

Supporting Information

Kinetic Resolution of Terminal Alkyne Substituted Quaternary Oxindoles via Copper Catalysed Azide-Alkyne Cycloadditions

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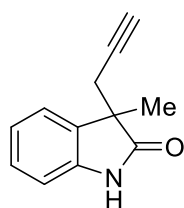
General

Reagents were used as purchased from suppliers without further purification; in cases where anhydrous solvents were required they were dried using a solvent purification system (SPS) which is monitored by Karl-Fisher titrations for water levels. ¹H NMR spectra were recorded at 300 MHz using a Bruker AVIII 300 NMR spectrometer. ¹⁹F NMR spectra were recorded at 282 MHz using a Bruker AVIII 300 NMR spectrometer. ¹³C NMR experiments were carried out on a Bruker AVIII400 NMR spectrometer recorded at 101 MHz; in cases where it was required 2D NMR techniques were used to confirm compound identity. ¹H NMR chemical shifts are reported in ppm relative to TMS (δ 0.00) and ¹³C NMR relative to chloroform (δ 77.36). Reactions carried out at low temperatures were cooled using a Lab Plant Cryoprobe. Melting points were carried out in triplicate and an average of the values taken and reported as a range using Stuart SMP10 melting point apparatus. IR spectra were recorded on a PerkinElmer 100FT-IR spectrometer at room temperature using ATR. Optical rotations were

recorded on a polar 2001 Automatic Polarimeter. Measurements of each sample were recorded three times and used as an average. HPLC analysis was carried out using a Shimadzu LC2010 and Phenomenex Lux cellulose 3 chiral column, traces were recorded at four UV wavelengths 210, 220, 254 and 280nm, calculations were carried out using the supplied traces recorded at 254 nm. Column chromatography was carried out using a Combiflash Rf 200i, column traces were recorded at two UV wavelengths (254 nm and 280nm).

Synthesis and screening

Synthesis of 3-methyl-3-(prop-2-yn-1-yl)indolin-2-one

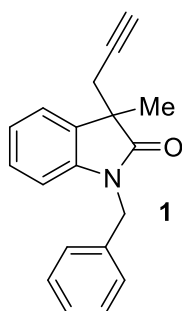


n-Butyl lithium in hexanes (1.5 M, 8.15 mmol, 5.09 mL, 1.2 equiv.) was transferred into a nitrogen flushed flask, THF (40 mL) was added and the solution cooled to -78°C . A solution of 3-methyl-2-oxindole (1.00 g, 6.79 mmol, 1 equiv.) dissolved in THF (10 mL) was added dropwise under stirring. The reaction mixture was stirred for 10 minutes before propargyl bromide (7.13 mmol, 0.76 mL, 1.05 equiv.) was added. The solution was allowed to warm to room temperature and stirred for 3 h. Methanol (20 mL) was added to decompose any remaining butyl lithium. The solution was concentrated *in vacuo* and the residual oil extracted with water (50 mL) and ethyl acetate (3 x 50 mL). The organic phase was dried over MgSO_4 and concentrated *in vacuo*. The oil was then purified using automated column chromatography combiflash Rf (0-25% EtOAc : Hexane gradient 20 mins) to yield the product as a cream solid 0.69 g, 55% yield.

^1H NMR (300 MHz, CDCl_3) δ 7.75 (s, 1H), 7.42 (d, $J = 7.4$ Hz, 1H), 7.29 – 7.21 (m, 2H), 7.07 (td, $J = 7.6, 1.0$ Hz, 1H), 6.91 (d, $J = 7.7$ Hz, 1H), 2.63 (ABqd, $J = 16.6, 2.7$ Hz, 2H),

1.98 (t, $J = 2.7$ Hz, 1H), 1.48 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 182.08, 140.24, 133.47, 128.22, 123.53, 122.54, 109.97, 79.55, 70.85, 47.21, 27.58, 21.93; IR ν_{max} (ATR)/ cm^{-1} 3255, 2981, 2968, 2925, 1705, 1667, 1622, 1471, 1341, 1235, 1191; MS ESI m/z 186.1 $[\text{M}+\text{H}]$ HRMS (ESI-TOF) Calculated for $\text{C}_{12}\text{H}_{11}\text{NONa} = 208.0738$ Found = 208.0739; MP 112-114 $^{\circ}\text{C}$.

Synthesis of 1-benzyl-3-methyl-3-(prop-2-yn-1-yl)indolin-2-one (**1**)



Sodium hydride (2.38 mmol, 0.082 g, 2.2 equiv) was suspended in THF (10 mL). The reaction was cooled to 0 $^{\circ}\text{C}$ using an ice bath, at this temperature a solution of 3-methyl-3-(prop-2-yn-1-yl)indolin-2-one (1.08 mmol, 0.20 g, 1 equiv) dissolved in THF (10 mL) was added dropwise. When the formation of gas had ceased, benzyl bromide (1.08 mmol, 0.185 g, 0.129 mL, 1 equiv)

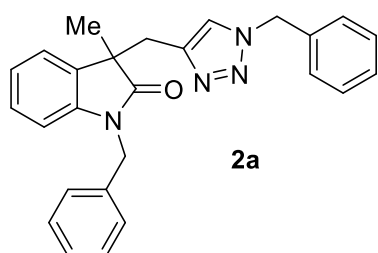
was added to the mixture. The reaction was allowed to warm to room temperature and left to stir for 2 h. Water (5 mL) was added to decompose any remaining sodium hydride and the solution was extracted with water (50 mL) and diethyl ether (3 x 50 mL). The organic phase was dried over magnesium sulphate and concentrated *in vacuo*. To yield the product as a colourless crystalline solid this was then washed with hexane (50 mL) to remove any residual benzyl bromide. Colourless crystalline solid 0.23g, 77% yield.

^1H NMR (300 MHz, CDCl_3) δ 7.43 (dd, $J = 7.4, 0.8$ Hz, 1H), 7.36 – 7.28 (m, 5H), 7.18 (td, $J = 7.7, 1.3$ Hz, 1H), 7.05 (td, $J = 7.6, 1.0$ Hz, 1H), 6.73 (d, $J = 7.8$ Hz, 1H), 4.96 (ABq, $J = 15.7$ Hz, 2H), 2.72 (ABqd, $J = 16.5, 2.7$ Hz, 2H), 1.92 (t, $J = 2.6$ Hz, 1H), 1.51 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 179.46, 142.16, 135.84, 132.96, 128.70, 128.14, 127.59, 127.32, 123.21, 122.60, 109.12, 79.76, 70.77, 46.77, 43.76, 27.76, 22.36; IR ν_{max} (ATR)/ cm^{-1} 3285, 2924, 1711, 1608, 1489, 1466, 1426, 1378, 1179; MS ESI m/z 276.1 $[\text{M}+\text{H}]$ HRMS (ESI-TOF) Calculated for $\text{C}_{19}\text{H}_{18}\text{NO} = 276.1388$ Found = 276.1378; MP 140-141 $^{\circ}\text{C}$; HPLC

(Cellulose 3) acetonitrile/water 50:50, 1.0 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 7.5$ min, $t_{\text{minor}} = 8.5$ min; $[\alpha]_D^{293} t_{\text{minor}} = -41$ ($c = 1.0$, CHCl_3)°

Enantiopure material was recovered by preparative HPLC using cellulose 1 acetonitrile/water 50:50 15 mL/min, $\lambda = 254$ nm. The enantiopure material was subjected to optical rotation analysis and from this it was calculated that the (–) enantiomer was eluting as the t_{minor} peak in the cellulose 3 analytical HPLC. Therefore the recovered enantioenriched alkyne from the kinetic resolution should have a positive optical rotation.

