC:

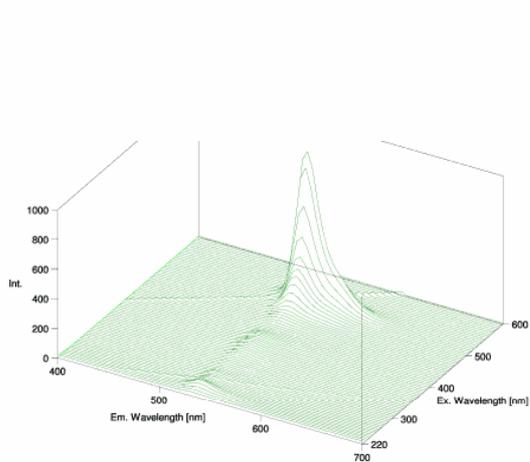
Chemist Sam Forshaw  
SF232  
HSQC.w CDCI3 /opt/topspin3.2 SF1 29



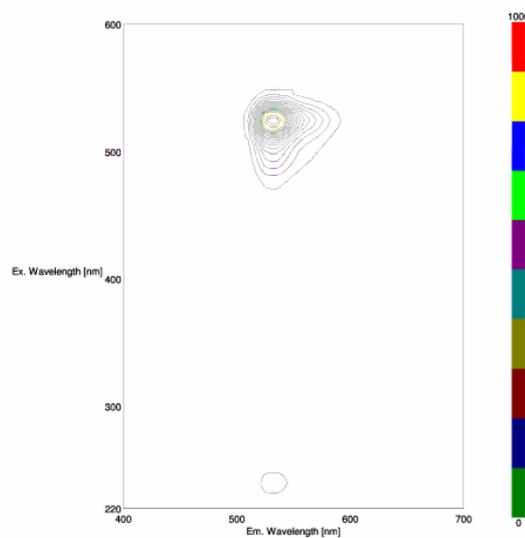
```

Current Data Parameters
NAME          Dec08-2011
EXPNO        13
PROCNO       1
----- Acquisition Parameters
Date_         2011029
Time         22:04
INSTRUM      spect
PROBHD       5 mm CPDQX 13C
PULPROG      hmcoplgpuf
TD           65536
SOLVENT      CDCl3
NS           6
DS           4
SWH           4624.770 Hz
F2RES        4.22120 Hz
AQ           0.1127720 sec
RG           137.00
WDW           EM
SS           133.000 usec
LB           60.00 usec
TE           298.2 K
CH212        145.000000
CH213        125.000000
DO           0.0000000 sec
DE           1.4000000 sec
DM           0.0017474 sec
DQ           0.0000000 sec
DI1          0.0000000 sec
DI2          0.0000000 sec
IND          0.0001930 sec
ROBOTMS
----- CHANNEL f1 -----
NUC1          13C
P1           9.10 usec
PC           13.0000000 W
----- CHANNEL f2 -----
NUC2          1H
P2           9.30 usec
PC           24.0000000 W
----- GRABBER CHANNEL -----
OPAMN11      SMIG1.100
OPAMN12      SMIG1.100
OPAMN13      SMIG1.100
OPF1         80.00 N
OPF2         80.00 N
OPF3         80.00 N
P15          1000.00 usec
P1 - Acquisition parameters
TD           65536
SF           125.762 MHz
FIDRES       206.47961 Hz
SFO          200.13143 MHz
PWRMODE      Echo-AntiEcho
P2 - Processing parameters
SI           32768
SF           500.1300125 MHz
WDW          EM
SS           0 Hz
LB           0 Hz
GB           0
PC           1.40
P1 - Processing parameters
SI           32768
SF           125.7577885 MHz
WDW          EM
SS           0 Hz
LB           0 Hz
GB           0
PC           0
  
```

## Fluorescence spectra and images.



Date: 05/03/18 15:47  
 File name: 01201\_chemistry\_ethyl\_6u2OPF\_aligned\_ethylm1818.job  
 Method: F2-6000  
 Sample No.: CF768892  
 Measurement Mode: Channel Spectrum  
 Slit width (nm): 3 nm  
 Slit width (nm): 2 nm  
 Response: 1 sec  
 Scan rate: 400  
 Measurement range: 400 - 700 nm  
 Data pitch: 500  
 Excitation Wavelength: 520.0 nm  
 Scanning speed: 50 rev/min  
 Sample ID: 18  
 No. of cycles: 1  
 Sample name: Chemistry\_JCEPS  
 Operator: Chemistry\_JCEPS  
 Comment:



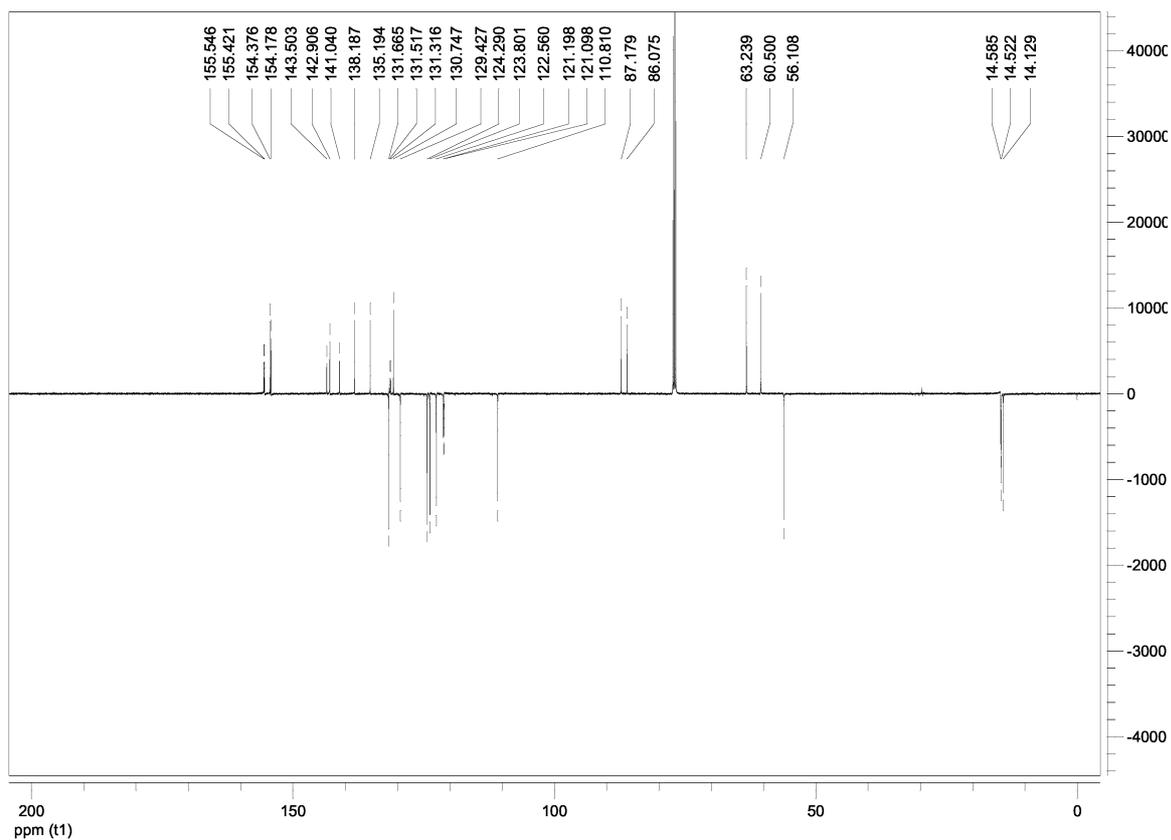
Date: 05/03/18 15:47  
 File name: 01201\_chemistry\_ethyl\_6u2OPF\_aligned\_ethylm1818.job  
 Method: F2-6000  
 Sample No.: CF768892  
 Measurement Mode: Channel Spectrum  
 Slit width (nm): 3 nm  
 Slit width (nm): 2 nm  
 Response: 1 sec  
 Scan rate: 400  
 Measurement range: 400 - 700 nm  
 Data pitch: 500  
 Excitation Wavelength: 520.0 nm  
 Scanning speed: 50 rev/min  
 Sample ID: 18  
 No. of cycles: 1  
 Sample name: Chemistry\_JCEPS  
 Operator: Chemistry\_JCEPS  
 Comment:



(above) Compound 36 in absence (left) and presence (right) of uv light.

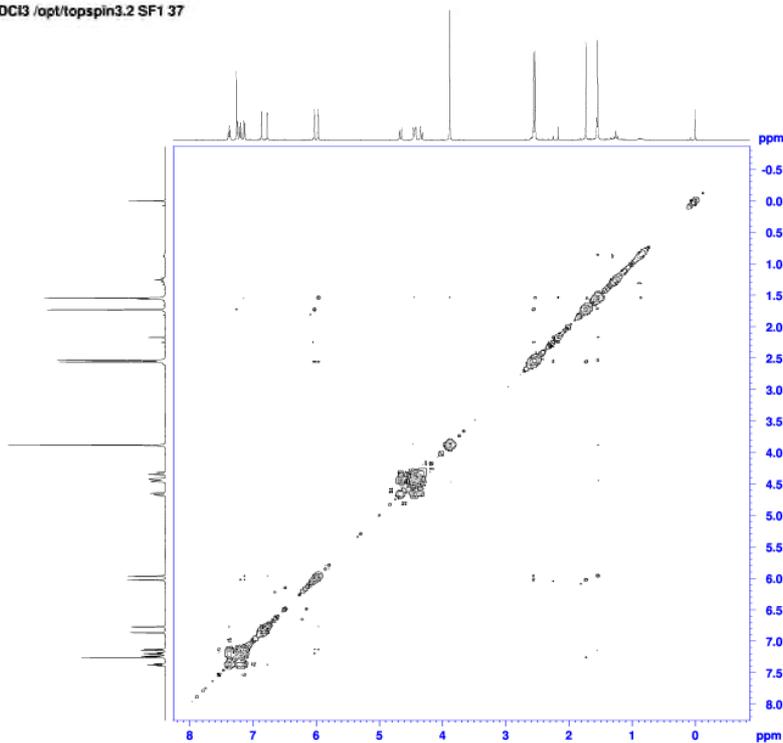


$\delta_C$  (126 MHz,  $CDCl_3$ ).



COSY:

Chemist Sam Forshaw  
SF261  
COSY.w  $CDCl_3$  /opt/topspin3.2 SF1 37



```

Current Data Parameters
NAME      Pauli-3118
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20180228
Time     19.16
INSTRUM  spect
PROBHD   5 mm CPDQX 13C
PULPROG  zgpg30
SOLVENT  CDCl3
NS       5
DS       2
SWH      4562.048 Hz
FIDRES   2.227661 Hz
AQ       0.2261608 sec
RG       40.07
SW       169.4000 usec
DE       40.00 usec
TE       298.0 K
D0       0.00000000 sec
D1       1.31388394 sec
D11      0.03000000 sec
D12      0.00000000 sec
D13      0.00000000 sec
D16      0.00000000 sec
INRG     0.00021820 sec

----- CHANNEL f1 -----
SFO1     500.136260 MHz
NUC1     13C
P0       9.10 usec
P1       9.10 usec
P17      2500.00 usec
PL1      13.00000000 W
PL12     1.18448981 W

----- GRABINQ CHANNEL -----
GRABINQ[1] SMSQ10.100
GRABINQ[1] 10.00 %
P16      1000.00 usec

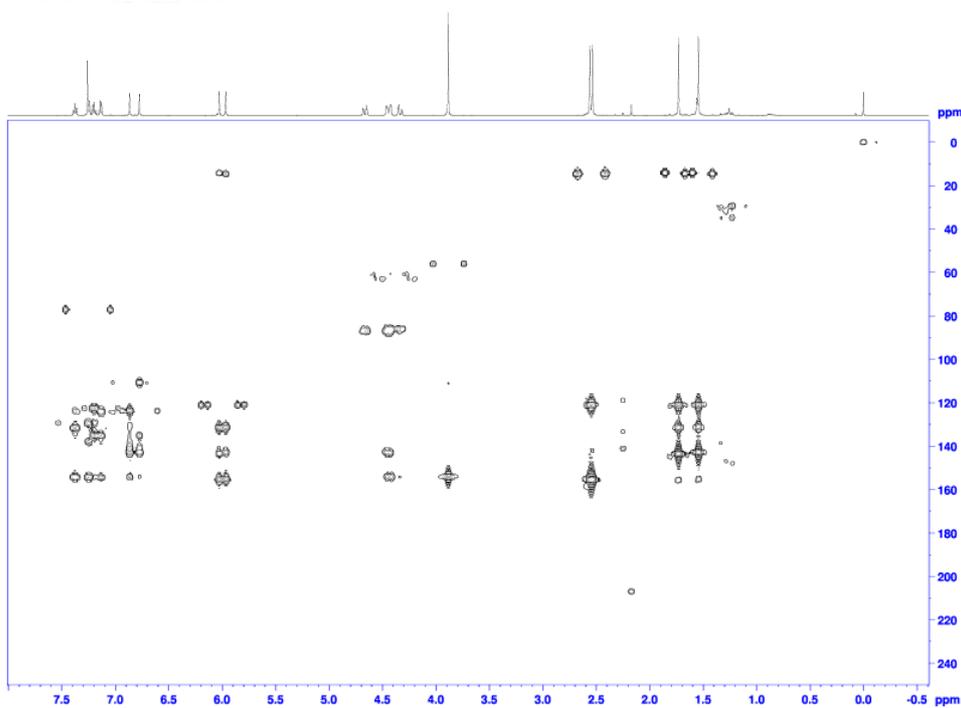
F1 - Acquisition parameters
FO      500.136260 MHz
SFO1    500.1319 MHz
FIDRES   35.660968 Hz
SW       9.122 ppm
FIDRES   QF

F2 - Processing parameters
SI       1624
SF       500.130173 MHz
WDW      QF
SSB      0 Hz
GB       0
PC       1.40

F1 - Processing parameters
SI       1624
SF       500.130173 MHz
WDW      QF
SSB      0 Hz
GB       0
PC       0
    
```

# HMBC:

Chemist Sam Forshaw  
SF261  
HMBC.w CDCl3 /opt/topspin3.2 SF1 37



```

Current Data Parameters
NAME Feb08-2018
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180228
Time 19:27
INSTRUM spect
PROBHD 5 mm CPDOR 13C
PULPROG zgpg30
TD 65536
SOLVENT cdcl3
NS 16
DS 4
SWH 4310.340 Hz
FIDRES 4.209221 Hz
AQ 0.1187840 sec
RG 655.50
DM 114.000 usec
DE 60.00 usec
TE 298.2 K
C1P2 140.000000 Hz
C1P1 10.000000 Hz
C2 0.00000000 sec
C3 1.40000000 sec
C4 0.00348828 sec
C5 0.00000000 sec
C6 0.00000000 sec
C7 0.00000000 sec

===== CHANNEL f1 =====
NUC1 500.1318620 MHz
P1 9.10 usec
P2 18.20 usec
P3 13.00000000 Hz

===== CHANNEL f2 =====
NUC2 125.7728190 MHz
P4 9.10 usec
P5 24.00000000 Hz

===== GRADIENT CHANNEL =====
GPM1[1] SMS210.100
GPM1[2] SMS210.100
GPM1[3] SMS210.100
CPL1 80.00 %
CPL2 80.00 %
CPL3 1000.00 usec

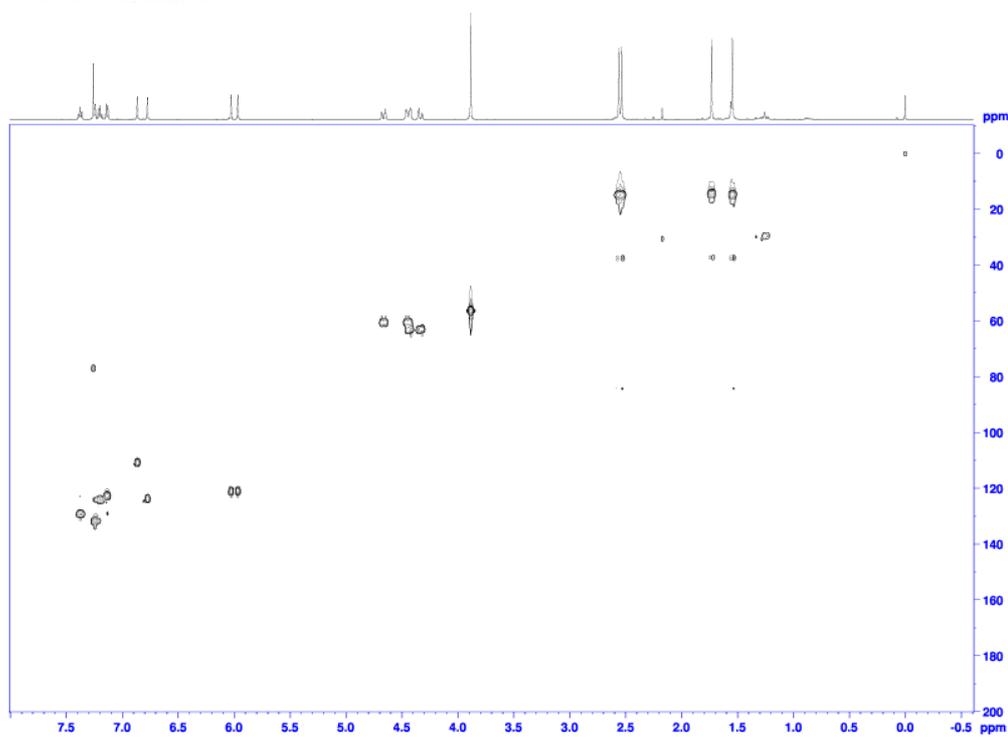
F1 - Acquisition parameters
SI 128
SF 125.7728 MHz
FIDRES 208.470501 Hz
AQ 258.831 ppm
PROCNO 13

F2 - Processing parameters
SI 1024
SF 500.1300100 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.60

F1 - Processing parameters
SI 1024
SF 125.7577885 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.60
    
```