Synthesis of 1-benzyl-3-((1-benzyl-1H-1,2,3-triazol-4-yl)methyl)-3-methylindolin-2-one (2a)

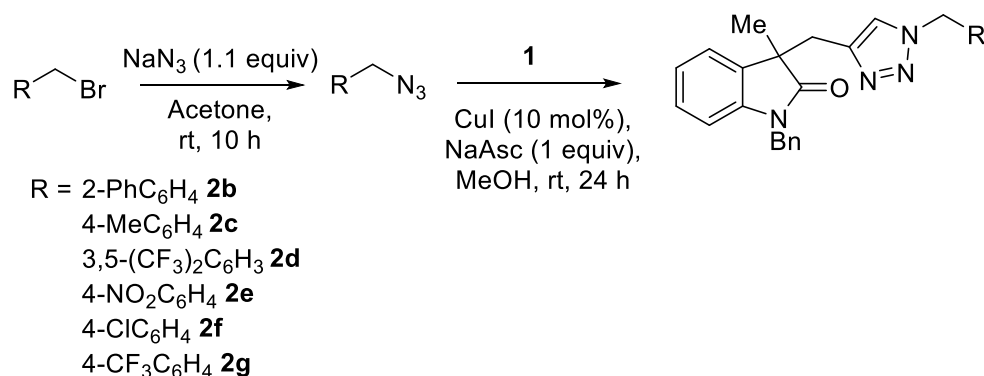


To a solution of **1** (0.24 mmol, 0.066 g, 1 equiv) in methanol (10 mL) was added copper (I) iodide (0.0046 g, 0.024 mmol, 10 mol%), sodium ascorbate (0.24 mmol, 0.048g, 1 equiv) and finally benzyl azide (0.24 mmol, 0.032 g, 1 equiv) this was allowed to stir overnight. The reaction was quenched with aqueous ammonia 5% v/v (10 mL) and extracted with diethyl ether (3 x 25 mL) then washed with water (100 mL). The remaining starting material and the triazolic product were isolated by combiflash chromatography. Rf petroleum ether: diethyl ether 0-100% gradient followed by EtOAc 100%. To yield the product as brown oil 0.070 g, 71% yield.

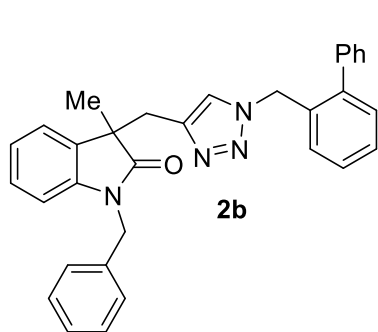
^1H NMR (300 MHz, CDCl_3) δ 7.35 – 6.93 (m, 13 H), 6.73 (s, 1 H), 6.57 (d, $J = 7.6$ Hz, 1H), 5.28 (s, 2 H), 4.70 (s, 2 H), 3.81 (ABq $J = 14.3$, 2H) 1.54 (s, 3 H); ^{13}C NMR (101 MHz, CDCl_3) δ 179.88, 143.35, 142.11, 135.94, 134.87, 132.91, 128.95, 128.71, 128.44, 127.85, 127.69, 127.52, 127.28, 123.25, 122.58, 121.84, 108.82, 53.71, 48.58, 43.48, 34.39, 23.46; IR ν_{max} (ATR)/ cm^{-1} 2924, 1708, 1611, 1489, 1468, 1454, 1355, 1176; MS ESI m/z 431.3 [M+Na] HRMS (ESI-TOF) Calculated for $\text{C}_{26}\text{H}_{24}\text{N}_4\text{ONa} = 431.1848$ Found = 431.1848;

HPLC (Cellulose 3) acetonitrile/water 40:60, 1.0 mL/min, $\lambda = 254$ nm, $t_{\text{major}} = 11.2$ min, $t_{\text{minor}} = 12.7$ min.

General procedure for synthesis of racemic oxindole triazoles via *in situ* azide formation



The alkyl bromide (0.12 mmol, 1 equiv) was stirred at rt with sodium azide (0.13 mmol, 0.0085 g, 1.1 equiv) in acetone (5 mL). After 10 hours the reaction was diluted with methanol (10 mL) and **1** (0.12 mmol, 0.033 g, 1 equiv) added along with copper (I) iodide (2.3 mg, 0.012 mmol, 0.1 equiv, 10 mol%) and sodium ascorbate (0.12 mmol, 0.024 g, 1 equiv) the reaction was allowed to stir at rt for 24 h. The reaction was quenched with aqueous ammonia 5% v/v (10 mL) and extracted with ether (3 x 25 mL). The triazolic product was isolated by combiflash Rf petroleum ether: diethyl ether 0-100% gradient followed by EtOAc 100%.

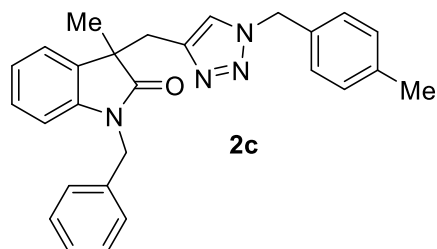


(**2b**) Brown oil 0.030 g, 52% yield ^1H NMR (300 MHz, CDCl_3) δ 7.46 – 6.97 (m, 16H), 6.75 (d, $J = 7.6$ Hz, 1H), 6.64 – 6.53 (m, 2H), 5.22 (s, 2H), 4.68 (q, $J = 15.6$ Hz, 2H), 3.26 (ABq, $J = 14.3$ Hz, 2H), 1.50 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 179.90, 143.04, 142.10, 141.68, 139.74,

135.89, 132.93, 132.27, 130.30, 128.99, 128.66, 128.54, 128.46, 128.30, 128.05, 127.85, 127.67, 127.49, 127.22, 123.31, 122.58, 121.95, 108.83, 51.35, 48.56, 43.48, 34.27, 23.57; IR

ν_{\max} (ATR)/ cm^{-1} 2925, 2854, 1707, 1611, 1488, 1467, 1453; MS ESI m/z 507.2 [M+Na]

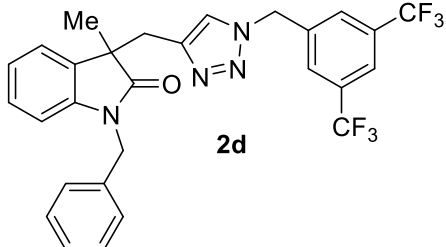
HRMS (ESI-TOF) Calculated for $\text{C}_{32}\text{H}_{28}\text{N}_4\text{ONa}$ = 507.2161 Found = 507.2166.



2c

(2c) Brown oil 0.038 g, 75% yield ^1H NMR (300 MHz, CDCl_3) δ 7.28 – 6.93 (m, 10H), 6.85 (d, J = 8.0, 2H), 6.70 (s, 1H), 6.55 (d, J = 7.6, 1H), 5.21 (s, 2H), 4.68 (s, 2H), 3.26 (Abq, J = 14.3, 2H), 2.34 (s, 3H), 1.51 (s,

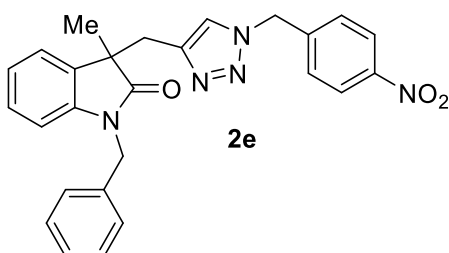
3H); ^{13}C NMR (101 MHz, CDCl_3) δ 179.89, 143.23, 142.11, 138.30, 135.95, 132.94, 131.84, 129.60, 128.70, 127.82, 127.77, 127.51, 127.23, 123.26, 122.57, 121.76, 108.83, 53.53, 48.57, 43.48, 34.39, 23.43, 21.16; IR ν_{\max} (ATR)/ cm^{-1} 2925, 1707, 1611, 1489, 1467, 1453, 1379, 1354, 1175; MS ESI m/z 445.2 [M+Na] HRMS (ESI-TOF) Calculated for $\text{C}_{27}\text{H}_{26}\text{N}_4\text{ONa}$ = 445.2004 Found = 445.2006.



2d

(2d) Brown oil 0.035g, 53% yield ^1H NMR (300 MHz, CDCl_3) δ 7.83 (s, 1H), 7.46 (s, 2H), 7.31 – 6.93 (m, 9H), 6.74 (s, 1H), 6.60 (d, J = 7.5 Hz, 1H), 5.32 (s, 2H), 4.76 (Abq, J = 15.5 Hz, 1H), 3.32 (Abq, J = 14.4 Hz, 2H),

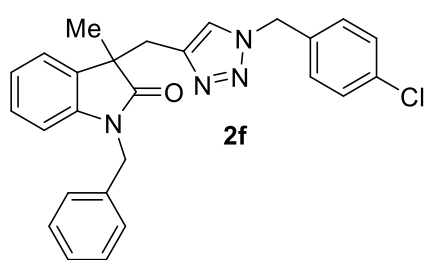
1.52 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 179.77, 143.92, 142.06, 137.28, 135.97, 132.81, 132.61, 132.27, 128.68, 128.00, 127.88, 127.53, 127.43, 123.17, 122.66, 122.00, 108.77, 52.51, 48.37, 43.51, 34.22, 23.62; ^{19}F NMR (282 MHz, CDCl_3) δ -62.83; IR ν_{\max} (ATR)/ cm^{-1} 2928, 1706, 1612, 1489, 1468, 1454, 1382, 1354, 1277, 1173, 1132; MS ESI m/z 567.2 [M+Na] HRMS (ESI-TOF) Calculated for $\text{C}_{28}\text{H}_{22}\text{N}_4\text{OF}_6\text{Na}$ = 567.1596 Found = 567.1593.



2e

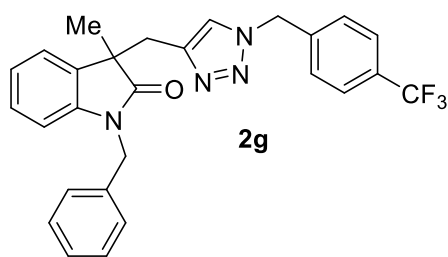
(2e) Colourless solid 0.035g 64% yield ^1H NMR (300 MHz, CDCl_3) δ 8.15 – 7.96 (m, 2H), 7.26 – 6.88 (m,

10H), 6.75 (s, 1H), 6.65 (d, $J = 7.7$, 1H), 5.35 (d, $J = 6.3$, 2H), 4.79 (Abq, $J = 15.5$, 2H), 3.36 (Abq, $J = 14.4$, 2H), 1.55 (3 H, s); ^{13}C NMR (101 MHz, CDCl_3) δ 179.74, 143.90, 142.19, 141.85, 135.94, 132.90, 128.69, 127.96, 127.90, 127.55, 124.06, 123.22, 122.67, 122.15, 108.77, 52.60, 48.56, 43.70, 34.18, 23.81; IR ν_{max} (ATR)/ cm^{-1} 2925, 1707, 1611, 1521, 1489, 1467, 1453, 1379, 1346, 1176; MS ESI m/z 476.2 $[\text{M}+\text{Na}]$ HRMS (ESI-TOF) Calculated for $\text{C}_{26}\text{H}_{23}\text{N}_5\text{O}_3\text{Na} = 476.1699$ Found = 476.1698.



(2f) Brown oil 0.043 g, 82% yield ^1H NMR (300 MHz, CDCl_3) δ 7.27 – 6.94 (m, 10H), 6.78 (t, $J = 9.7$ Hz, 2H), 6.65 (s, 1H), 6.56 (d, $J = 7.7$ Hz, 1H), 5.27 – 5.12 (m, 2H), 4.70 (Abq, $J = 15.6$ Hz, 2H), 3.29 (Abq, $J = 14.3$ Hz, 2H)

1.52 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 179.80, 143.54, 142.13, 135.92, 134.41, 133.32, 132.89, 129.11, 128.97, 128.71, 127.86, 127.55, 127.37, 123.20, 122.60, 121.79, 108.81, 52.93, 48.57, 43.56, 34.34, 23.57; IR ν_{max} (ATR)/ cm^{-1} 2925, 1707, 1611, 1490, 1467, 1454, 1380, 1355, 1174; MS ESI m/z 465.2 $[\text{M}+\text{Na}]$ HRMS (ESI-TOF) Calculated for $\text{C}_{27}\text{H}_{23}\text{N}_4\text{OF}_3\text{Na} = 465.1458$ Found = 465.1453.

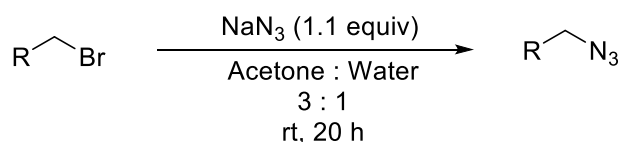


(2g) Brown oil 0.036 g, 63% yield ^1H NMR (300 MHz, CDCl_3) δ 7.49 (d, $J = 8.1$ Hz, 2H), 7.28 – 6.89 (m, 10H), 6.67 (s, 1H), 6.57 (d, $J = 7.6$ Hz, 1H), 5.36 – 5.21 (m, 2H), 4.72 (Abq, $J = 15.5$ Hz, 2H), 3.31 (Abq, $J = 14.3$ Hz, 2H), 1.53 (s, 3H); ^{13}C NMR (101 MHz, CDCl_3) δ 179.77, 143.68, 142.16, 138.75, 135.92, 132.88, 130.47, 128.70, 127.88, 127.73, 127.56, 127.42, 125.88, 125.85, 123.19, 122.60, 121.96, 108.77, 52.97, 48.56, 43.61, 34.28, 23.67; ^{19}F NMR (282 MHz, CDCl_3) δ -62.69; IR ν_{max} (ATR)/ cm^{-1} 2925, 1709, 1612, 1490, 1468, 1381, 1326, 1169, 1124;

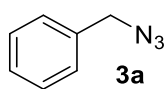
^{19}F NMR (282 MHz, CDCl_3) δ -62.69; IR ν_{max} (ATR)/ cm^{-1} 2925, 1709, 1612, 1490, 1468, 1381, 1326, 1169, 1124;

MS ESI m/z 499.2 [M+Na] HRMS [M+Na] Calculated for $C_{27}H_{23}N_4OF_3Na$ = 499.1722
Found = 499.1721.