# HSQC:

Chemist Sam Forshaw  
SF261  
HSQC.w CDCl3 /opt/topspin3.2 SF1 37



```

Current Data Parameters
NAME Feb08-2018
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180228
Time 19:42
INSTRUM spect
PROBHD 5 mm CPDOR 13C
PULPROG zgpg30
TD 65536
SOLVENT cdcl3
NS 16
DS 4
SWH 4310.340 Hz
FIDRES 4.209221 Hz
AQ 0.1187840 sec
RG 655.50
DM 114.000 usec
DE 60.00 usec
TE 298.2 K
C1P2 140.000000 Hz
C1P1 10.000000 Hz
C2 0.00000000 sec
C3 1.40000000 sec
C4 0.00372414 sec
C5 0.00000000 sec
C6 0.00000000 sec
C7 0.00000000 sec

===== CHANNEL f1 =====
NUC1 500.1318620 MHz
P1 9.10 usec
P2 18.20 usec
P3 13.00000000 Hz

===== CHANNEL f2 =====
NUC2 125.7637355 MHz
P4 9.10 usec
P5 24.00000000 Hz

===== GRADIENT CHANNEL =====
GPM1[1] SMS210.100
GPM1[2] SMS210.100
GPM1[3] SMS210.100
CPL1 80.00 %
CPL2 80.00 %
CPL3 1000.00 usec

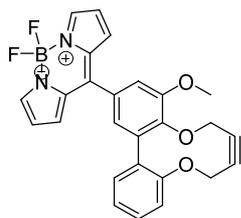
F1 - Acquisition parameters
SI 128
SF 125.7607 MHz
FIDRES 208.470501 Hz
AQ 258.835 ppm
PROCNO 13

F2 - Processing parameters
SI 1024
SF 500.1300100 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.60

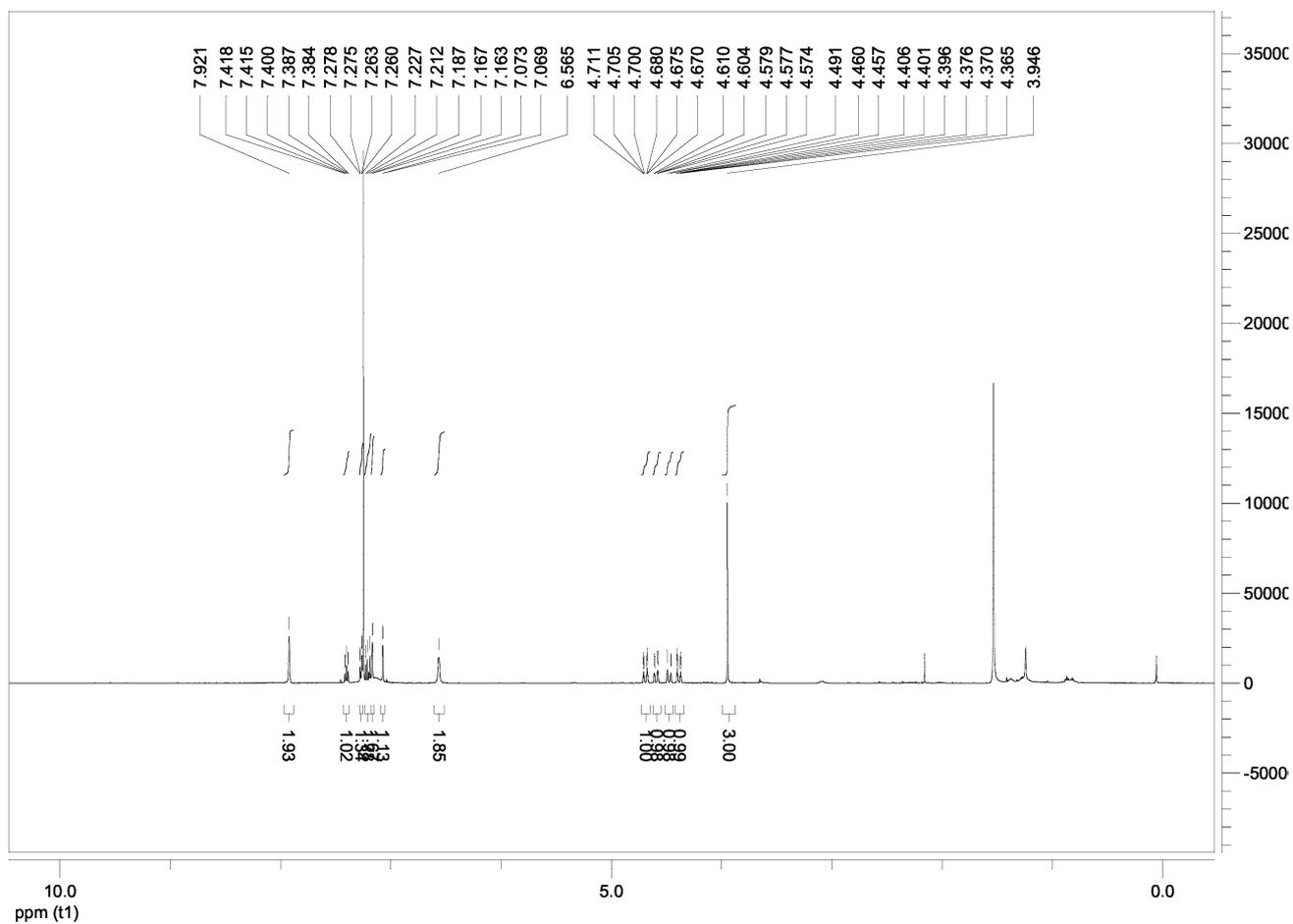
F1 - Processing parameters
SI 1024
SF 125.7577885 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.60
    
```



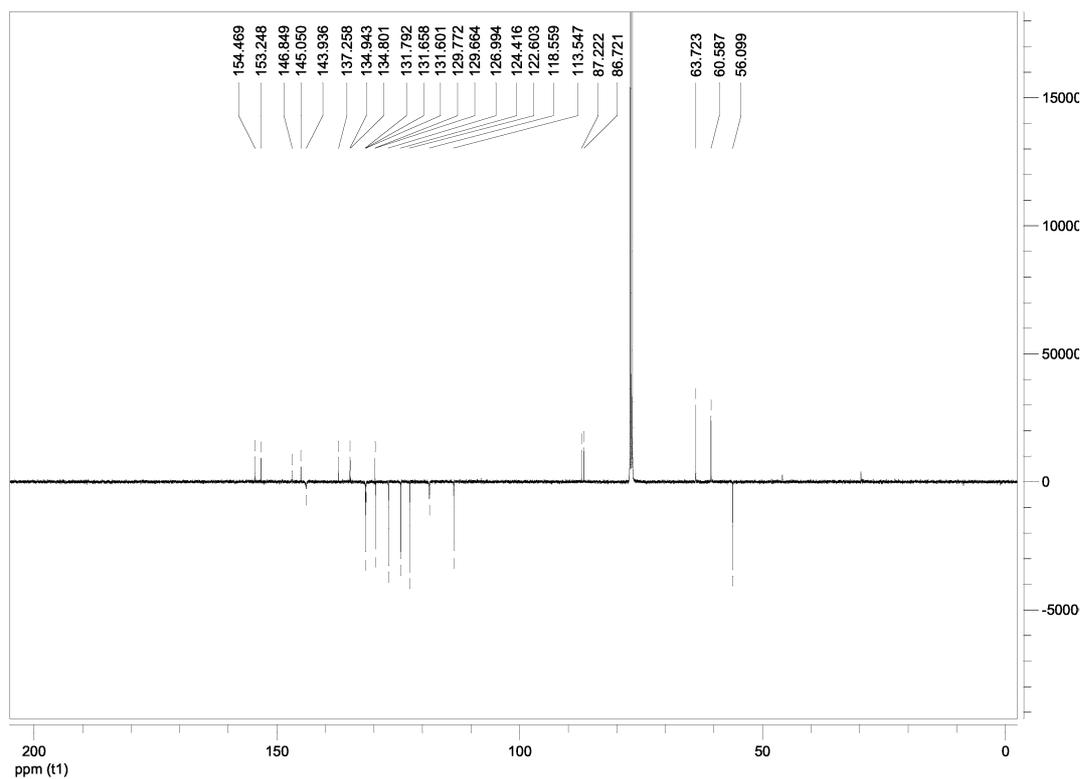
**BoDIPY derivative with H,H,H substitution 38 (SF262):**



$\delta_H$  (500 MHz,  $CDCl_3$ ).

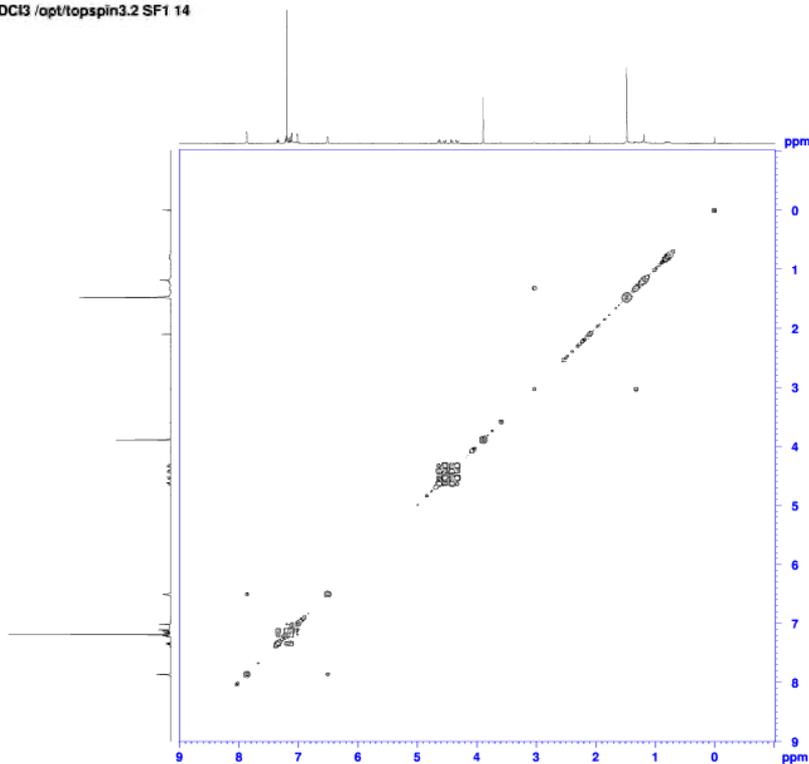


$\delta_C$  (126 MHz, CDCl<sub>3</sub>).



COSY:

Chemist Sam Forshaw  
SF262  
COSY.w CDCl<sub>3</sub>/opt/topspin3.2 SF1 14



```

Current Data Parameters
NAME          Mar23-2018
EXPNO        21
PROCNO       1

F2 - Acquisition Parameters
Date_        20180320
Time         16.30
INSTRUM      spect
PROBHD       5 mm CPDCH 13C
PULPROG      zgpg30
SOLVENT      CDCl3
NS           2048
DS           4
SWH          3013.169 Hz
FIDRES      2.447834 Hz
AQ          0.306333 sec
RG          180.93
CW          99.133 usec
DE          40.00 usec
TE          298.2 K
D0          0.0000300 sec
D1          1.3160119 sec
D11         0.03000000 sec
D12         0.00000000 sec
D13         0.00000400 sec
D16         0.00020000 sec
IND         0.00019860 sec

===== CHANNEL f1 =====
NUC1         13C
P1           4.10 usec
PL1         200.00 usec
PL12        13.00000000 Hz
PL10        1.58249997 Hz

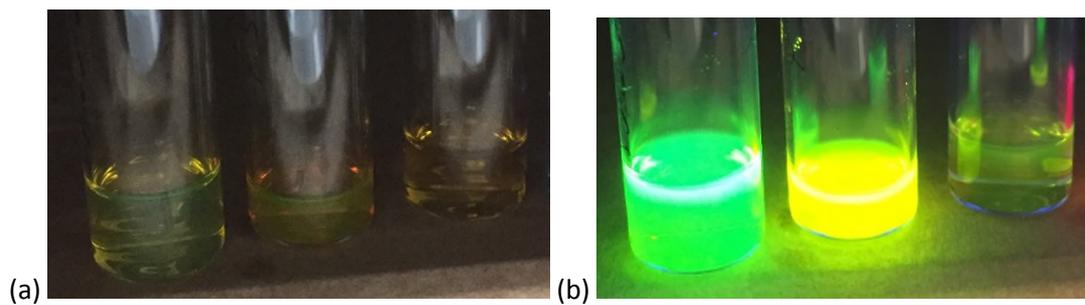
===== GRADIENT CHANNEL =====
GPMAX[1]    10.00 %
GPR1        1000.00 usec

F1 - Acquisition parameters
TD          131
SFO1        500.132 MHz
FIDRES      39.140781 Hz
CW          10.011 ppm
PRWIDE      0

F2 - Processing parameters
SI          32768
SF          500.1300471 MHz
WDW         EM
SSB         0
LB          0 Hz
GB          0
PC          1.40

F1 - Processing parameters
SI          65536
SF          500.1300471 MHz
WDW         EM
SSB         0
LB          0 Hz
GB          0
    
```





Effect of UV irradiation on the fluorescence of compound **37**, **36** and **38** (from left to right).  
(a) in the absence of UV light, (b) upon irradiation at 365 nm.

**Single crystal X-ray crystallographic structure of 38 CCDC 1852224**

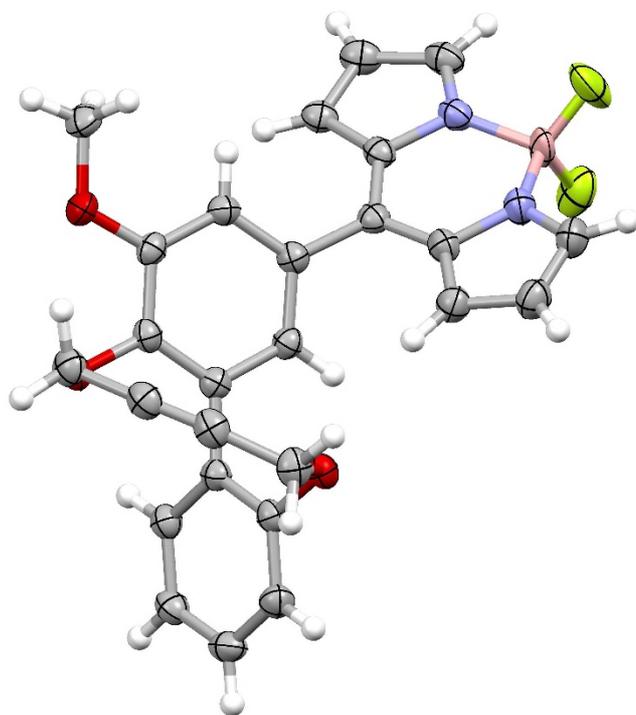


Figure 1: Single crystal X-ray structure of **38** (ellipsoids are plotted at the 50% probability level, disorder omitted for clarity)

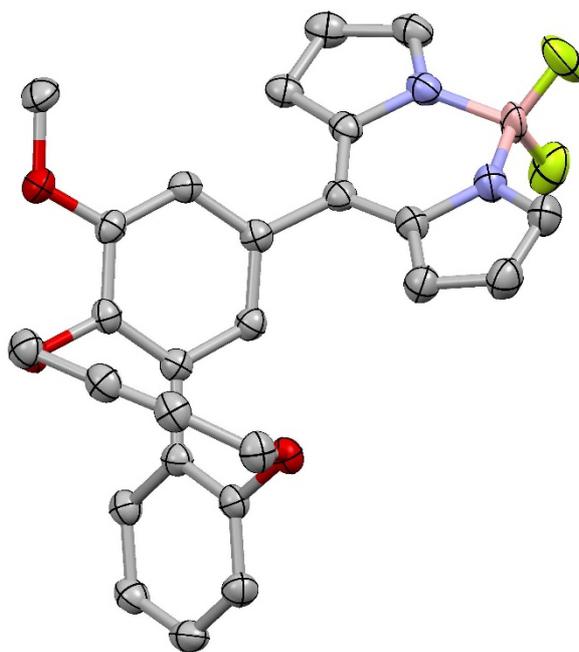


Figure 2: Single crystal X-ray structure of **38** (ellipsoids are plotted at the 50% probability level, disorder and H-atoms omitted for clarity)

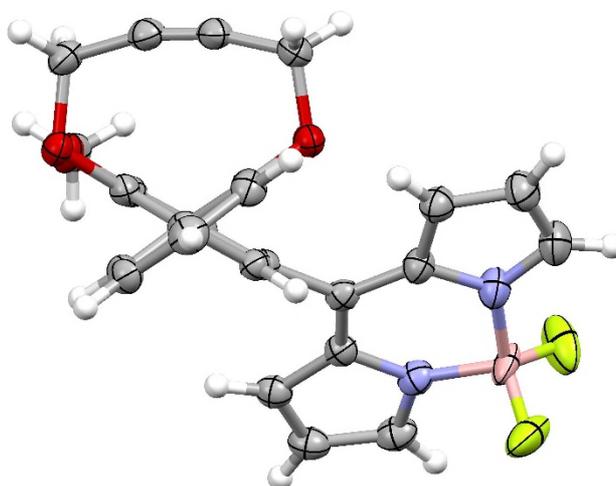


Figure 3: Single crystal X-ray structure of **38** (ellipsoids are plotted at the 50% probability level, disorder omitted for clarity)

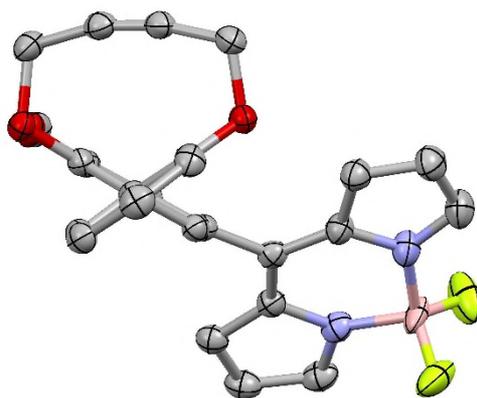


Figure 4: Single crystal X-ray structure of **38** (ellipsoids are plotted at the 50% probability level, disorder and H-atoms omitted for clarity)

**CCDC 1852224** contains the supplementary crystallographic data for this paper. These can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

Single crystals were grown from slow evaporation of a chloroform solution of the compound over several days. A suitable crystal was mounted on a Mitegen head with Fomblin oil and collected on an Xcalibur Gemini diffractometer with a Ruby CCD area detector at 150(2) K. The structure was solved using Olex2<sup>1</sup> and the ShelXT<sup>2</sup> structure solution program using Direct Methods and refined with the ShelXL<sup>3</sup> refinement package using Least Squares refinement.

The asymmetric unit contains one crystallographically distinct molecule, with  $Z = 4$ . The crystal packing indicates aromatic donor-acceptor ( $\pi$ - $\pi$ ) interactions of the biphenyl and BODIPY moieties of adjacent molecules in the crystal lattice.

The alkyne group displays significant deviation from linearity, the C14-C15-C16 and C15-C16-C17 bond angles are 165.7(4)° and 168.1(4)° respectively. This is accompanied by a large biphenyl torsion angle (C5-C6-C7-C8) of 68.6(4)°.

**Table 1: single-crystal X-ray data for compound 38**

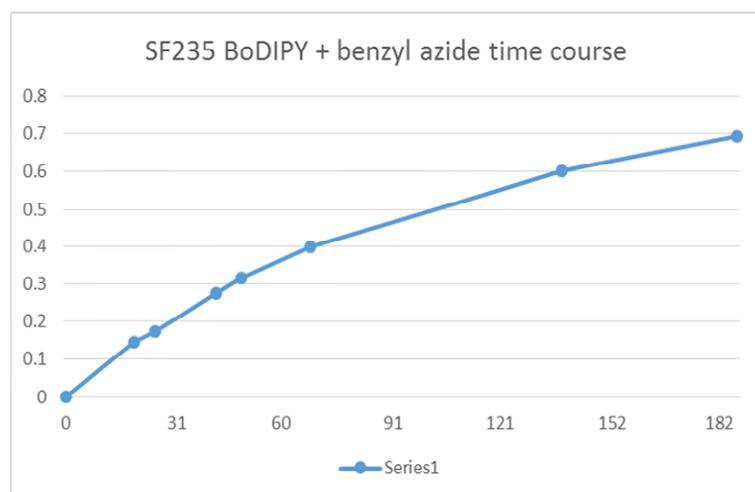
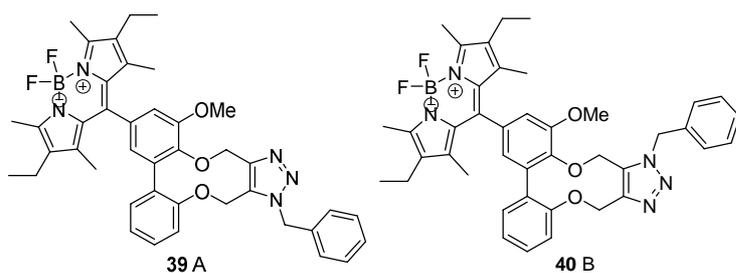
Compound Reference	Compound 38
Chemical Formula	C <sub>26</sub> H <sub>19</sub> BF <sub>2</sub> N <sub>2</sub> O <sub>3</sub>
Formula Mass	456.24
Crystal system	Monoclinic
$a/\text{Å}$	11.4282(2)
$b/\text{Å}$	25.2193(4)
$c/\text{Å}$	7.59768(13)
$\alpha/^\circ$	90
$\beta/^\circ$	104.5629(17)
$\gamma/^\circ$	90
Unit cell volume/ Å <sup>3</sup>	2119.39(6)

Temperature/ K	150(2) K
Space group	<i>-P 2ybc</i>
Crystal size/ mm	0.10 × 0.10 × 0.25
Radiation	CuK $\alpha$
Goodness-of-fit on F <sup>2</sup>	1.063
No. of formula units per unit cell, Z	4
No. of reflections measured	13289
No. of independent reflections	4488
Final R <sub>1</sub> values (I > 2 $\sigma$ (I))	0.0424
Final wR(F <sup>2</sup> ) values (I > 2 $\sigma$ (I))	0.1123
Final R <sub>I</sub> values (all data)	0.0449
Final wR(F <sup>2</sup> ) (all data)	0.1148

---

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K. & Puschmann, H. (2009), *J. Appl. Cryst.* 42, 339-341.
2. Sheldrick, G.M. (2015). *Acta Cryst.* A71, 3-8.
3. Sheldrick, G.M. (2015). *Acta Cryst.* C71, 3-8

**Click reactions using 36 to form two separated regioisomers 39 and 40 (SF235):**



Conversion / time (in hours) above.

Tentative assignment of regioisomers are shown. It is difficult to establish exactly which regioisomer is which in this case. However there is a distinctive difference in the  $^{13}\text{C}$  NMR spectra for the three  $\text{CH}_2$  groups (adjacent to heteroatoms) in each molecule. In the case where the pattern corresponds to that in the simpler compound lacking the OMe group (reference 9), and all its previously-reported derivatives, we have assumed that the benzyl group is further away from the OMe and therefore causes no distortion to the shape of the molecule (hence assigned as **39**). In the other regioisomer, we assume that the change is due to the closer proximity of the benzyl to the OMe, causing distortion to the ring and a change in the characteristic  $^{13}\text{C}$  NMR positions.

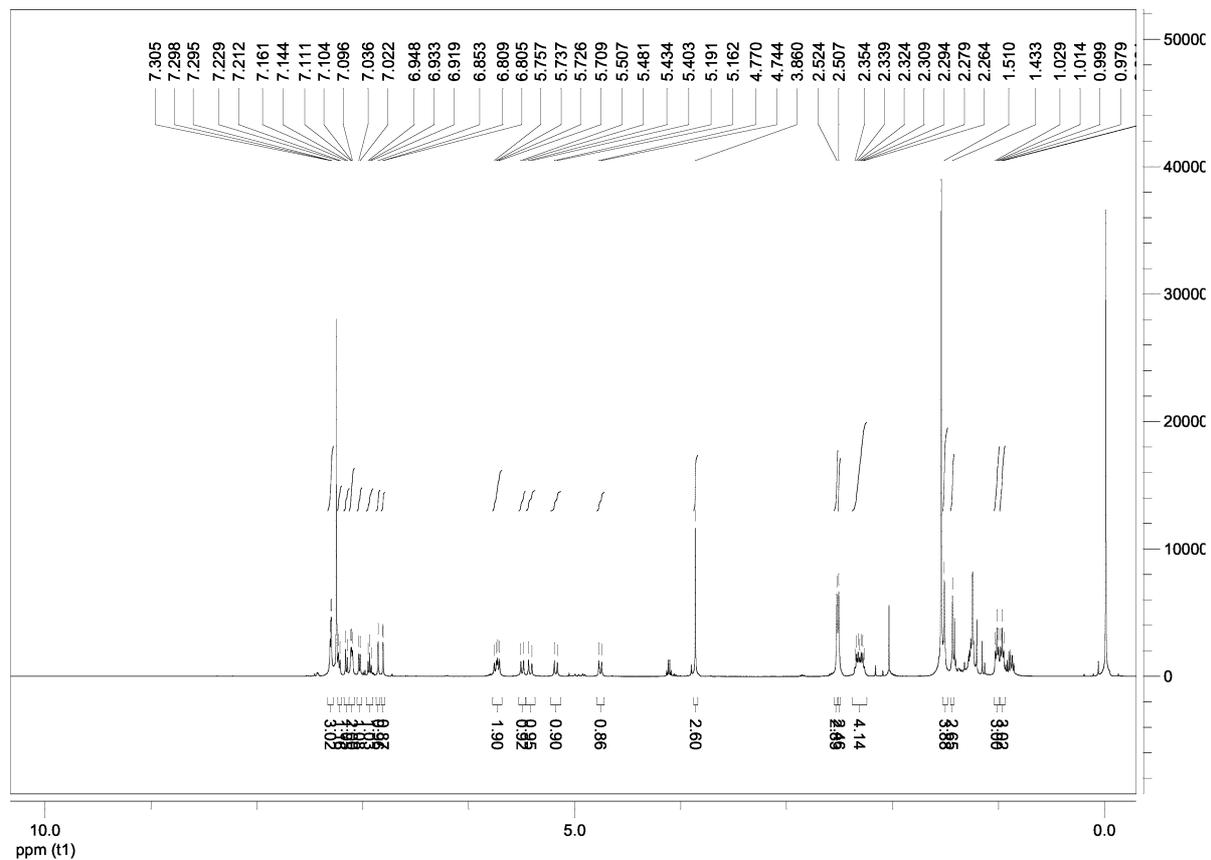
Compound **39**:  $\text{OCH}_2$  at 63.0 and 61.4 ppm,  $\text{NCH}_2$  at 52.0 ppm.

Compound **40**:  $\text{OCH}_2$  at 67.6 and 58.1 ppm,  $\text{NCH}_2$  at 52.9 ppm.

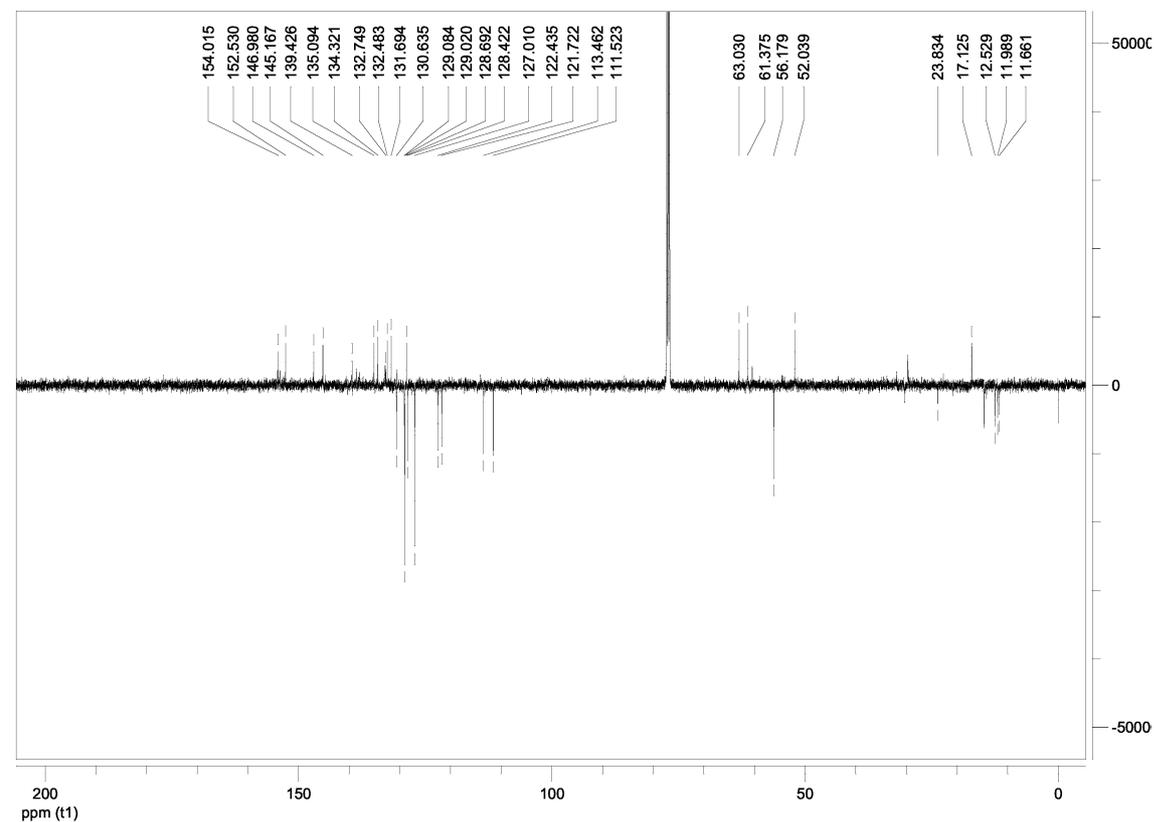
Compound lacking BoDIPY and OMe group (reported in reference 9):  $\text{OCH}_2$  at 62.9 and 60.5 ppm,  $\text{NCH}_2$  at 52.3 ppm. The two  $\text{OCH}_2$  groups are close in chemical shift, as in **39**.

### Regioisomer A:

$\delta_H$  (500 MHz,  $CDCl_3$ ).

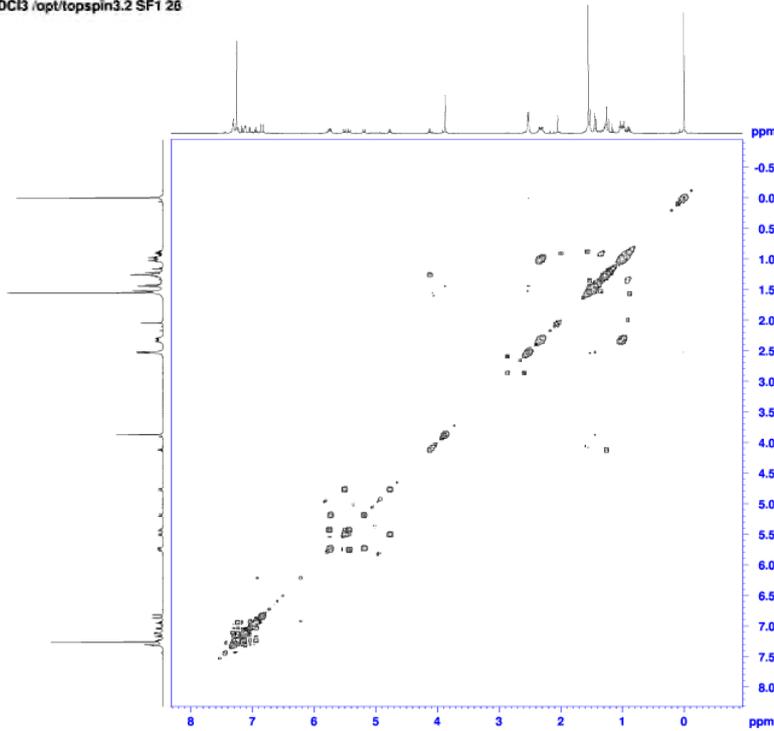


$\delta_C$  (126 MHz,  $CDCl_3$ ).



# COSY:

Chemist Sam Forshaw  
SF235A  
COSY.w CDCl3 /opt/topspin3.2 SF1 28



```
Current Data Parameters
NAME Jan10-2018
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180110
Time 15.55
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 4638.215 Hz
F2RES 2.264765 Hz
AQ 0.2207761 sec
RG 165.11
DM 101.800 usec
DE 85.00 usec
TE 300.2 K
D0 0.0000000 sec
D1 1.4188799 sec
D11 0.0300000 sec
D12 0.0000000 sec
D13 0.0000000 sec
D14 0.0000000 sec
D16 0.0000000 sec
TNO 0.0002180 sec

===== CHANNEL f1 =====
NUC1 13C
P1 18.00 usec
P12 0.0000000 usec
PL1 2000.000 MHz
PL12 13.00000000 MHz
PL12 1.52449997 MHz

===== GRABF2 CHANNEL =====
GRABF2[1] SMO210.100
GRABF2[2] SMO210.100
P16 1000.00 usec

F1 - Acquisition parameters
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 4638.215 Hz
F1RES 2.264765 Hz
AQ 0.2207761 sec
RG 165.11
DM 101.800 usec
DE 85.00 usec
TE 300.2 K
D0 0.0000000 sec
D1 1.4188799 sec
D11 0.0300000 sec
D12 0.0000000 sec
D13 0.0000000 sec
D14 0.0000000 sec
D16 0.0000000 sec
TNO 0.0002180 sec

===== CHANNEL f2 =====
NUC2 1H
P2 9.10 usec
P21 0 usec
P22 18.00 usec
PL2 500.1305119 MHz
PL21 0 Hz
PL22 13.00000000 MHz
PL22 1.52449997 MHz

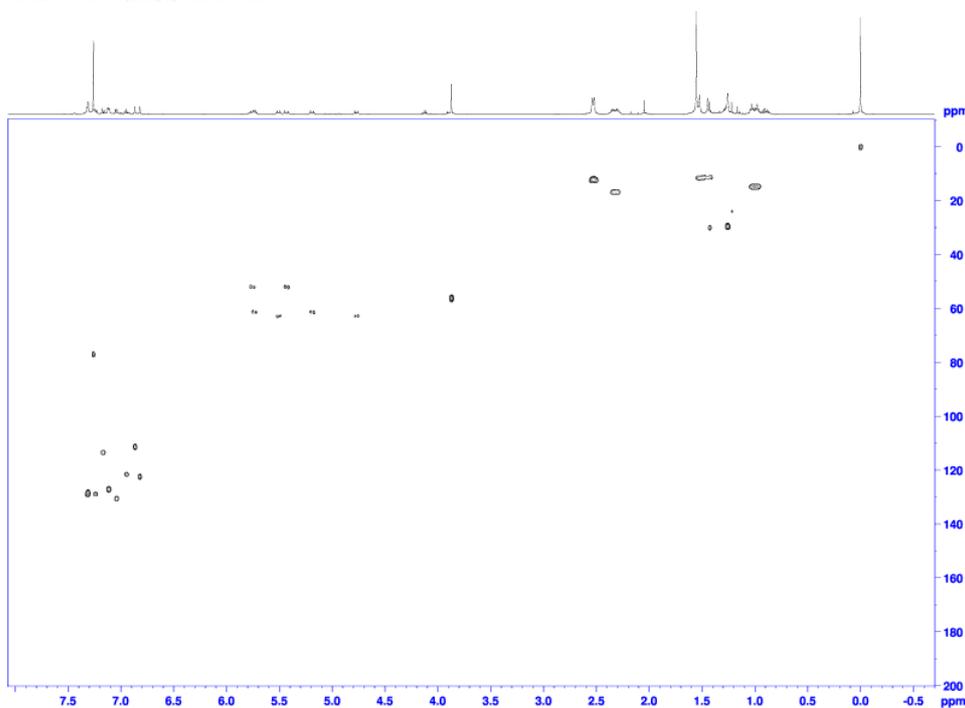
===== GRABF1 CHANNEL =====
GRABF1[1] SMO210.100
GRABF1[2] SMO210.100
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.1305119 MHz
WDW EM
SSB 0
GB 0 Hz
PC 1.40

F1 - Processing parameters
SI 32768
SF 500.1305119 MHz
WDW EM
SSB 0
GB 0 Hz
PC 1.40
```

# HSQC:

Chemist Sam Forshaw  
SF235A  
HSQC.w CDCl3 /opt/topspin3.2 SF1 28



```
Current Data Parameters
NAME Jan10-2018
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20180110
Time 16.01
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 4385.265 Hz
F2RES 4.282450 Hz
AQ 0.1187842 sec
RG 165.11
DM 114.000 usec
DE 80.00 usec
TE 300.2 K
D0 0.0000000 sec
D1 1.4300000 sec
D11 0.0300000 sec
D12 0.0000000 sec
D13 0.0000000 sec
D14 0.0000000 sec
D16 0.0000000 sec
TNO 0.0001890 sec
SOLVENT CDCl3

===== CHANNEL f1 =====
NUC1 13C
P1 18.00 usec
P12 0 usec
P13 18.00 usec
PL1 2000.000 MHz
PL12 13.00000000 MHz
PL13 1.52449997 MHz

===== CHANNEL f2 =====
NUC2 1H
P2 9.10 usec
P21 0 usec
P22 18.00 usec
PL2 500.1305119 MHz
PL21 0 Hz
PL22 13.00000000 MHz
PL22 1.52449997 MHz

===== GRABF2 CHANNEL =====
GRABF2[1] SMO210.100
GRABF2[2] SMO210.100
P16 1000.00 usec

F1 - Acquisition parameters
TD 65536
SOLVENT CDCl3
NS 4
DS 4
SWH 4385.265 Hz
F1RES 4.282450 Hz
AQ 0.1187842 sec
RG 165.11
DM 114.000 usec
DE 80.00 usec
TE 300.2 K
D0 0.0000000 sec
D1 1.4300000 sec
D11 0.0300000 sec
D12 0.0000000 sec
D13 0.0000000 sec
D14 0.0000000 sec
D16 0.0000000 sec
TNO 0.0001890 sec

===== CHANNEL f1 =====
NUC1 13C
P1 18.00 usec
P12 0 usec
P13 18.00 usec
PL1 2000.000 MHz
PL12 13.00000000 MHz
PL13 1.52449997 MHz

===== CHANNEL f2 =====
NUC2 1H
P2 9.10 usec
P21 0 usec
P22 18.00 usec
PL2 500.1305119 MHz
PL21 0 Hz
PL22 13.00000000 MHz
PL22 1.52449997 MHz

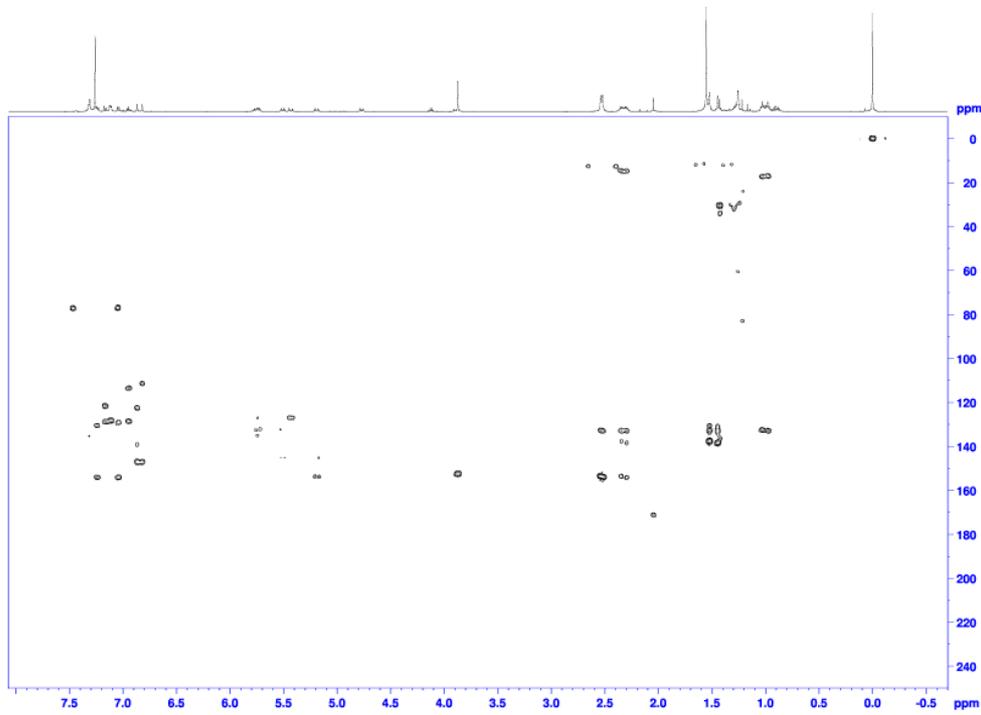
===== GRABF1 CHANNEL =====
GRABF1[1] SMO210.100
GRABF1[2] SMO210.100
P16 1000.00 usec

F2 - Processing parameters
SI 32768
SF 500.1305119 MHz
WDW EM
SSB 0
GB 0 Hz
PC 1.40

F1 - Processing parameters
SI 32768
SF 500.1305119 MHz
WDW EM
SSB 0
GB 0 Hz
PC 1.40
```