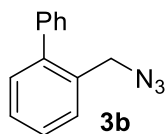
General procedure for synthesis and isolation of azides



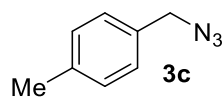
Sodium azide (1.20 g, 18.5 mmol, 1.1 equiv) was added to a mixture of acetone : water (3:1), to this benzyl bromide was added (2.88 g, 16.8 mmol, 2 mL, 1 equiv) and the reaction mixture was stirred at rt for 20 h. Water (100 mL) was added and the reaction mixture was extracted with diethyl ether (3 x 25 mL). The combined organic extracts were combined and removed *in vacuo* to yield benzyl azide **3a** as a colourless oil 1.79 g 80% yield



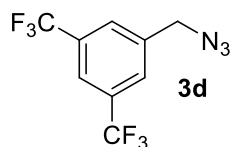
Characterisation was in agreement with the reported literature values.¹ ^1H NMR (300 MHz, CDCl_3) δ 7.35 – 7.18 (m, 5H), 4.18 (s, 2H); ^{13}C NMR (101 MHz, CDCl_3) δ 135.65, 128.98, 128.43, 128.38, 54.83; MS EI m/z 133.1 [M], 104 [M-N₂], 91.1 [M-N₃], 77.0 [M-CH₂N₃].



Prepared from 2-phenylbenzyl bromide (0.50 g, 2.39 mmol, 1 equiv) and sodium azide (0.17 g, 2.63 mmol, 1.1 equiv) according to the general procedure. Isolated as a yellow oil 0.42 g, 85% yield. Characterisation was consistent with the literature.² ^1H NMR (300 MHz, CDCl_3) δ 7.44 – 7.25 (m, 9H), 4.24 (s, 2H); ^{13}C NMR (101 MHz, CDCl_3) δ 142.33, 140.37, 132.92, 130.56, 129.69, 129.31, 128.45, 127.91, 127.57, 52.68.

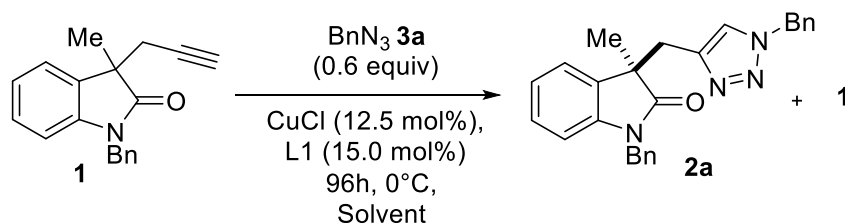


Prepared from 4-methylbenzyl bromide (0.5 g, 2.70 mmol, 1 equiv) and sodium azide (0.19 g, 3.00 mmol, 1.1 equiv) according to the general procedure. Isolated as an orange oil 0.40 g, 64% yield. Characterisation was consistent with the literature.³ ¹H NMR (300 MHz, CDCl₃) δ 7.21 – 7.13 (m, 4H), 4.25 (s, 2H), 2.33 (s, 3H); ¹³C NMR (101 MHz, CDCl₃) δ 138.18, 132.39, 129.56, 128.33, 54.65, 21.20. MS EI m/z 147.1 [M], 118.1 [M-N₂], 105.1 [M-N₃], 91.1 [M-CH₂N₃].



Prepared from 3,5-bis(trifluoromethyl)benzyl bromide (0.20 g, 0.65 mmol, 1 equiv) and sodium azide (0.05 g, 0.72 mmol, 1.1 equiv) according to the general procedure. Isolated as a yellow oil 0.096 g, 55% yield. Characterisation was consistent with the literature.³ ¹H NMR (300 MHz, CDCl₃) δ 7.86 (s, 1H), 7.79 (s, 2H), 4.56 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 138.30, 132.63, 132.29, 131.96, 131.63, 127.86, 127.83, 127.13, 124.42, 122.06, 122.03, 121.99, 121.71, 53.44.

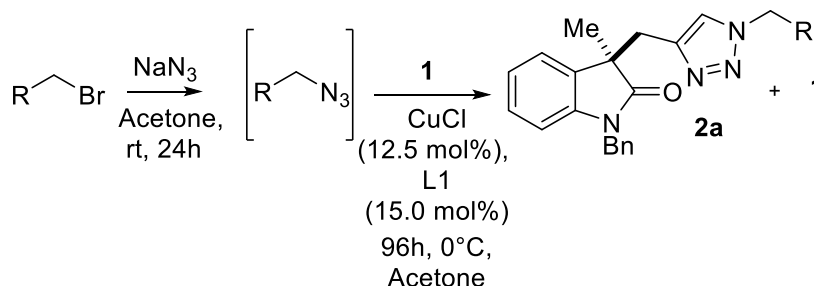
General procedure for oxindole kinetic resolution screening



To an oven dried Radleys multi reactor tube, under an atmosphere of nitrogen, were added **L1** (0.0067 g, 0.018 mmol, 15.0 mol%) and CuCl (0.0015 g, 0.015 mmol, 12.5 mol%) followed by 2,5-hexanedione (1 mL). After the solution was stirred at rt for 1 h, compound **1** (0.033 g, 0.12 mmol, 1 equiv) dissolved in 2,5-hexanedione (1 mL) was added. The reaction mixture was stirred for a further 15 mins at rt then cooled to 0 °C for another 15 mins, benzyl azide (0.0095 g, 0.07 mmol, 0.6 equiv) was then added. The resulting mixture was maintained at 0 °C for 96 h with stirring. The reaction was quenched with aqueous ammonia 5% v/v (10 mL) and extracted with diethyl ether (2 x 25mL) dried over MgSO₄ and

concentrated *in vacuo*. A crude ^1H NMR spectrum was taken to determine conversion. The remaining starting material and the triazolic product were then isolated by automated column chromatography combiflash Rf petroleum ether: diethyl ether 0-100% gradient followed by EtOAc 100%. Conversion was measured by ^1H NMR and enantiomeric excess via chiral HPLC.

Representative procedure for oxindole kinetic resolution screening using *in situ* azide formation



4-Nitrobenzyl bromide (0.015 g, 0.07 mmol, 0.6 equiv) was stirred at rt with sodium azide (0.0052 g, 0.08 mmol, 1.1 equiv) in acetone (5 mL) for 10 h. In a separate reaction vessel a solution of **L1** (0.0067 g, 0.018 mmol, 15.0 mol%) and CuCl (0.0015 g, 0.015 mmol, 12.5 mol%) in acetone (1mL) was stirred for 1 h at rt. To this **1** (0.033 g, 0.12 mmol, 1 equiv) in acetone (1 mL) was added. The reaction was then cooled to 0°C and stirred for 30 minutes the azide formation solution was then transferred by syringe this was then maintained at 0°C for 96 h. The reaction was quenched with aqueous ammonia 5% v/v (10 mL) and extracted with diethyl ether (2 x 25 mL). The remaining starting material and the triazolic product were isolated by combiflash Rf petroleum ether: diethyl ether 0-100% gradient followed by EtOAc 100% and enantiomeric excess determined by chiral HPLC.

In situ azide screening table

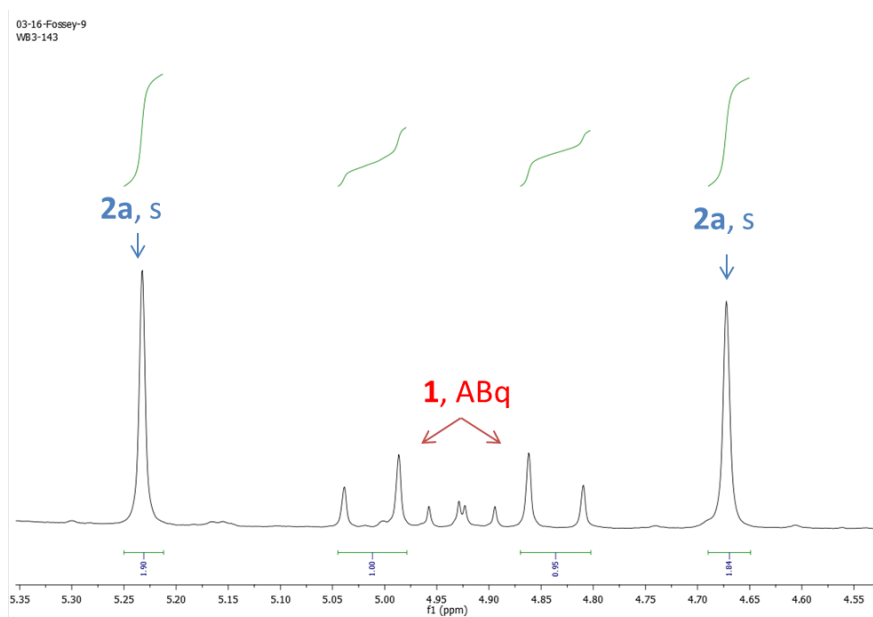
Entry	R	Conv (%) ^a	ee SM (%) ^b	Selectivity Factor (S) ^c
1	2b 2-PhC ₆ H ₄	13	14	35.2

2	2c 4-MeC ₆ H ₄	12	8	4.2
3	2d 3,5-CF ₃ C ₆ H ₃	12	7	3.3
4	2e 4-NO ₂ C ₆ H ₄	22	6	1.6
5	2f 4-ClC ₆ H ₄	12	6	2.7
6	2g 4-CF ₃ C ₆ H ₄	6	0	1

^a Conversion determined by inspection of ¹H NMR spectra (see ESI); ^b E.e. of recovered starting material (HPLC); ^c $S = \ln[(1-c)(1-ee)]/\ln[(1-c)(1+ee)]$.

Representative determination of conversion via ¹H NMR Spectroscopy

Conversion of alkyne **1** to triazole **2a** was determined by direct comparison of the integrations of a series of peaks in the crude ¹H NMR spectra of the resolution reaction mixture. Due to the high boiling point of 2,5-hexanedione it was only possible to remove this *via* column chromatography therefore HPLC was not an appropriate manner for conversion analysis. The signals in the benzylic region were used as this was a clear area away from any interference from remaining solvent. The ABq centred at 4.96 of compound **1** was directly compared with the two singlets at 5.28 and 4.70 of compound **2a**. A representative example is shown in Figure 1. When the azide was varied the analogous signals in the triazolic product were used.



$$\text{Integration per proton in } \mathbf{1} = \frac{1.00+0.95}{2} = 0.98 \quad \text{Integration per proton in } \mathbf{2a} = \frac{1.90+1.84}{4} = 0.94$$

$$\% \text{ Conversion} = \frac{0.94}{0.94+0.98} \times 100 = 49\%$$

Figure 1 Crude ^1H NMR showing direct comparison of signals related to product and starting material for calculation of conversion

Screening Tables

Solvents

Entry	Solvent	Conv (%) ^a	ee SM (%) ^b	Selectivity Factor (<i>S</i>) ^c
1	2,5-Hexanedione	46	72	23.2 ^d
2	Acetone	34	35	5.3
3	Acetonitrile	14	1	1.1
4	DMF	0	-	-
5	DMSO	44	18	1.9
6	1,4-Dioxane	17	6	1.9
7	^t BuOH	19	0	1.0
8	^t BuOH/H ₂ O	13	0	1.0
9	H ₂ O	6	-	-
10	Toluene	5	-	-
11	2-Butanone	0	-	-
12	THF	37	46	8.7
13	1:10 Acetone:2,5-Hexanedione	61	56	3.5
14	NMP	4	-	-
15	Cyclohexanone	0	-	-
16	2,3-Butandione	34	18	1.1
17	Furan	3	1	2.0
18	^t BuOH:2,5 Hexanedione 100:1, 25°C, 7 days	63	28	1.8
19	THF:2,5 Hexanedione 100:1	23	12	2.6
20	THF:2,5 Hexanedione 1:1	30	15	2.4
21	^t BuOH	34	7	1.4

^a Conversion determined by inspection of ¹H NMR spectra (see ESI); ^b E.e. of recovered starting material (HPLC); ^c $S = \ln[(1-c)(1-ee)]/\ln[(1-c)(1+ee)]$; ^d Average of three $S = 22.1 \pm 0.5$, best unique case $S = 23.2$.