# HMBC:

HMBC.w CDC13 /opt/topspin3.2 SF1 28

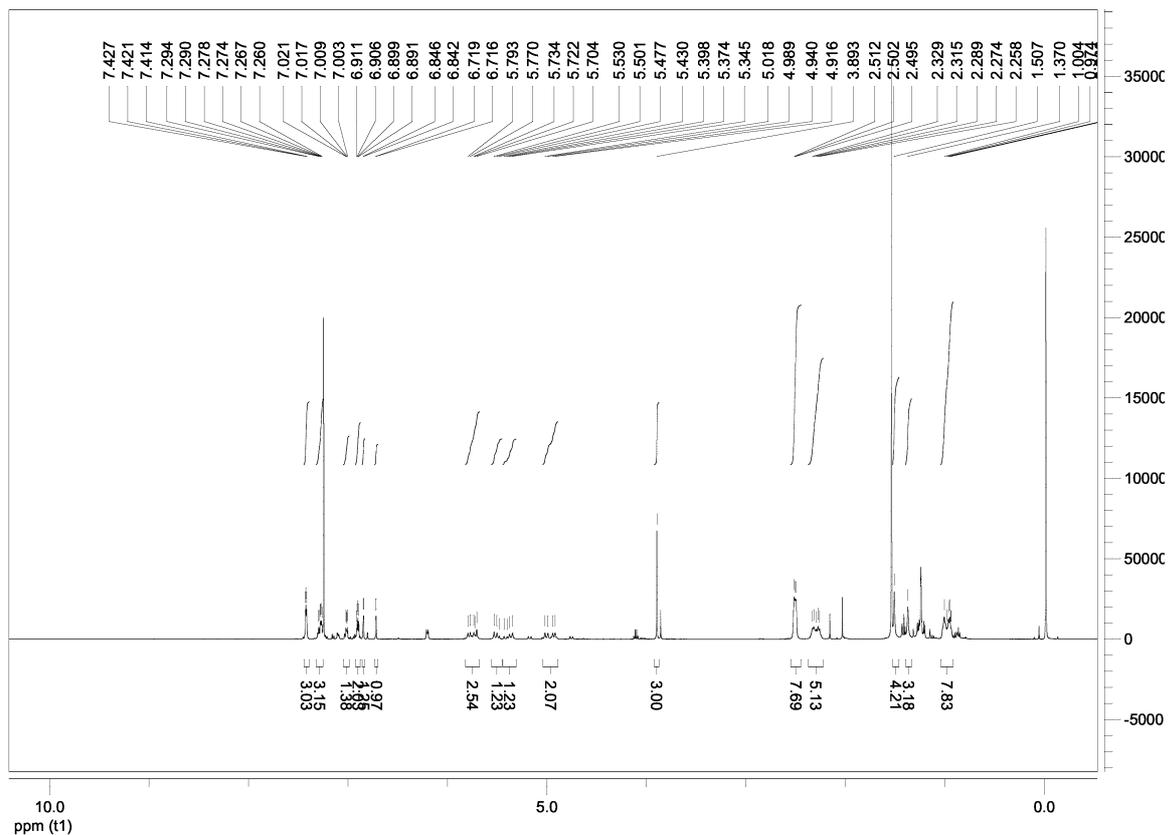


```

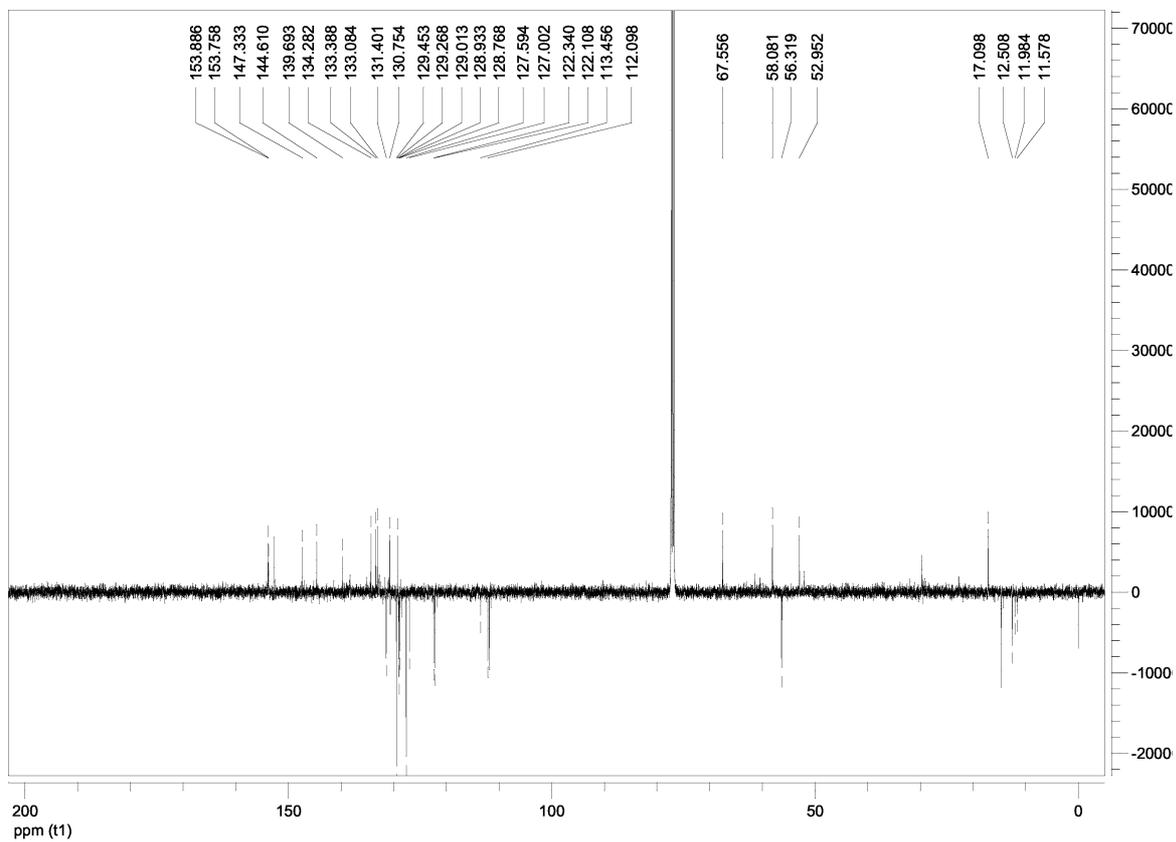
Current Data Parameters
NAME      Jan10-2018
PROCNO    1
----- Acquisition Parameters
Date_     20180510
Time      16.16
INSTRUM   spect
PROBHD    5 mm CPDQX 13C
PULPROG   hbhpcgpp04f
TD         65536
SOLVENT   CDCl3
DE         8
DS         16
SWH        4381.565 Hz
F2RES      4.28219 Hz
AQ         0.1187562 sec
RG         187.50
DM         114.000 usec
DE         60.00 usec
TE         298.0 K
CMT2      145.0000000
CMET13    10.0000000
DC         0.0000000 sec
D1         1.4500000 sec
D2         0.2000000 sec
D3         0.0000000 sec
D4         0.0000000 sec
D5         0.0000000 sec
D6         0.0000000 sec
D7         0.0000000 sec
D8         0.0000000 sec
D9         0.0000000 sec
----- CHANNEL f1 -----
SFO1      500.1318166 MHz
NUC1       13C
P1         9.10 usec
PL1        0.00 dB
SFO2      125.7727935 MHz
NUC2       1H
P2         9.10 usec
PL2        0.00 dB
----- CHANNEL f2 -----
SFO3      500.1318166 MHz
NUC3       13C
P3         9.10 usec
PL3        0.00 dB
----- CHANNEL f3 -----
SFO4      500.1318166 MHz
NUC4       13C
P4         9.10 usec
PL4        0.00 dB
----- CHANNEL f4 -----
SFO5      500.1318166 MHz
NUC5       13C
P5         9.10 usec
PL5        0.00 dB
----- Acquisition Parameters
TD         65536
SFO1      500.1318166 MHz
SFO2      125.7727935 MHz
P1R68     205.210455 Hz
DM         259.850 usec
RG         187.50
DE         60.00 usec
TE         298.0 K
----- Processing parameters
SI         32768
SF         500.1300119 MHz
WDW        EM
SSB        0
GB         0
PC         1.60
----- Processing parameters
SI         32768
SF         125.7717865 MHz
WDW        EM
SSB        0
GB         0
PC         1.60
  
```

**Regioisomer B:**

$\delta_H$  (500 MHz,  $CDCl_3$ ).

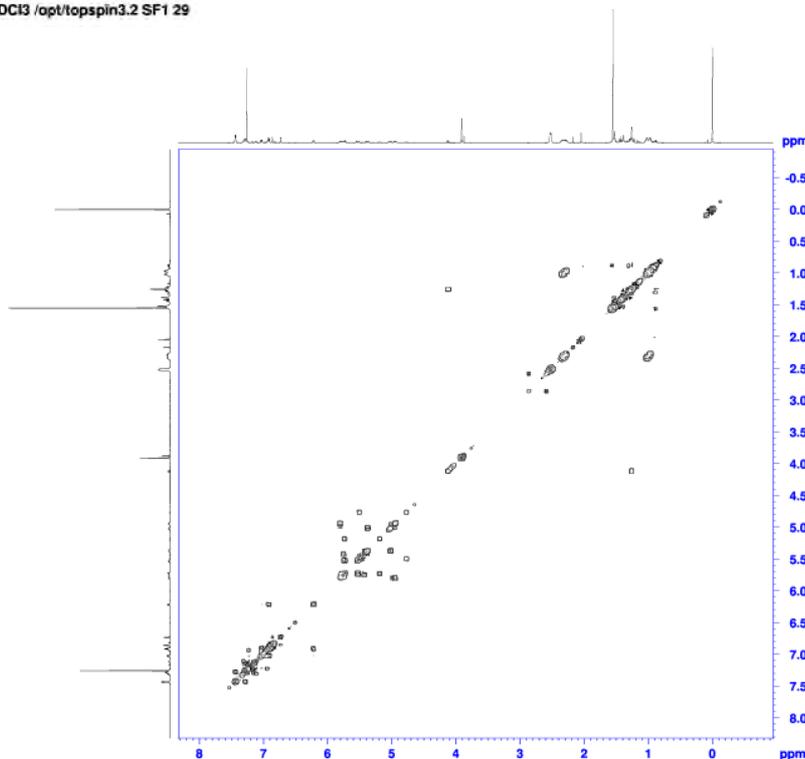


$\delta_C$  (126 MHz,  $CDCl_3$ ).



# COSY:

Chemist Sam Forshaw  
SF235B  
COSY.w CDCI3 /opt/topspin3.2 SF1 29



```
Current Data Parameters
NAME      Jan10-2018
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20180110
Time     17.18
INSTRUM  spect
PROBHD   5 mm CPDOR 13C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        2
DS        4
SWH       4638.219 Hz
FIDRES   0.264755 Hz
AQ        0.200746 sec
RG        385.92
AQ        107.800 usec
DE        40.00 usec
TE        298.0 K
D0        0.0000300 sec
D1        1.9180789 sec
D11       0.0000000 sec
D12       0.0000000 sec
D13       0.0000400 sec
D16       0.0000000 sec
IND       0.0002188 sec

===== CHANNEL f1 =====
SFO1     500.130870 MHz
NUC1     13
P1        9.10 usec
P12      9.10 usec
P17      2500.00 usec
PLW1     13.0000000 W
PLW2     1.59249999 W

===== GRABBER CHANNEL =====
SFO2     100.626150 MHz
P2        10.00 usec
P22      1000.00 usec

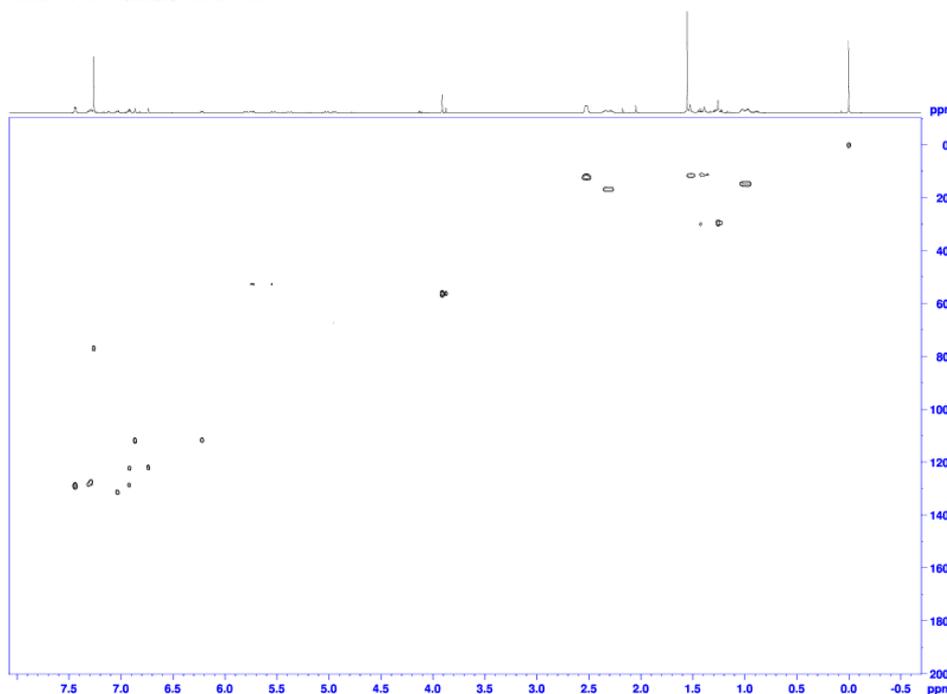
F1 - Acquisition parameters
TD        128
SFO1     500.130119 MHz
FIDRES   36.202803 Hz
SW        9.245 ppm
F0MHZ    0

F2 - Processing parameters
SI        1024
SF        500.1300119 MHz
WDW       GQ1MC
SSB       0
LB        0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
SFO1     500.1300119 MHz
WDW       GQ1MC
SSB       0
LB        0 Hz
GB        0
```

# HSQC:

Chemist Sam Forshaw  
SF235B  
HSQC.w CDCI3 /opt/topspin3.2 SF1 29



```
Current Data Parameters
NAME      Jan10-2018
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20180110
Time     17.24
INSTRUM  spect
PROBHD   5 mm CPDOR 13C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        2
DS        4
SWH       4382.862 Hz
FIDRES   0.1187340 Hz
AQ        0.181150 sec
RG        385.92
AQ        111.800 usec
DE        40.00 usec
TE        298.0 K
D0        0.0000000 sec
D1        1.4504402 sec
D4        0.0017414 sec
D11       0.0000000 sec
D12       0.0000000 sec
D16       0.0001890 sec
IND       0.0001890 sec

===== CHANNEL f1 =====
SFO1     500.130119 MHz
NUC1     13
P1        9.10 usec
P22      16.20 usec
PLW1     13.0000000 W

===== CHANNEL f2 =====
SFO2     100.626150 MHz
NUC2     13C
CFORC2   13C
P1        9.10 usec
P4        19.00 usec
P42       82.70 usec
PLW1     24.0000000 W
PLW2     0.11186003 W

===== GRABBER CHANNEL =====
SFO2     100.626150 MHz
SFO3     100.626150 MHz
SFO4     80.00 MHz
SFO5     20.10 MHz
P16      1000.00 usec

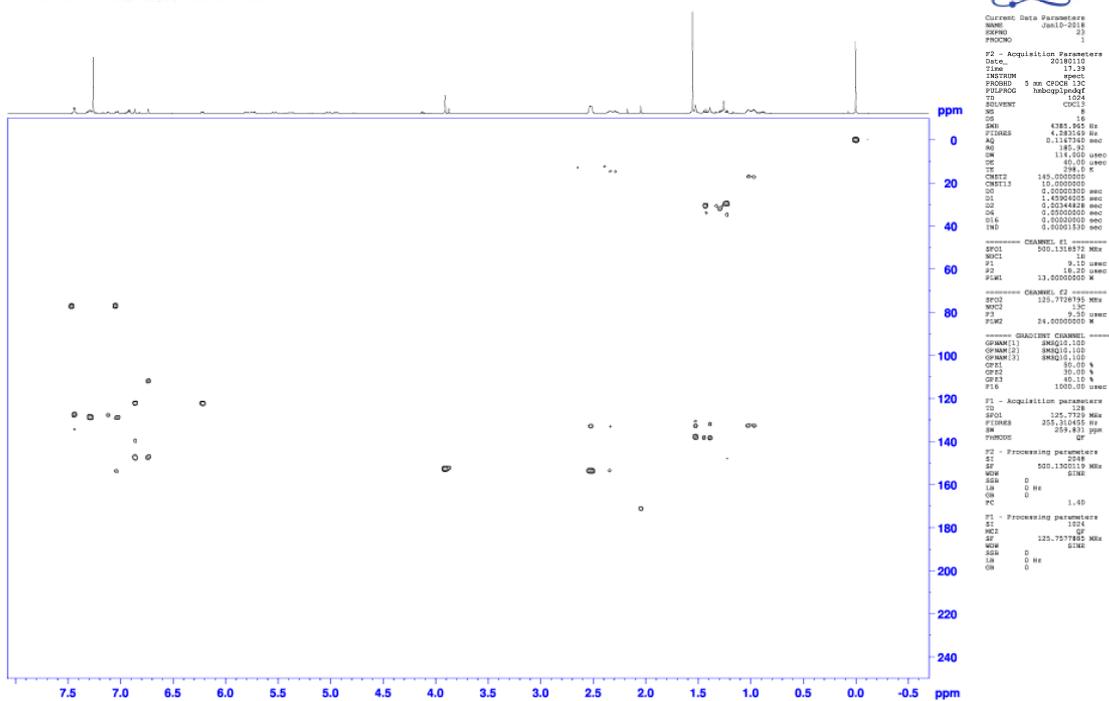
F1 - Acquisition parameters
TD        128
SFO1     500.130119 MHz
FIDRES   36.202803 Hz
SW        9.245 ppm
F0MHZ    0

F2 - Processing parameters
SI        1024
SF        500.1300119 MHz
WDW       GQ1MC
SSB       2
LB        0 Hz
GB        0
PC        1.40

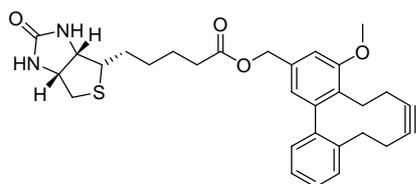
F1 - Processing parameters
SI        1024
SFO1     500.1300119 MHz
WDW       GQ1MC
SSB       2
LB        0 Hz
GB        0
```