Copper Sources

Entry	Copper Source	Conv (%) ^a	ee SM (%) ^b	Selectivity Factor (S) ^c
1	CuCl	34	35	5.3
2	CuBr	14	8	3.2
3	CuI	51	26	2.1
4	CuSO ₄ , NaAsc	13	1	1.2
5	Cu(OTf) ₂	0	0	-
6	Cu(OTf).0.5Toluene	10	3	1.8
7	Cu(OAc)	0	0	-
8	Cu Metal	0	0	-
9	Cu(OAc) ₂	8	4	2.8

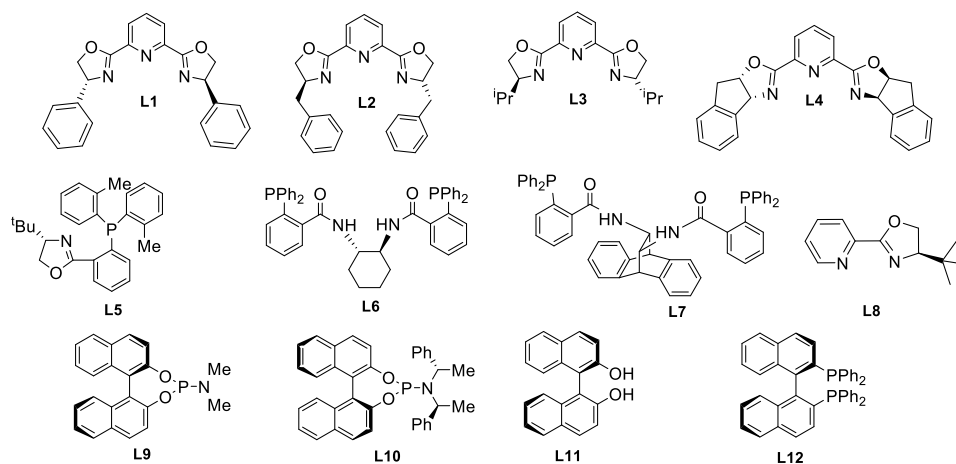
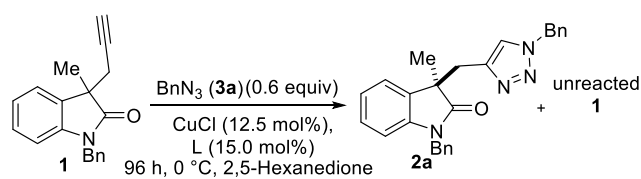
^a Conversion determined by inspection of ¹H NMR spectra (see ESI); ^b E.e. of recovered starting material (HPLC); ^c $S = \ln[(1-c)(1-ee)]/\ln[(1-c)(1+ee)]$.

Temperature and additives

Entry	Solvent	Additive	Copper Source	Temp (°C)	Reaction Time (h)	Conv (%) ^a	ee SM (%) ^b	S ^c
1	THF		CuCl	50	24	45	8	1.3
2	Acetone		CuCl	50	24	46	24	2.3
3	tBuOH		CuCl	50	24	7	0	1.0
4	Acetone		Cu(OTf).0.5Toluene	50	24	51	11	1.4
5	Acetone		Cu(OAc) ₂	50	24	61	20	1.5
6	Acetone	NaAsc	Cu(OTf).0.5Toluene	50	24	50	1	1.0
7	Acetone	NaAsc	Cu(OAc) ₂	50	24	53	16	1.5
8	DMSO		CuCl	50	24	38	4	1.2
9	DMF		CuCl	50	24	40	8	1.4
10	2,5-Hexanedione	NaAsc	CuCl	0	96	52	21	1.8
11	THF	NaAsc	CuCl	0	96	16	1	1.1
12	THF	DIPEA	CuCl	0	96	28	27	7.2
13	Acetone	DIPEA	CuCl	0	96	20	16	5.3

^a Conversion determined by inspection of ¹H NMR spectra (see ESI); ^b E.e. of recovered starting material (HPLC); ^c $S = \ln[(1-c)(1-ee)]/\ln[(1-c)(1+ee)]$.

Ligands



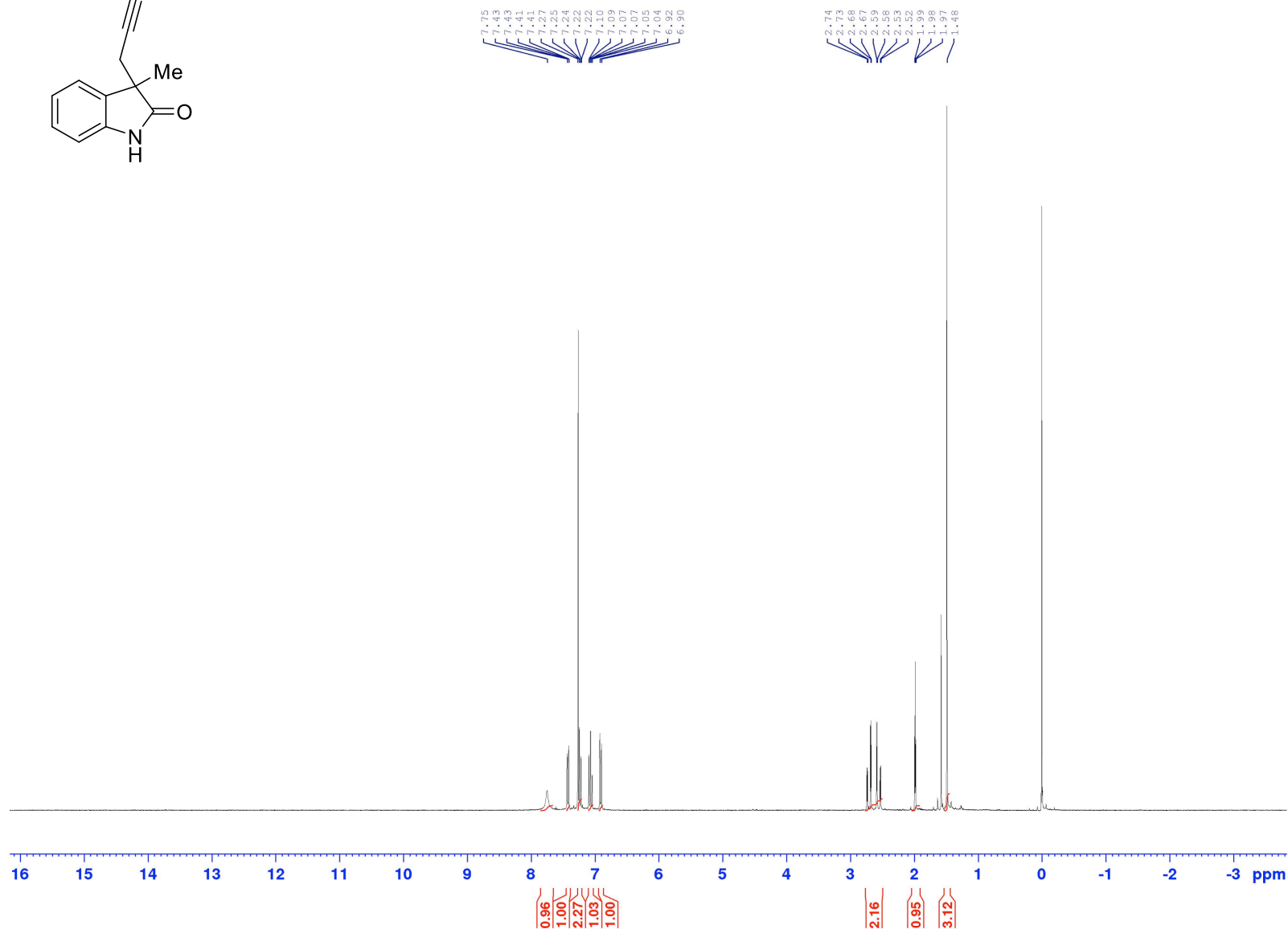
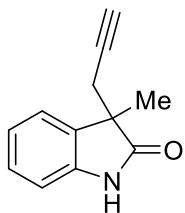
Entry	Ligand	Conv (%) ^a	ee SM (%) ^b	Selectivity Factor (<i>S</i>) ^c
1	L1	46	72	23.2
2	L2	6	1	1.4
3	L3	0	-	-
4	L4	0	-	-
5	L5	0	-	-
6	L6	0	-	-
7	L7	0	-	-
8	L8	5	1	1.5
9	L9	0	-	-
10	L10	0	-	-
11	L11	4	0	1.0
12	L12	0	-	-

^a Conversion determined by inspection of ^1H NMR spectra (see ESI); ^b E.e. of recovered starting material (HPLC); ^c $S = \ln[(1-c)(1-ee)]/\ln[(1-c)(1+ee)]$.