# HMBC:

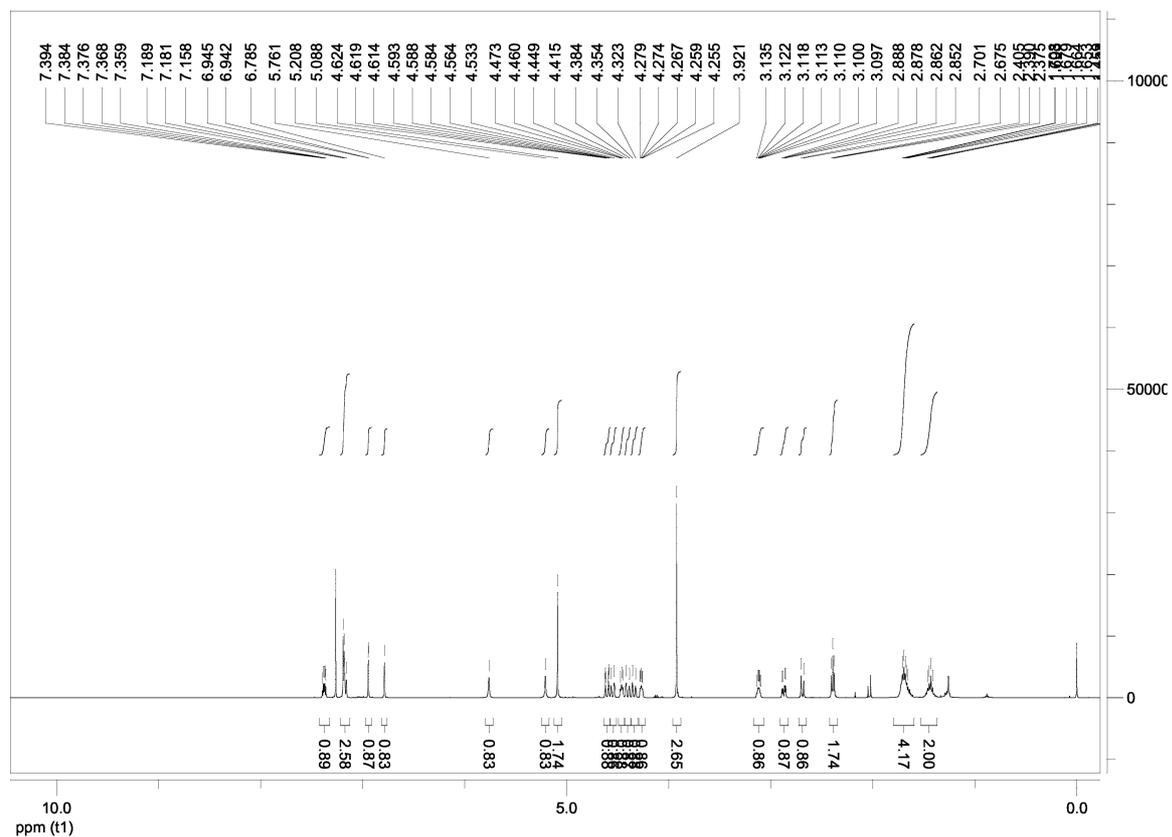
Chemist Sam Forshaw  
SF235B  
HMBC.w CDCl3 /opt/topspin3.2 SF1 29



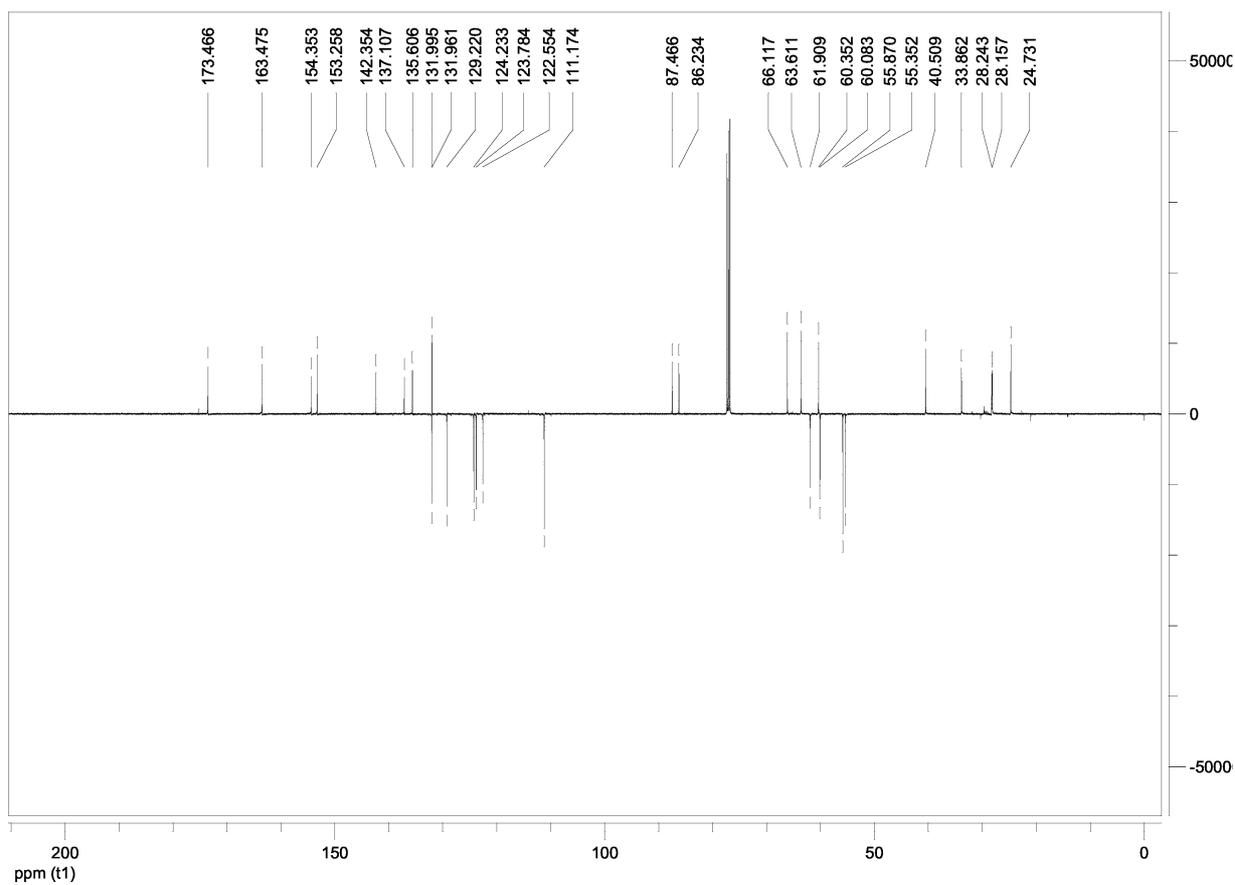
**Biotin functionalised alkyne 41. AM89, SF269 (data).**



$\delta_H$  (500 MHz,  $CDCl_3$ ).

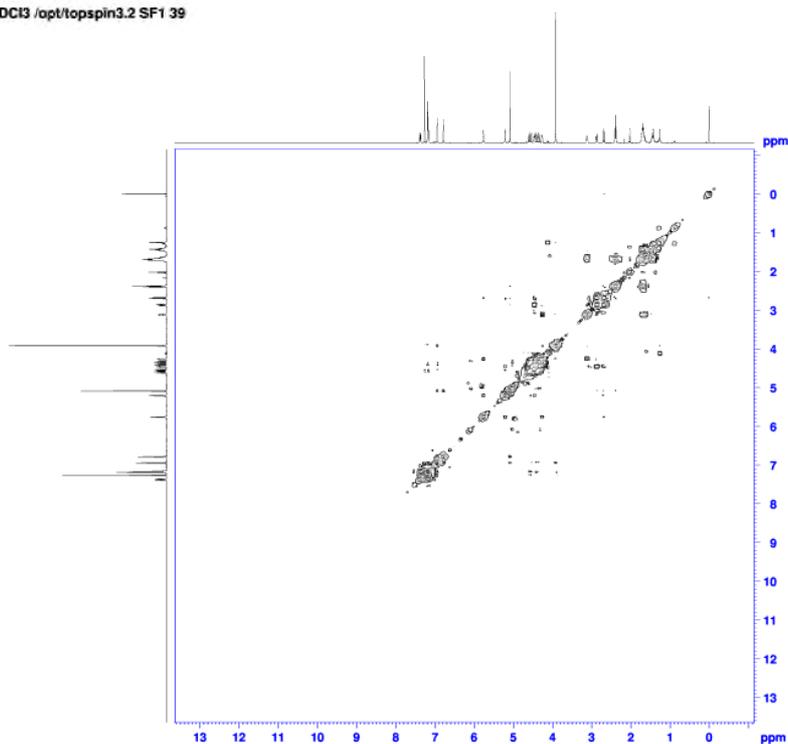


$\delta_C$  (126 MHz,  $CDCl_3$ ).



COSY:

Chemist Sam Forshaw  
SF269  
COSY.w  $CDCl_3$  /opt/topspin3.2 SF1 39



```

Current Data Parameters
NAME      Mar14-2018
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20180314
Time     20.50
INSTRUM  spect
PROBHD   5 mm CPDCH 13C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        1
DS        4
SWH       7396.450 Hz
FIDRES    3.611848 Hz
AQ        0.1384448 sec
RG         65.01
DE        67.600 usec
TE        300.2 K
D1         0.0000200 sec
D11        2.00819200 sec
D12        0.00002000 sec
D13        0.00002000 sec
D14        0.00002000 sec
D15        0.00002000 sec
D16        0.00002000 sec
D17        0.00013520 sec

===== CHANNEL f1 =====
SFO1     500.1361354 MHz
NUC1     13
P1        9.10 usec
PL1       0.10 usec
PL2       2500.00 usec
PL3       13.00000000 M
PL4       1.35043981 M

===== GRADIENT CHANNEL =====
GPRAM[1]  zmsq[0.100
           10.00 %
           1000.00 usec

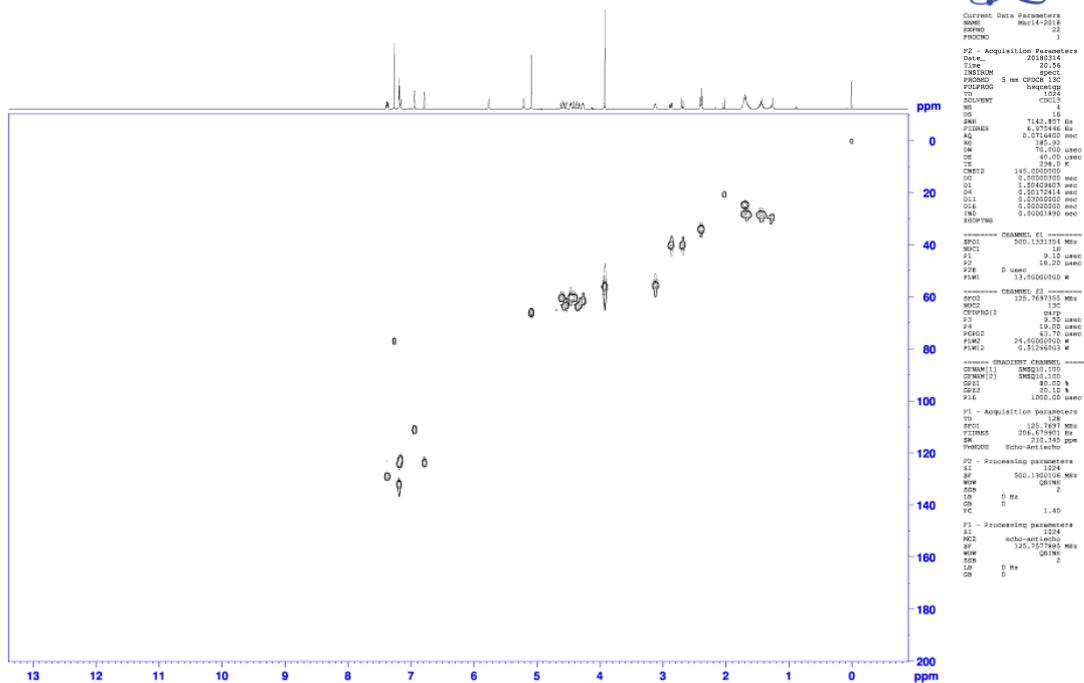
F1 - Acquisition parameters
TE        300.2 K
SFO1     500.1361354 MHz
FIDRES    3.611848 Hz
AQ        0.1384448 sec
RG         65.01
DE        67.600 usec
TE        300.2 K
D1         0.0000200 sec
D11        2.00819200 sec
D12        0.00002000 sec
D13        0.00002000 sec
D14        0.00002000 sec
D15        0.00002000 sec
D16        0.00002000 sec
D17        0.00013520 sec

F2 - Processing parameters
SI        65536
SF        500.1361354 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        65536
SF        500.1361354 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.40
    
```

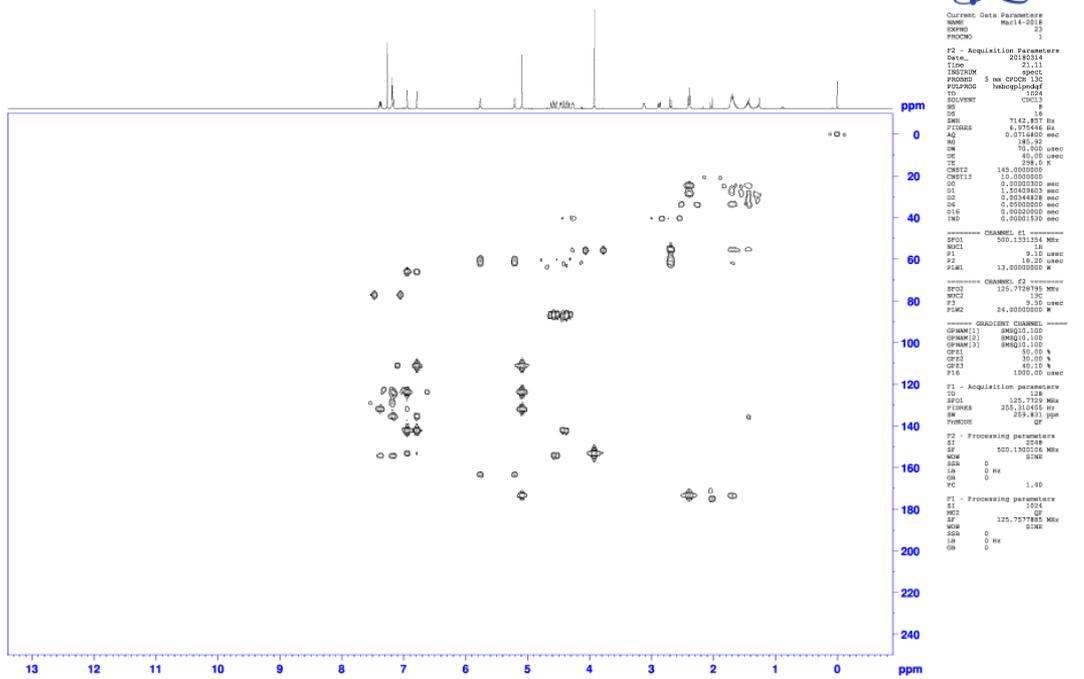
# HSQC:

Chemist Sam Forshaw  
SF269  
HSQC.w CDCI3 /opt/topspin3.2 SF1 39

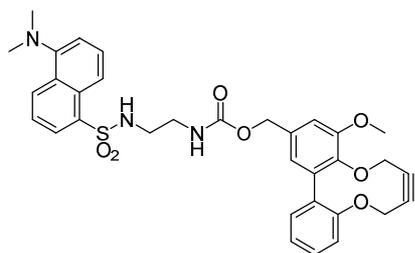


# HMBC:

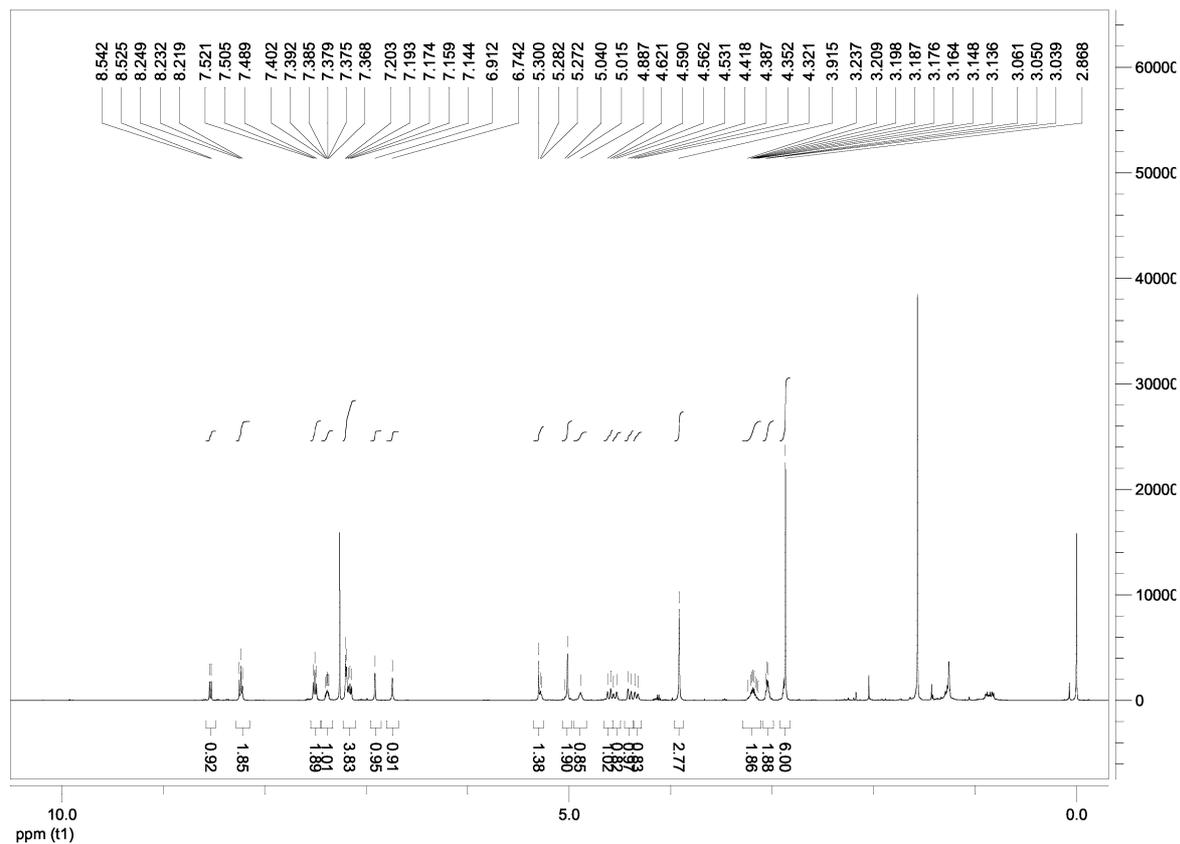
Chemist Sam Forshaw  
SF269  
HMBC.w CDCI3 /opt/topspin3.2 SF1 39



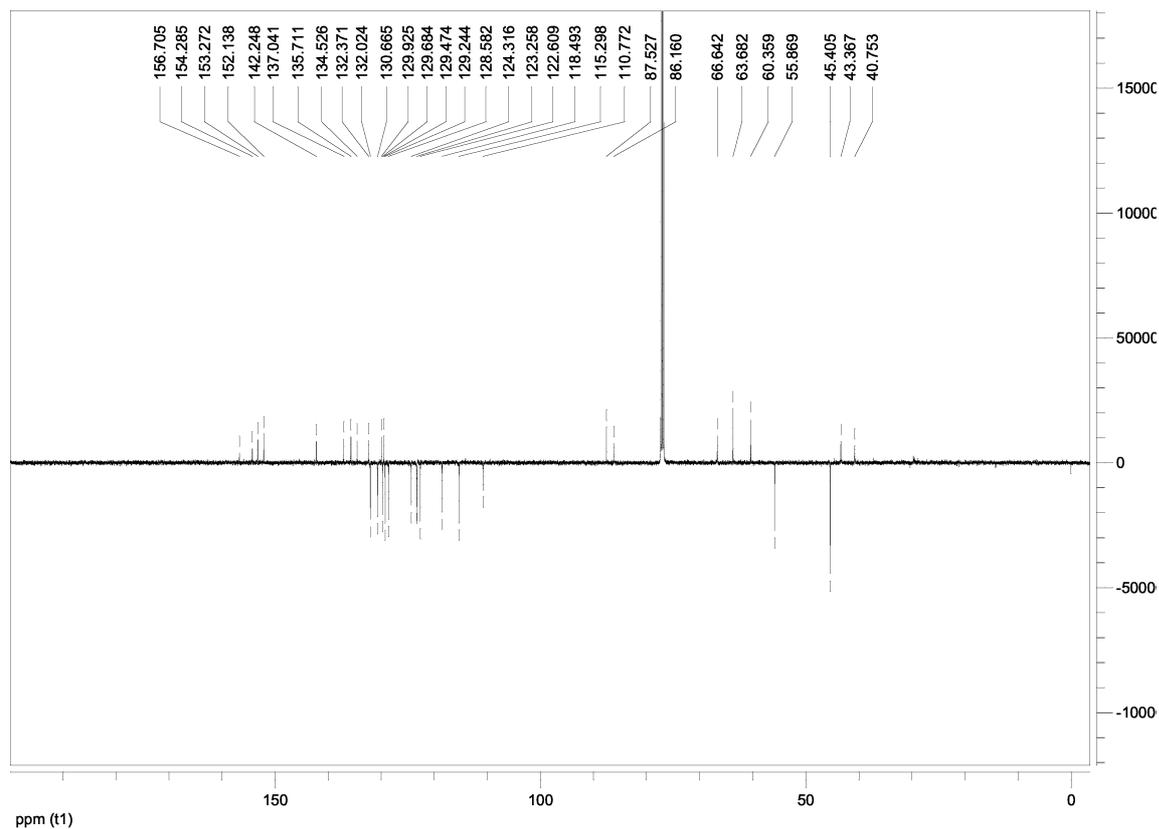
### Dansyl carbamate derivative 42 (SF268).