NMR Data

¹H NMR 3-methyl-3-(prop-2-yn-1-yl)indolin-2-one

WB-NH-SM



Current Data Parameters
NAME 01-19-Fossey-13
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150119
Time 11.06
INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 645
DW 83.200 use
DE 12.89 use
TE 292.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
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NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
SI 32768
SF 300.1300068 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR 3-methyl-3-(prop-2-yn-1-yl)indolin-2-one

WBNH



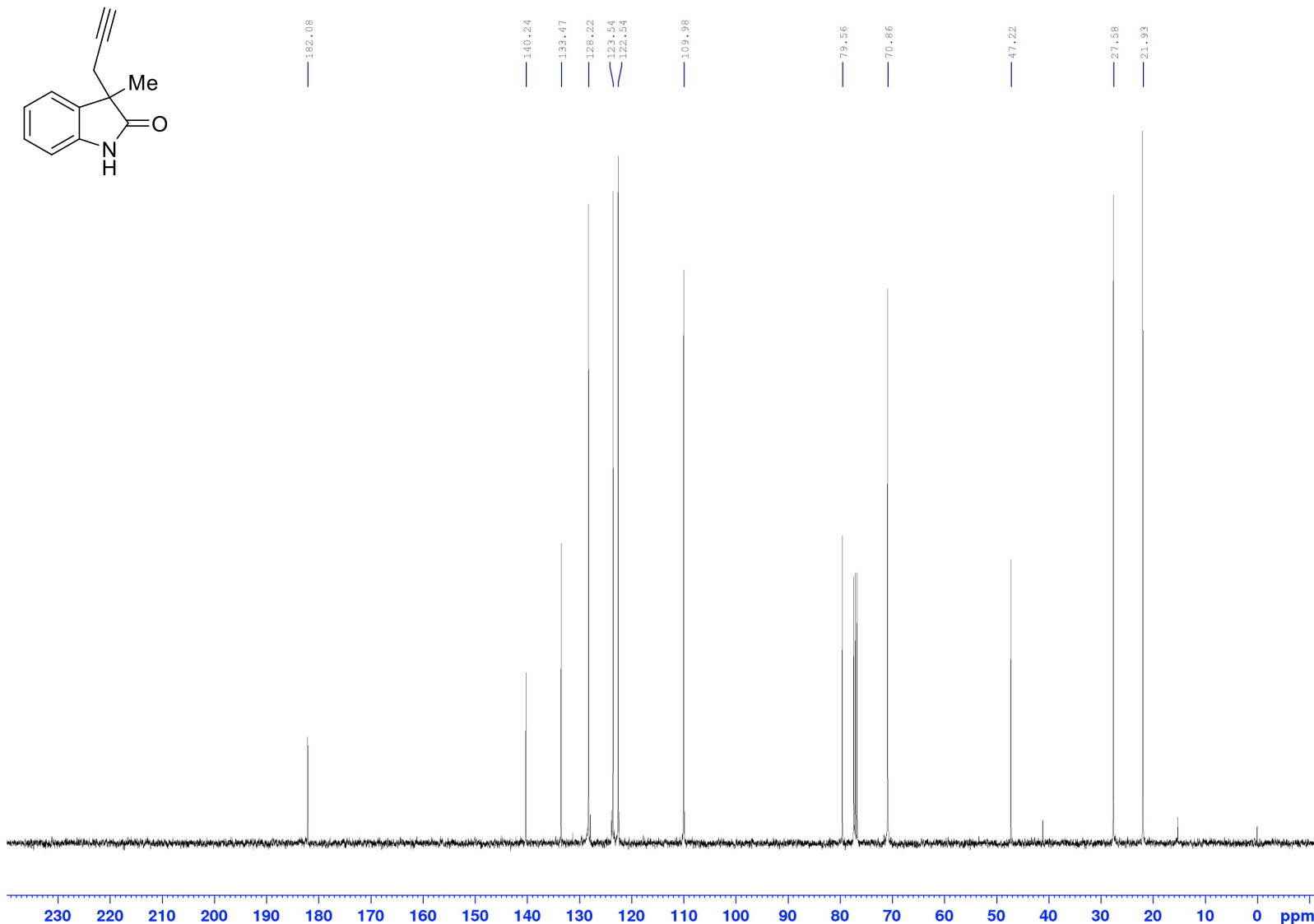
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EXPNO 10
PROCNO 1

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INSTRUM spect
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PULPROG udeft
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.1 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

===== CHANNEL f1 =====
SFO1 100.6242690 MHz
NUC1 13C
P1 8.80 use
P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

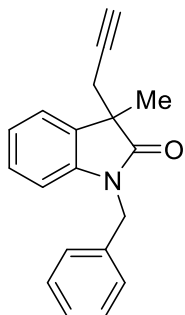
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CPDPRG[2] waltz16
PCPD2 90.00 use
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PLW12 0.28218001 W

F2 - Processing parameters
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WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



¹H NMR (1)