$\delta_H$  (500 MHz,  $CDCl_3$ ).

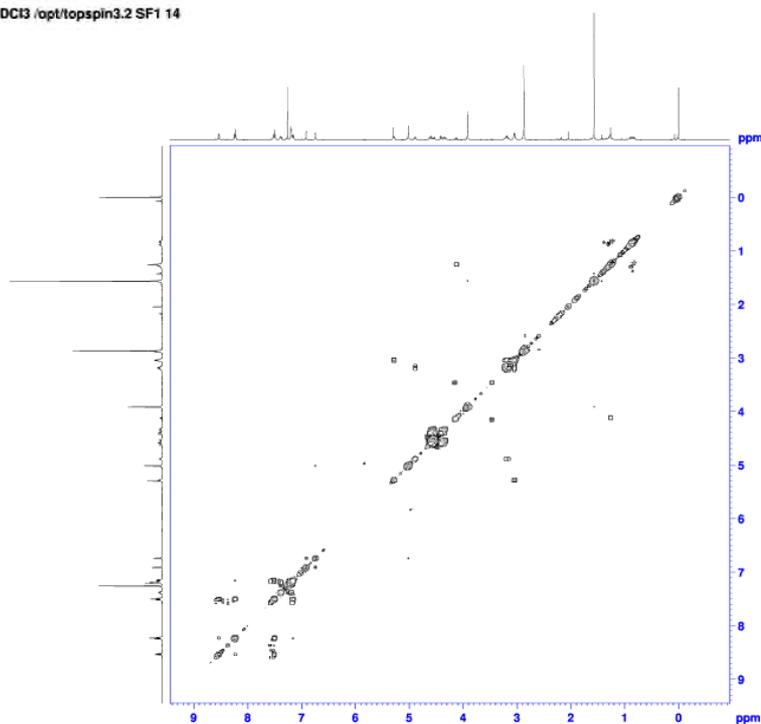


$\delta_C$  (126 MHz,  $CDCl_3$ ).



COSY

Chemist Sam Forshaw  
SF268  
COSY.w  $CDCl_3$  /opt/topspin.3.2 SF1 14



```

Current Data Parameters
NAME      Mar12-2018
EXPNO    21
PROCNO   1

F2 - Acquisition Parameters
Date_    20180312
Time     14.23
INSTRUM spect
PROBHD   5 mm CPDCH-13C
PULPROG zgpg30
TD        65536
SOLVENT  CDCl3
DE        234.6
DS        8
SI        5197.300 Hz
FIDRES   2.537844 Hz
AQ        0.1370776 sec
RG        385.82
RM        86.200 usec
DE        40.00 usec
TE        298.0 K
DQ        0.0000000 sec
D1        1.8447000 sec
D11       0.0300000 sec
D12       0.0000000 sec
D13       0.0000000 sec
D16       0.0000000 sec
TD0       0.00019220 sec

===== CHANNEL f1 =====
NUC1      13C
P1        9.10 usec
P2        9.10 usec
P3        2900.00 usec
PL1       11.0000000 W
PL2       1.3000000 W

===== GRAF1001 CHANNEL =====
SFOK(1)  SFOK(2) 10.00 %
P16      1000.00 usec

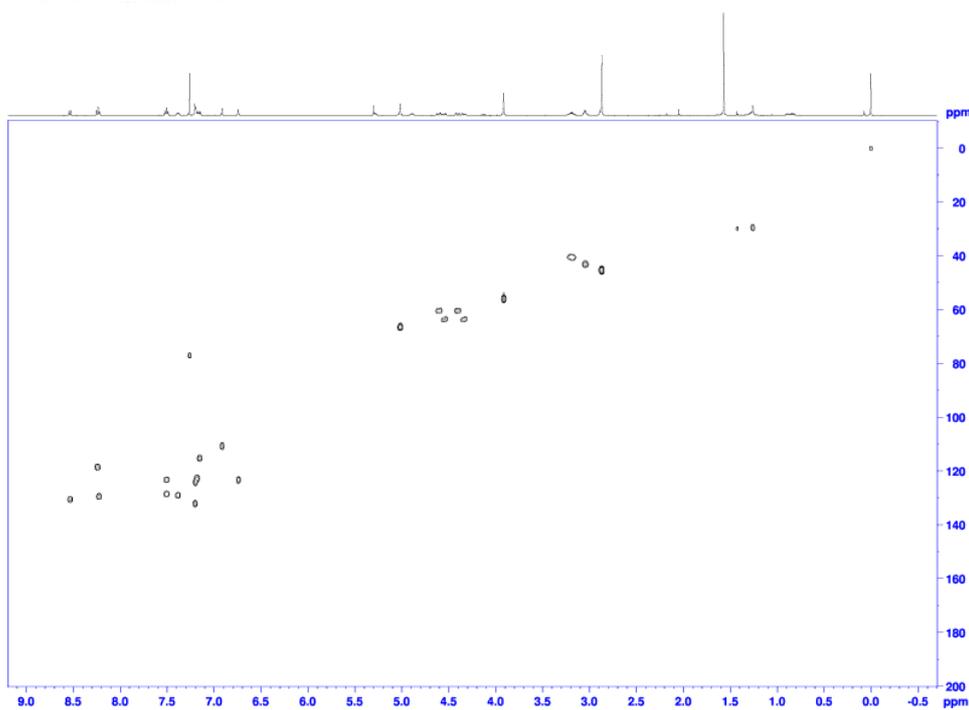
F1 - Acquisition Parameters
TD        65536
SOLVENT  CDCl3
FIDRES   40.647762 Hz
AQ        0.1370776 sec
RG        385.82
RM        86.200 usec
DE        40.00 usec
TE        298.0 K

F2 - Processing parameters
SI        65536
SF        500.1300116 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        65536
SF        500.1300116 MHz
WDW       EM
SSB       0
LB        0 Hz
GB        0
PC        1.40
    
```

# HSQC:

Chemist Sam Forshaw  
SF268  
HSQC.w CDCl3 /opt/topspin3.2 SF1 14



```

Current Data Parameters
NAME      Mar12-2018
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20180312
Time     16:29
INSTRUM  spect
PROBHD   5 mm CPQNP 13C
PULPROG  hbpgpg
TD        65536
SOLVENT  CDCl3
NS        4
DS        16
SWH       4950.492 Hz
FIDRES   4.834448 Hz
AQ        0.134240 sec
RG        185.92
SQ        101.600 usec
TE        300.2 K
NUC1      13C
NUC2      1H
CHFT2    149.000000 sec
DE        0.000000 sec
D1        1.4733203 sec
D4        0.20177414 sec
D11       0.2000000 sec
D16       0.2000000 sec
IND       0.0001830 sec

===== CHANNEL f1 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M
===== CHANNEL f2 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

===== GRADIENT CHANNEL =====
OPAM1[1]  SM4210.100
OPAM1[2]  SM4210.100
OP2       30.00 %
OP3       30.00 %
OP4       40.10 %
P16       1000.00 usec

F1 - Acquisition Parameters
TI        128
SF        125.76195 MHz
FIDRES   206.679801 Hz
AQ        0.134240 sec
RG        185.92
SQ        101.600 usec
TE        300.2 K
NUC1      13C
NUC2      1H
CHFT2    149.000000 sec
DE        0.000000 sec
D1        1.4733203 sec
D4        0.20177414 sec
D11       0.2000000 sec
D16       0.2000000 sec
IND       0.0001830 sec

===== CHANNEL f1 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

===== CHANNEL f2 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

===== GRADIENT CHANNEL =====
OPAM1[1]  SM4210.100
OPAM1[2]  SM4210.100
OP2       30.00 %
OP3       30.00 %
OP4       40.10 %
P16       1000.00 usec

F1 - Acquisition Parameters
TI        128
SF        125.76195 MHz
FIDRES   206.679801 Hz
AQ        0.134240 sec
RG        185.92
SQ        101.600 usec
TE        300.2 K
NUC1      13C
NUC2      1H
CHFT2    149.000000 sec
DE        0.000000 sec
D1        1.4733203 sec
D4        0.20177414 sec
D11       0.2000000 sec
D16       0.2000000 sec
IND       0.0001830 sec

===== CHANNEL f1 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

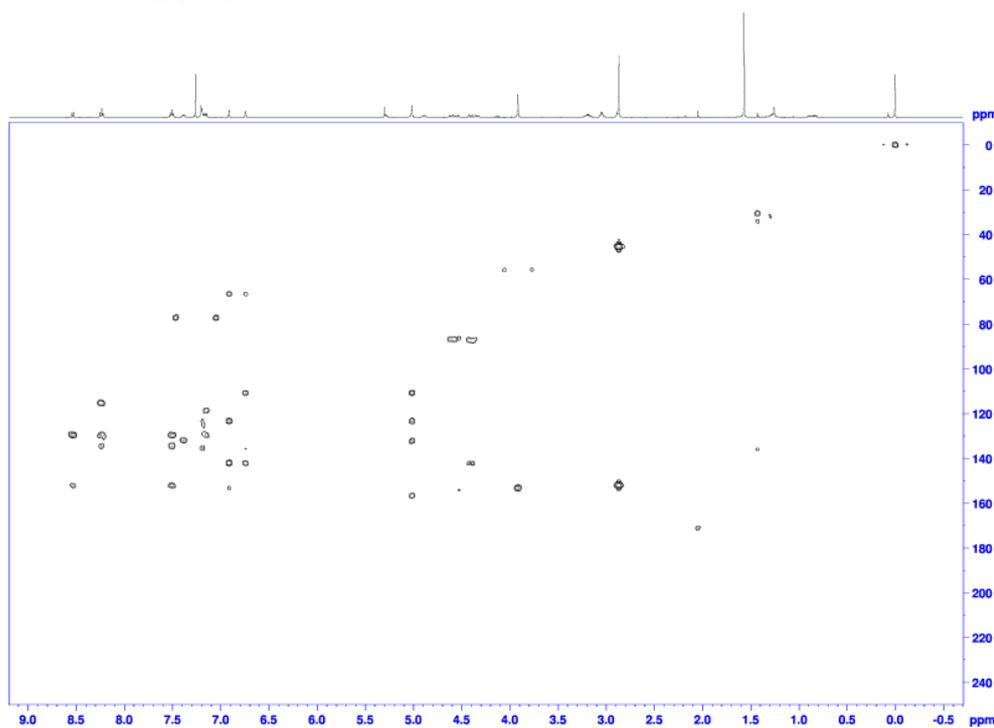
===== CHANNEL f2 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

===== GRADIENT CHANNEL =====
OPAM1[1]  SM4210.100
OPAM1[2]  SM4210.100
OP2       30.00 %
OP3       30.00 %
OP4       40.10 %
P16       1000.00 usec

F1 - Acquisition Parameters
TI        128
SF        125.76195 MHz
FIDRES   206.679801 Hz
AQ        0.134240 sec
RG        185.92
SQ        101.600 usec
TE        300.2 K
NUC1      13C
NUC2      1H
CHFT2    149.000000 sec
DE        0.000000 sec
D1        1.4733203 sec
D4        0.20177414 sec
D11       0.2000000 sec
D16       0.2000000 sec
IND       0.0001830 sec
    
```

# HMBC:

Chemist Sam Forshaw  
SF268  
HMBC.w CDCl3 /opt/topspin3.2 SF1 14



```

Current Data Parameters
NAME      Mar12-2018
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20180312
Time     16:29
INSTRUM  spect
PROBHD   5 mm CPQNP 13C
PULPROG  hbpgpg
TD        65536
SOLVENT  CDCl3
NS        4
DS        16
SWH       4950.492 Hz
FIDRES   4.834448 Hz
AQ        0.134240 sec
RG        185.92
SQ        101.600 usec
TE        300.2 K
NUC1      13C
NUC2      1H
CHFT2    149.000000 sec
DE        0.000000 sec
D1        1.4733203 sec
D4        0.20177414 sec
D11       0.2000000 sec
D16       0.2000000 sec
IND       0.0001830 sec

===== CHANNEL f1 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M
===== CHANNEL f2 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

===== GRADIENT CHANNEL =====
OPAM1[1]  SM4210.100
OPAM1[2]  SM4210.100
OP2       30.00 %
OP3       30.00 %
OP4       40.10 %
P16       1000.00 usec

F1 - Acquisition Parameters
TI        128
SF        125.76195 MHz
FIDRES   206.679801 Hz
AQ        0.134240 sec
RG        185.92
SQ        101.600 usec
TE        300.2 K
NUC1      13C
NUC2      1H
CHFT2    149.000000 sec
DE        0.000000 sec
D1        1.4733203 sec
D4        0.20177414 sec
D11       0.2000000 sec
D16       0.2000000 sec
IND       0.0001830 sec

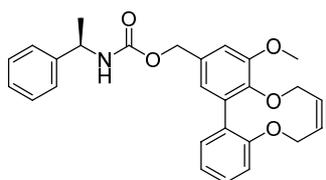
===== CHANNEL f1 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

===== CHANNEL f2 =====
NUC1      13C
NUC2      1H
P1        9.10 usec
P2        18.20 usec
P3        0 usec
P4        13.0000000 M

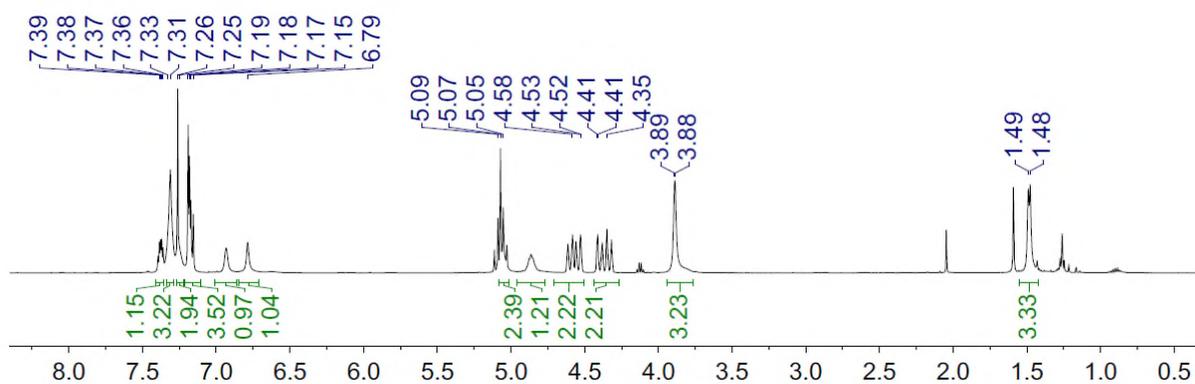
===== GRADIENT CHANNEL =====
OPAM1[1]  SM4210.100
OPAM1[2]  SM4210.100
OP2       30.00 %
OP3       30.00 %
OP4       40.10 %
P16       1000.00 usec

F1 - Acquisition Parameters
TI        128
SF        125.76195 MHz
FIDRES   206.679801 Hz
AQ        0.134240 sec
RG        185.92
SQ        101.600 usec
TE        300.2 K
NUC1      13C
NUC2      1H
CHFT2    149.000000 sec
DE        0.000000 sec
D1        1.4733203 sec
D4        0.20177414 sec
D11       0.2000000 sec
D16       0.2000000 sec
IND       0.0001830 sec
    
```

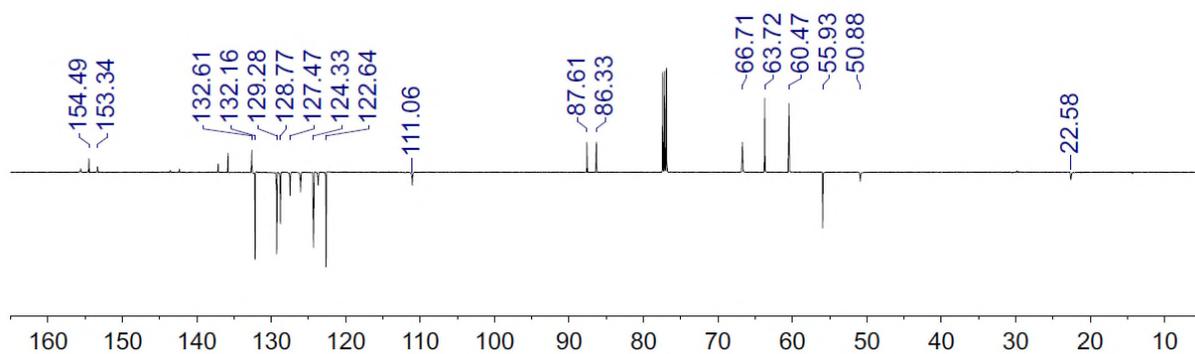
**Carbamate alkyne 43 (RK-3-220).**



$\delta_H$  (500 MHz,  $CDCl_3$ ).

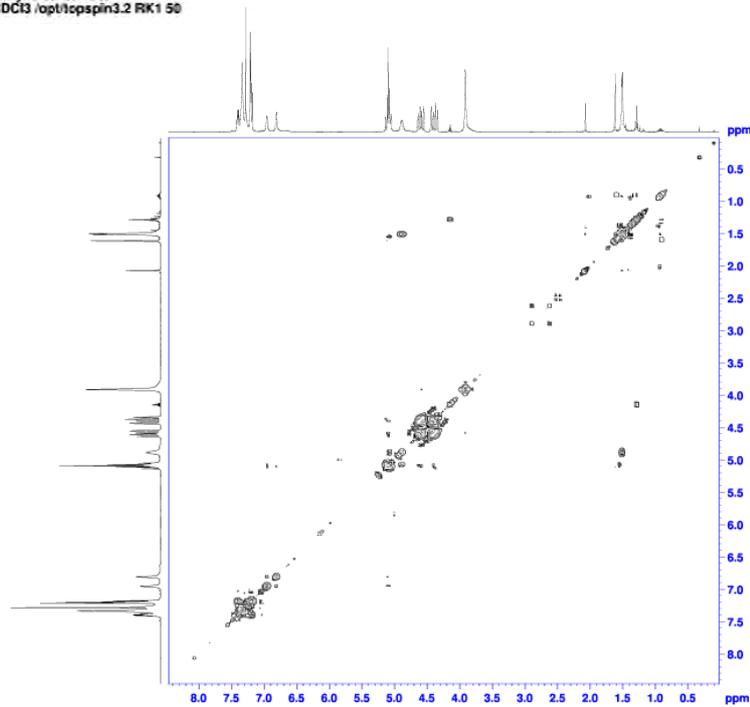


$\delta_C$  (126 MHz,  $CDCl_3$ ).



# COSY

Chemist Richard Knighton  
 RK-3-220 alkyne carbamate  
 COSY.w CDC13 /opt/topspin3.2 RK1 50



```

Current Data Parameters
NAME      Jan15-2018
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20180115
Time     8.16
INSTRUM  spect
PROBHD   5 mm CPDQX 13C
PULPROG  megarzgpg
TD        65536
SOLVENT  CDCl3
NS        2
DS        4
SWH       4222.313 Hz
FIDRES   2.261899 Hz
AQ        0.2424832 sec
RG         40.27
AQ        138.400 usec
TE        298.2 K
DQ        0.0000000 sec
D1        1.8930403 sec
D11       0.0000000 sec
D12       0.0000000 sec
D13       0.0000000 sec
D16       0.0000000 sec
TD0       0.0002100 sec

----- CHANNEL f1 -----
SFO1     500.1321221 MHz
NUC1     13C
P1        9.10 usec
P2        9.10 usec
P12       2000.00 usec
PL1       13.0000000 W
PL12      1.5924999 W

----- GRABF2 CHANNEL -----
SFO2     500.1321221 MHz
NUC2     13C
P1        9.10 usec
P2        9.10 usec
P12       2000.00 usec
PL1       13.0000000 W
PL12      1.5924999 W

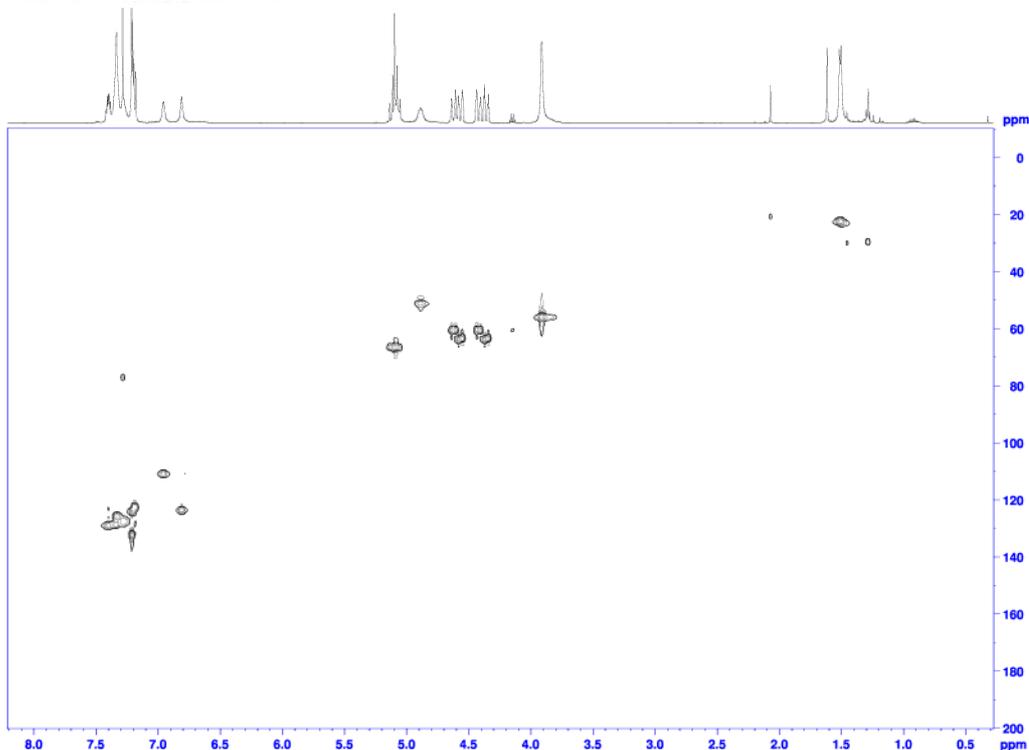
F1 - Acquisition parameters
TS        128
SFO1     500.1321 MHz
FIDRES   32.846134 Hz
SW        8.417 ppm
FWDONE   QF

F2 - Processing parameters
SI        1024
SF        500.1300000 MHz
WDW       0
SSB       0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
SF        500.1300000 MHz
WDW       0
SSB       0 Hz
GB        0
PC        1.40
  
```

# HSQC

Chemist Richard Knighton  
 RK-3-220 alkyne carbamate  
 HSQC.w CDC13 /opt/topspin3.2 RK1 50



```

Current Data Parameters
NAME      Jan15-2018
EXPNO    12
PROCNO   1

F2 - Acquisition Parameters
Date_    20180115
Time     8.22
INSTRUM  spect
PROBHD   5 mm CPDQX 13C
PULPROG  megarzgpg
TD        65536
SOLVENT  CDCl3
NS        2
DS        4
SWH       3968.254 Hz
FIDRES   2.875248 Hz
AQ        0.1350240 sec
RG         80.27
AQ        124.000 usec
TE        298.2 K
DQ        0.0000000 sec
D1        1.8487109 sec
D11       0.20172414 sec
D12       0.0000000 sec
D16       0.0000000 sec
TD0       0.00001890 sec

----- CHANNEL f1 -----
SFO1     500.1321221 MHz
NUC1     13C
P1        9.10 usec
P2        18.20 usec
P12       0 usec
PL1       13.0000000 W

----- CHANNEL f2 -----
SFO2     125.767355 MHz
NUC2     13C
CPDPRG2  gpg
P1        9.10 usec
P2        18.20 usec
P12       0 usec
PL1       24.0000000 W
PL12      0.11245000 W

----- GRABF2 CHANNEL -----
SFO2     125.767355 MHz
NUC2     13C
CPDPRG2  gpg
P1        9.10 usec
P2        18.20 usec
P12       0 usec
PL1       24.0000000 W
PL12      0.11245000 W

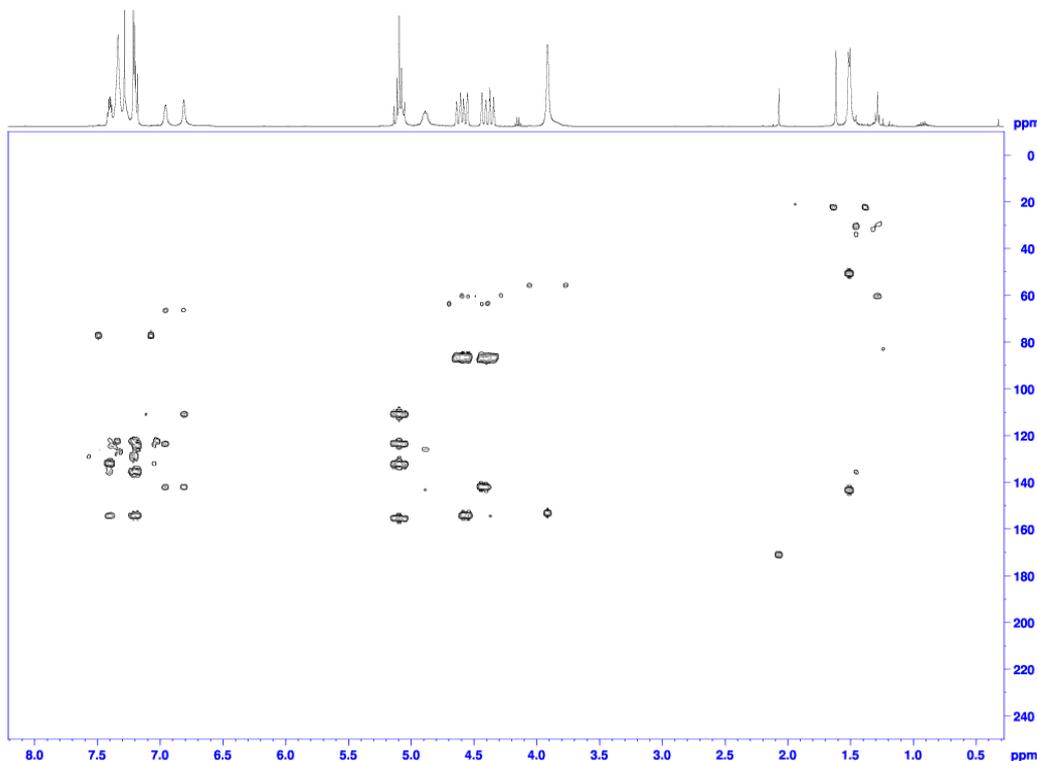
F1 - Acquisition parameters
TS        128
SFO1     125.767 MHz
FIDRES   205.679601 Hz
SW        212.343 ppm
FWDONE   Echo-Multisect

F2 - Processing parameters
SI        1024
SF        500.1300000 MHz
WDW       0
SSB       0 Hz
GB        0
PC        1.40

F1 - Processing parameters
SI        1024
SF        125.767355 MHz
WDW       0
SSB       0 Hz
GB        0
PC        1.40
  
```

# HMBC

HMBC.w CDCI3 /opt/topspin3.2 RK1 50  
kyne carbamate



Current Data Parameters  
NAME Jan13-2018  
EXPNO 13  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20180115  
Time 9.29  
INSTRUM spect  
PROBHD 5 mm CPMAS 125  
PULPROG hmcgpp1pdqf  
TD 124  
SOLVENT CDCl3  
NS 4  
DS 16  
SWH 3968.204 Hz  
FIDRES 3.875248 Hz  
AQ 0.1292000 sec  
RG 185.92  
DM 259.000 usec  
DE 40.00 usec  
TE 298.0 K  
CNS12 145.000000  
CNS13 10.000000  
D0 0.0000000 sec  
D1 1.4467195 sec  
D2 0.0034828 sec  
D3 1.0000000 sec  
D5 0.0002000 sec  
D6 0.0000000 sec  
D7 0.0000000 sec  
D8 0.0000000 sec

----- CHANNEL f1 -----  
SFO1 500.132127 MHz  
NUC1 1H  
P1 9.10 usec  
P2 16.00 usec  
PLA1 13.0000000 W

----- CHANNEL f2 -----  
SFO2 125.772795 MHz  
NUC2 13C  
P3 9.50 usec  
PLA2 24.0000000 W

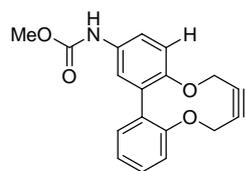
----- GRABBER CHANNEL -----  
GPRAM11 SMSG0.100  
GPRAM12 SMSG0.100  
GPRAM13 SMSG0.100  
GPRAM14 SMSG0.100  
GPRAM15 SMSG0.100  
GPRAM16 SMSG0.100  
GPRAM17 SMSG0.100  
GPRAM18 SMSG0.100  
GPRAM19 SMSG0.100  
GPRAM20 SMSG0.100

F1 - Acquisition parameters  
TD 124  
SFO1 125.7729 MHz  
FIDRES 259.318405 Hz  
SW 259.831 ppm  
PULPROG QP

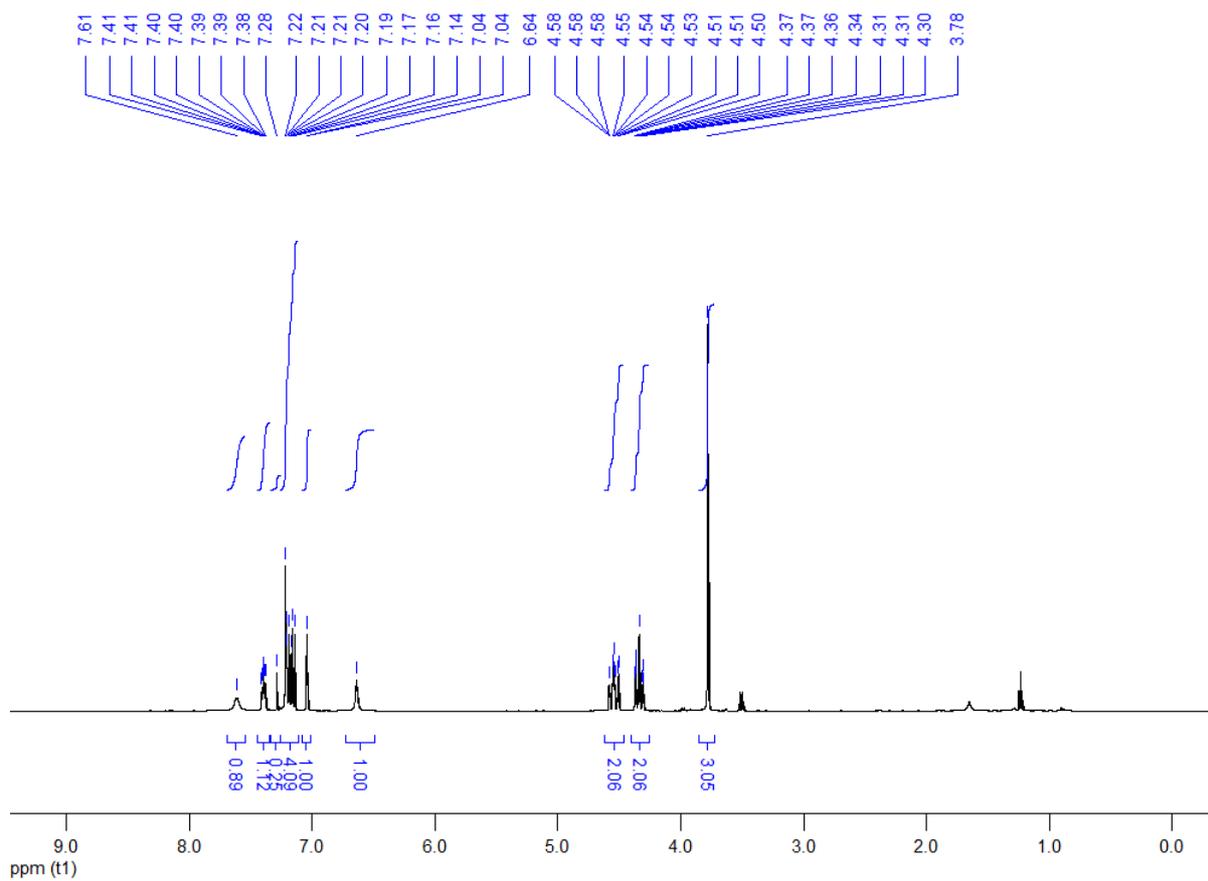
F2 - Processing parameters  
SI 32768  
SF 500.130000 MHz  
WDW SINE  
SSB 0  
LB 0 Hz  
GB 0  
PC 1.00

F1 - Processing parameters  
SI 1024  
SF 125.757795 MHz  
WDW SINE  
SSB 0  
LB 0 Hz  
GB 0

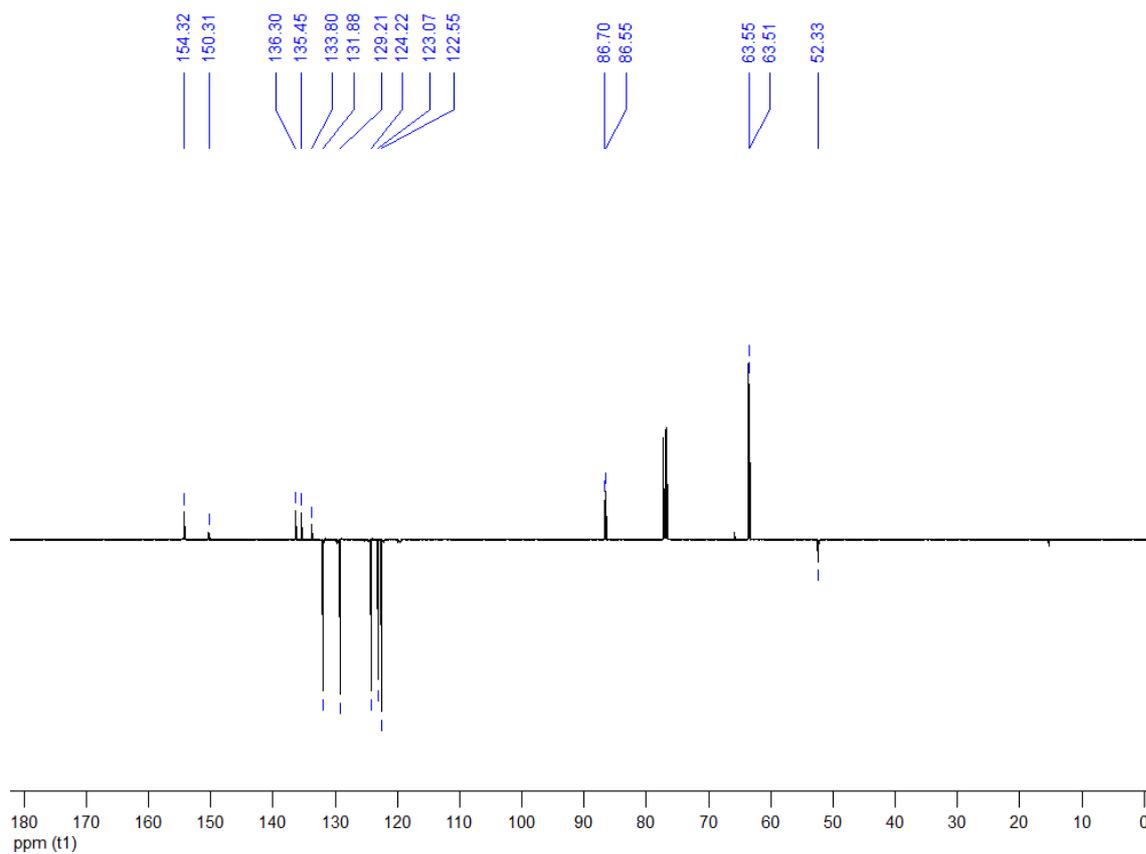
**Carbamate 44. (AM102).**



$\delta_H$  (500 MHz,  $CDCl_3$ ).



$\delta_C$  (126 MHz,  $CDCl_3$ ).



COSY

Chemist Anish Mistry  
AM-102  
COSY.w  $CDCl_3$  /opt/topspin3.2 AM 13



```

Current Data Parameters
NAME      April-2013
EXPNO    11
PROCNO   1

P2 - Acquisition Parameters
Date_    20170419
Time     15:33
INSTRUM  spect
PROBHD   5 mm CPDQX 13C
PULPROG  zgpg30
TD        65536
SOLVENT  CDCl3
NS        3
DS        4
SWH       4485.646 Hz
FIDRES    2.180297 Hz
AQ        0.3282837 sec
RG         60.07
DM        111.462 usec
DE        288.0 Hz
TE        300.2 K
D0        0.0000300 sec
D1        1.000000000 sec
D11       0.030000000 sec
D12       0.000000000 sec
D13       0.000000000 sec
D16       0.000000000 sec
RG0       0.0002288 sec

----- CHANNEL f1 -----
NUC1      13C
P2        10 usec
P1        8.10 usec
P12       2000.00 usec
P1M1     13.00000000 W
P1M2     1.33248993 V

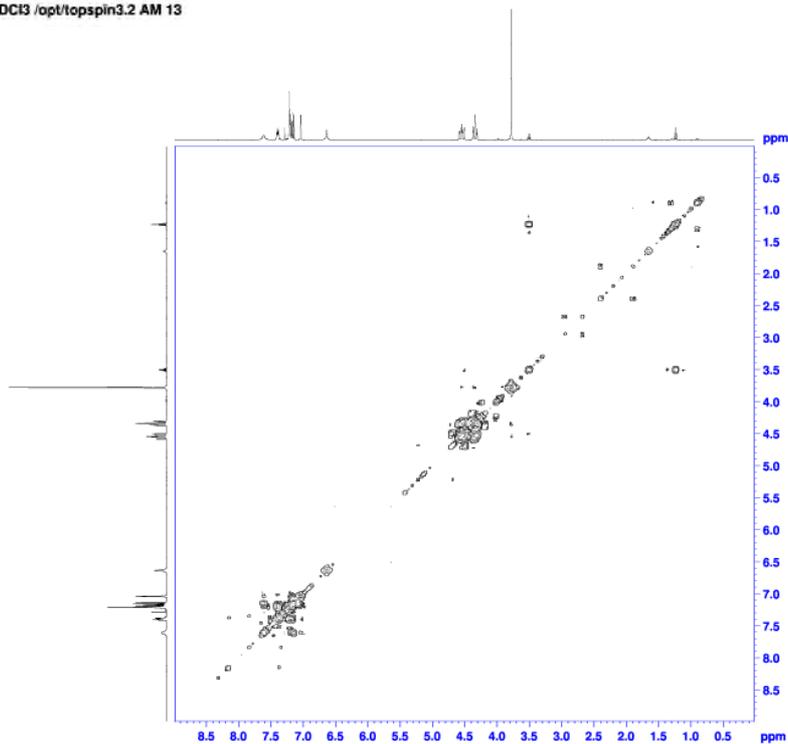
----- GRADIENT CHANNEL -----
GPMAX1[] 88000.100
SFO1[]    10.00 MHz
P16       1000.00 usec

P1 - Acquisition parameters
P2        10 usec
SFO1      500.132524 MHz
FIDRES    35.045083 Hz
CK        4.974 ppm
F0H0      0

P2 - Processing parameters
SI        1024
SF        500.1300000 MHz
SFO1      500.132524 MHz
GB        0 Hz
GB1       0 Hz
GB2       0 Hz
PC        1.40

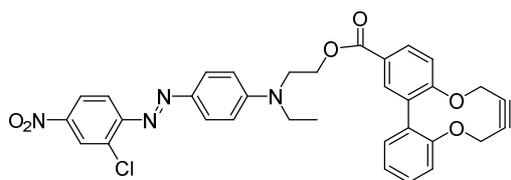
P1 - Processing parameters
SI        1024
SF        500.1300000 MHz
SFO1      500.132524 MHz
GB        0 Hz
GB1       0 Hz
GB2       0 Hz

```

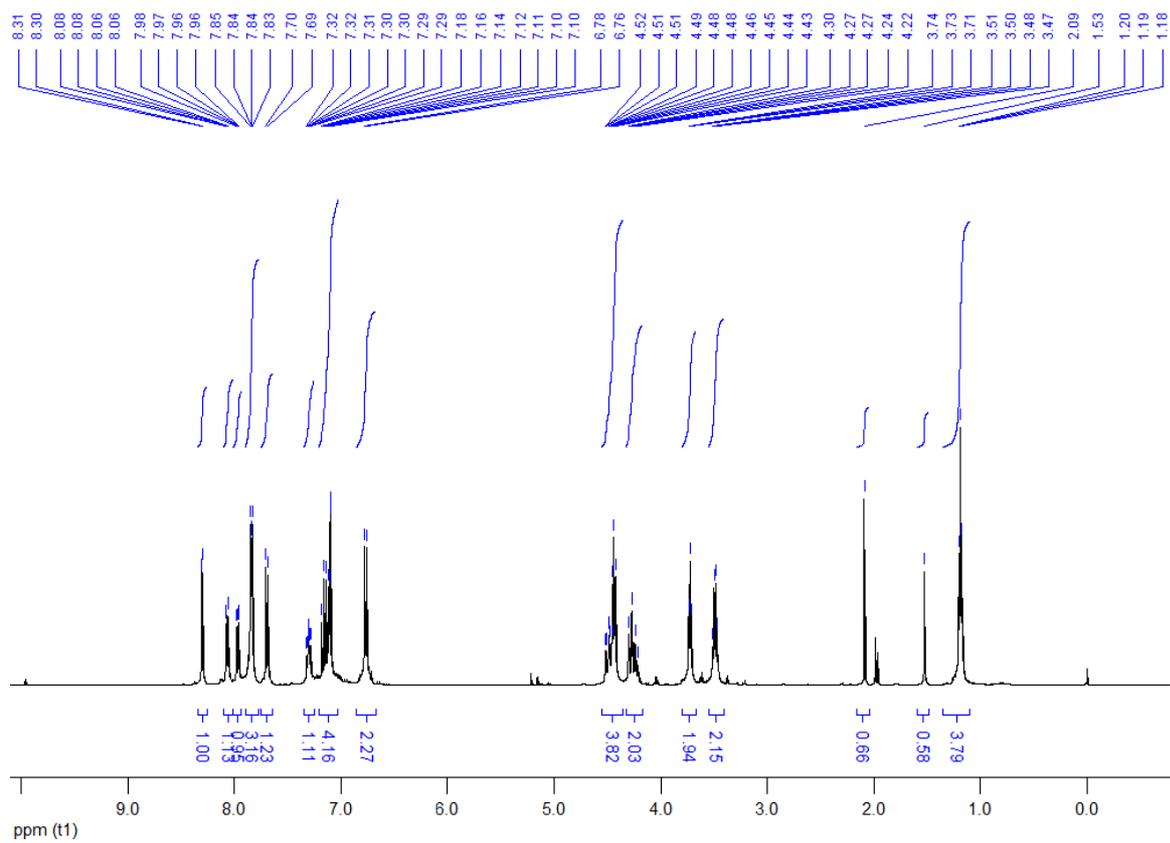




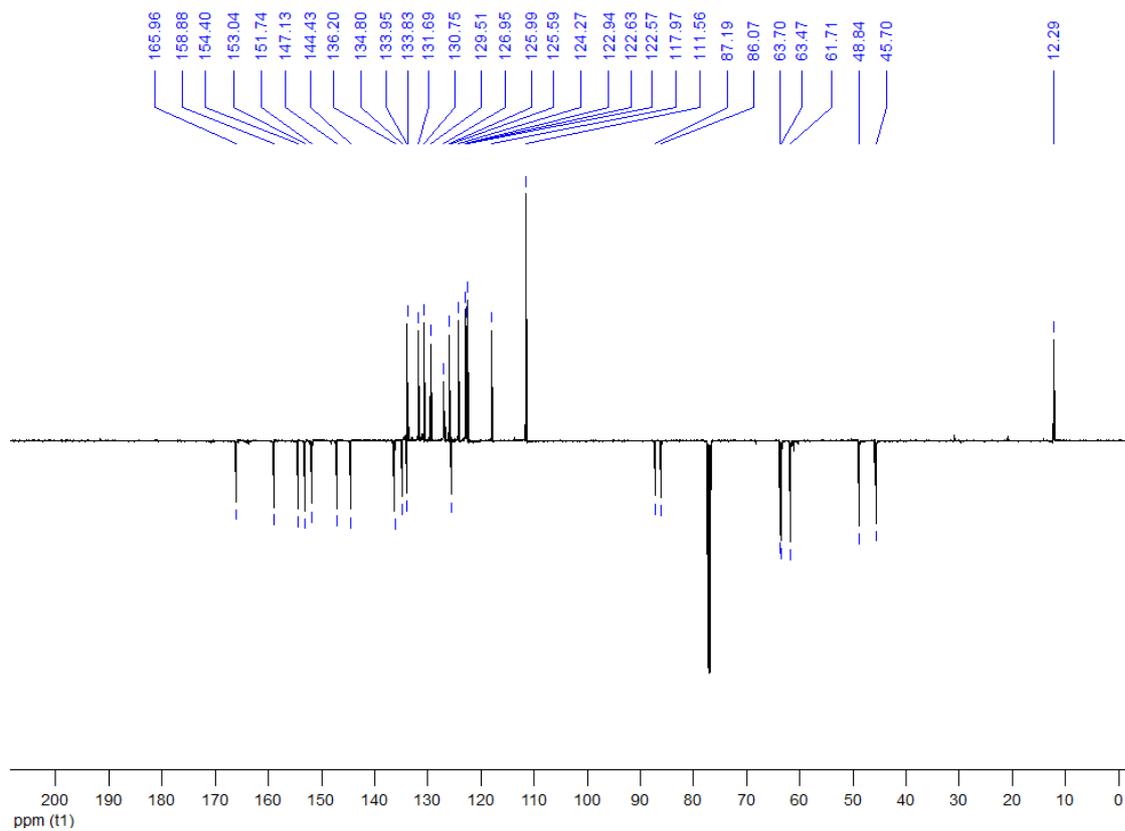
**Ester linked to disperse red 45. AM90.**



$\delta_H$  (500 MHz,  $CDCl_3$ ).



$\delta_C$  (126 MHz, CDCl<sub>3</sub>).



COSY

Chemist Anish Mistry  
AM-90  
COSY.w CDCl<sub>3</sub> /opt/topspin3.2 AM 14



```

Current Data Parameters
NAME      Mar28-2017
EXPNO    11
PROCNO    1

F2 - Acquisition Parameters
Date_     20170328
Time      19:37
INSTRUM   spect
PROBHD    5 mm CPDCl 13C
PULPROG   zgpg30
SOLVENT   CDCl3
NS         1
DS         8
SWH        4795.136 Hz
FIDRES     2.341502 Hz
AQ         0.2130381 sec
RG         27.55
DE         104.265 usec
TE         298.0 K
D0         0.0000300 sec
D1         1.9242700 sec
D11        0.0300000 sec
D12        0.0000000 sec
D13        0.0000400 sec
D16        0.0000000 sec
TD         0.00000860 sec

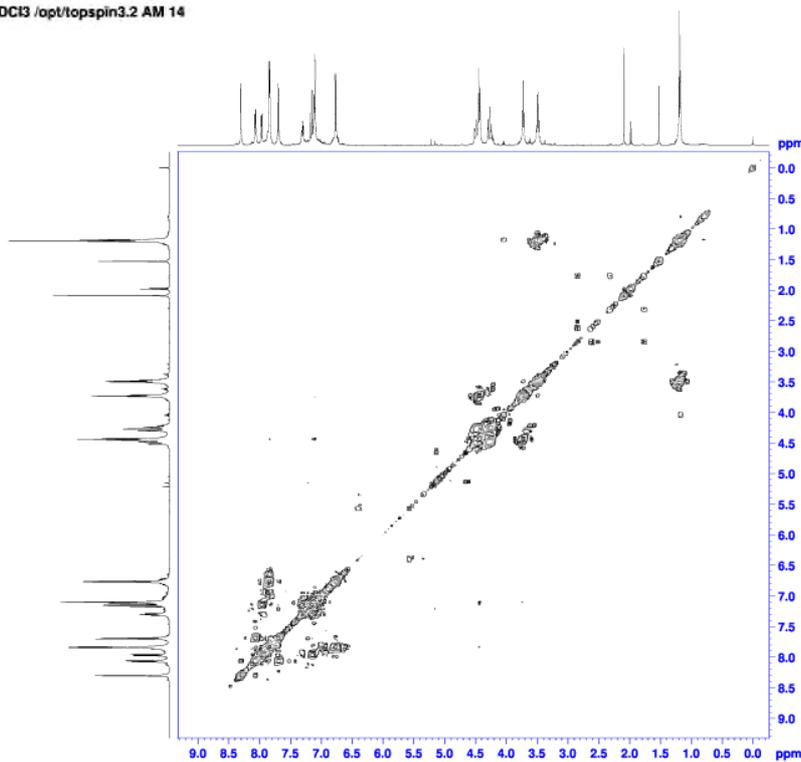
----- CHANNEL f1 -----
NUC1       13C
P1         12.00 usec
PL1        0.00 dB
PL12       13.0000000 W
PL120      1.5924999 W

----- GRADIENT CHANNEL -----
GR1         1000.000 Hz
SP1         10.00 usec
P14         1000.00 usec

F1 - Acquisition parameters
TD          128
SFO1        500.130233 Mhz
FIDRES      31.452061 Hz
SWH         9.849 ppm
F0MDEX     CF

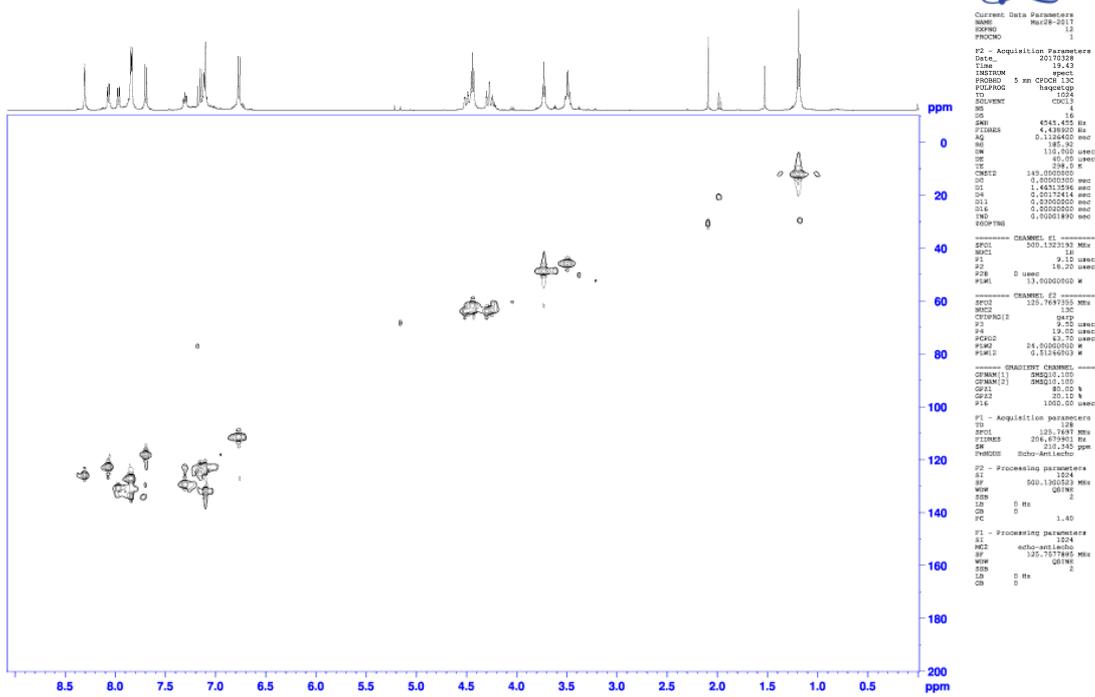
F2 - Processing parameters
SI          1024
SF          500.1300223 Mhz
WDW         0
SSB         0
LB          0 Hz
GB          0
PC          1.40

F1 - Processing parameters
SI          1024
SF          500.1300223 Mhz
WDW         0
SSB         0
LB          0 Hz
GB          0
PC          0
    
```



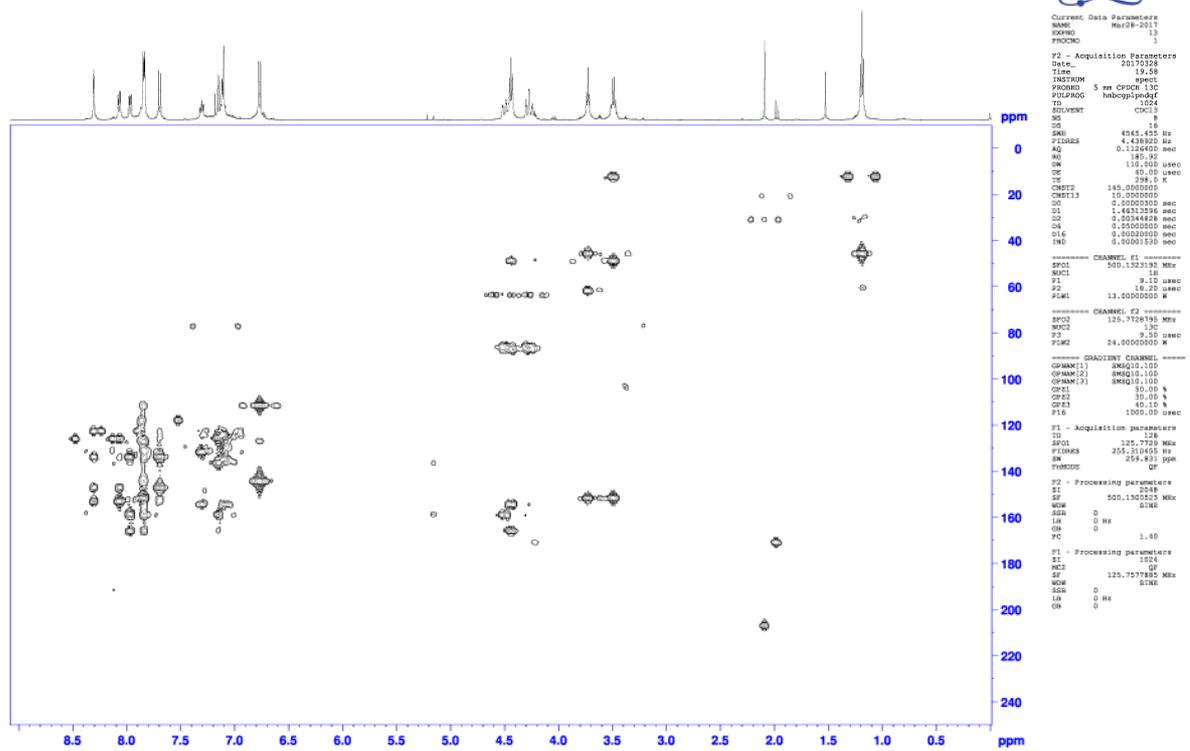
# HSQC

HSQC.w CDCI3 /opt/topspin3.2 AM 14



# HMBC

HMBC.w CDCI3 /opt/topspin3.2 AM 14



### Polystyrene bead functionalisation (AM113,114).

Tris(2-aminoethyl)amine polymer beads (polystyrene based, Aldrich, 3.5-5.0 mmol/g N loading, 1% cross-linked with dibvinylbenzene) (10 mg) and aldehyde **16** (10 mg, 0.034 mmol) were stirred in MeOH (1.5 mL) and NaBH<sub>3</sub>CN (5 mg, 0.131 mmol) was added. The mixture was stirred overnight. At the end of this time the beads were filtered using filter paper and washed with MeOH, then collected and dried. Disperse red azide **22** (7 mg, 0.019 mmol) was dissolved in MeCN (2 mL) and the dried beads were added to this solution. A control reaction was set up containing unfunctionalised beads (5 mg) and **22** (7 mg, 0.019 mmol) in MeCN (2 mL). After stirring for 4 days (left hand picture below) the beads were filtered and washed with acetonitrile until no more colour was washed from the beads (right hand picture below). The beads which had been functionalised with **16** retained the red colour (right hand vial) whereas the colour was washed from the control vial (left) indicating that the bead has become functionalised by **16** and retained the ability to react in ‘click’ cycloadditions with azides. The control reaction indicated that no non-specific interactions were responsible for the retention of the red colour by the beads.

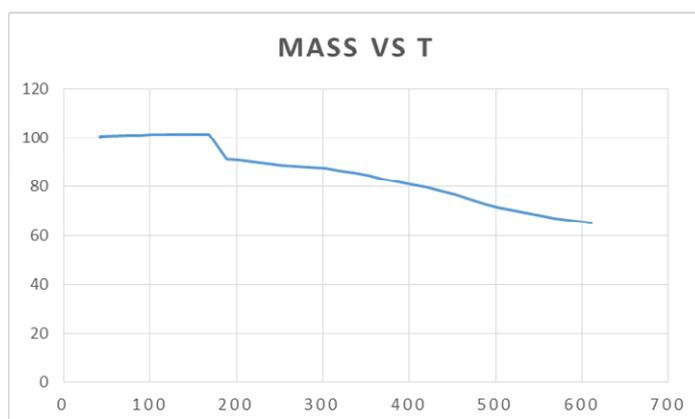


**(above)** Labelled polystyrene beads – before (left) and after (right) washing with MeCN.

### **TGA test results:**

TGA measurements were performed on Mettler-Toledo DSC1 equipped with an autosampler. Samples were heated in 40 ul aluminium pans from 25-600 °C at a rate of 10°C min in a nitrogen atmosphere. Samples were analysed used Mettler-Toledo STARe software.

### **Aldehyde 16:**



### **Methyl ester of acid 5:**