WB2-86 0mol%

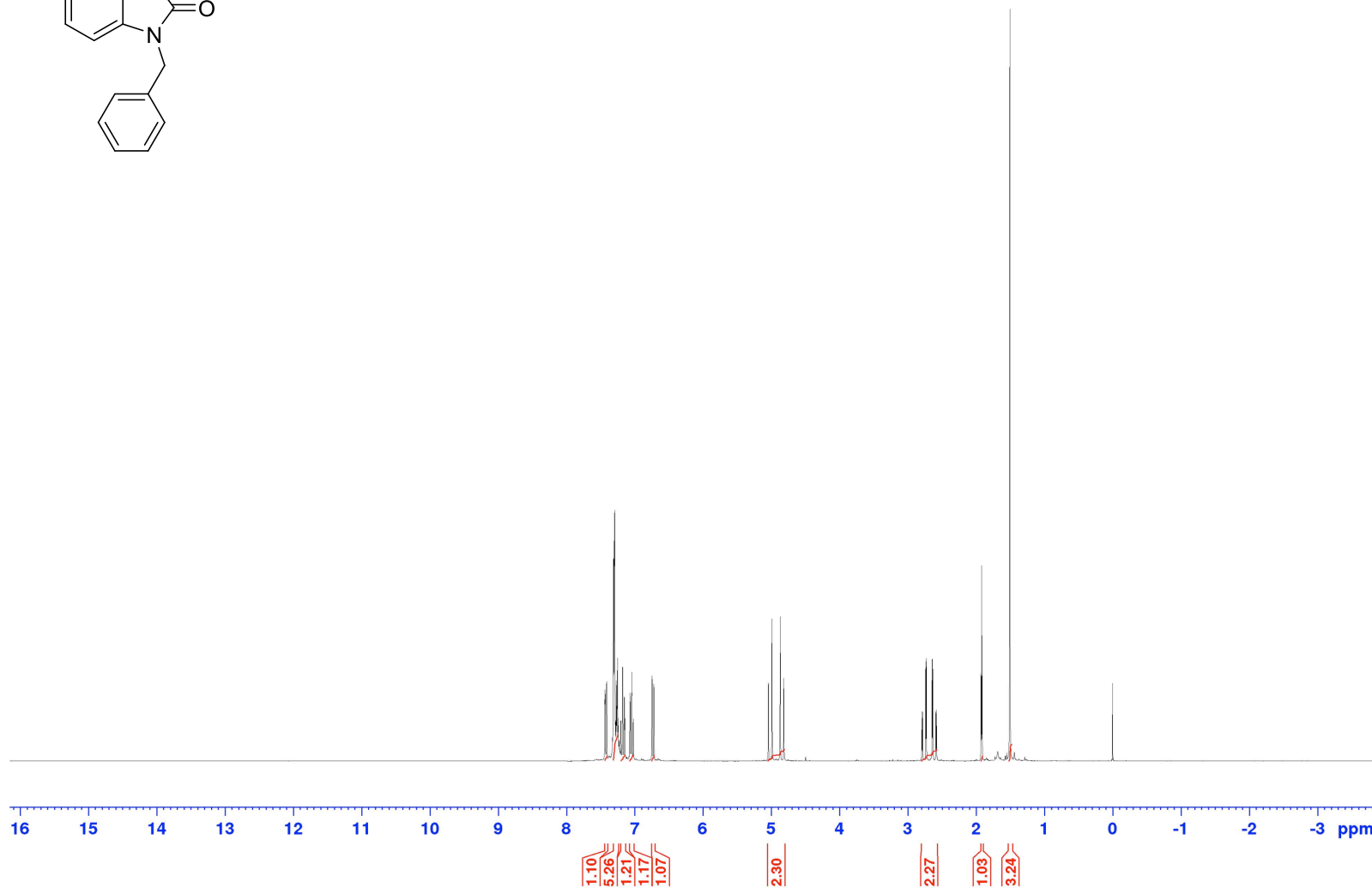
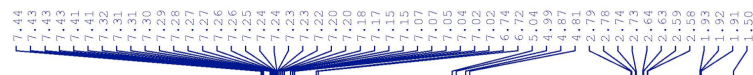


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EXPNO 10
PROCNO 1

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Time 17.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 228
DW 83.200 use
DE 12.89 use
TE 294.2 K
D1 1.00000000 sec
TD0 1

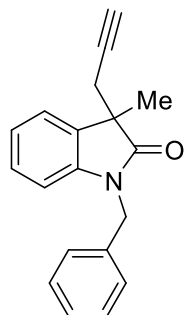
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NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
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SF 300.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR (1)

WBNBn



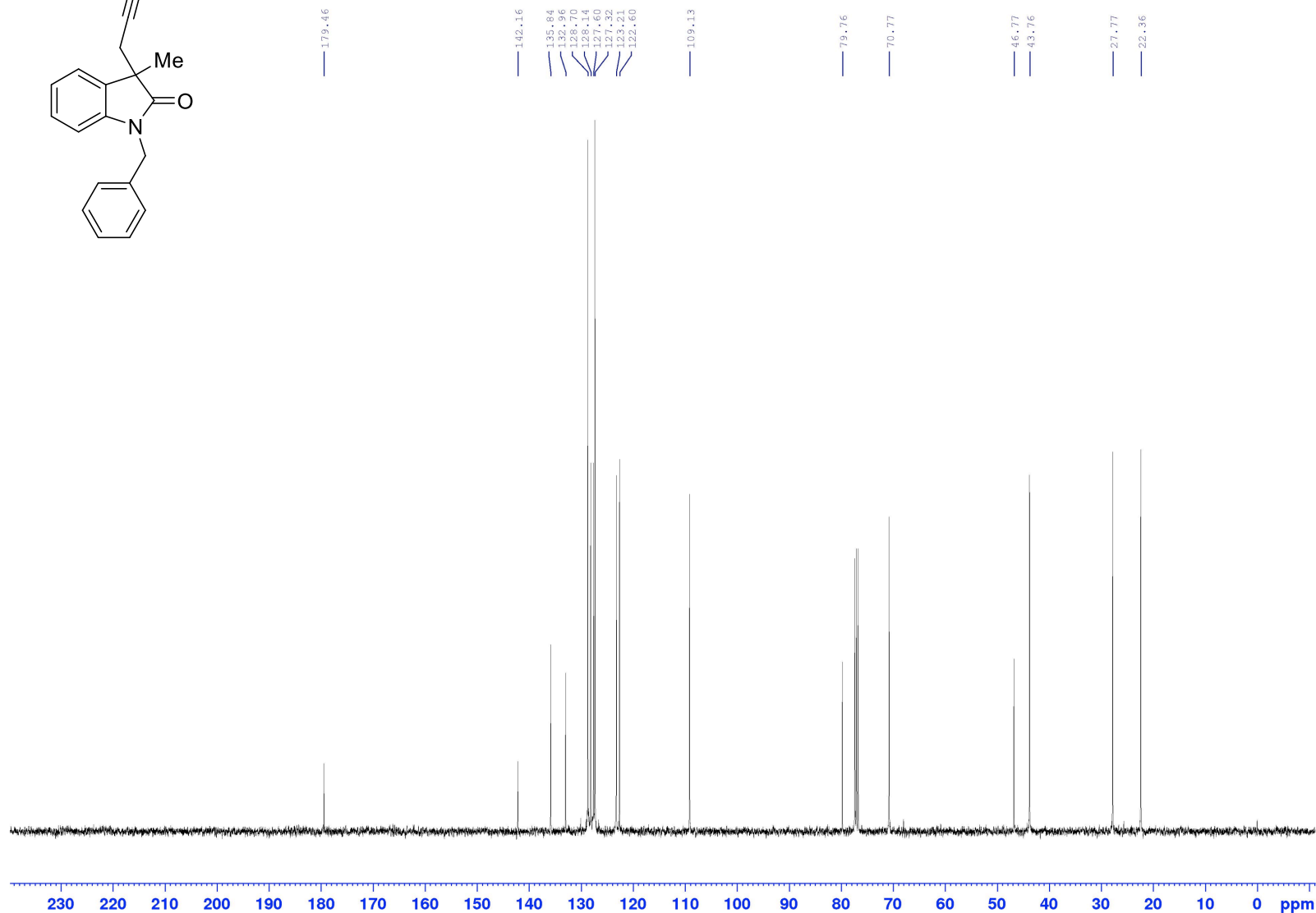
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EXPNO 10
PROCNO 1

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PULPROG udeflt
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.4 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

===== CHANNEL f1 =====
SFO1 100.6242690 MHz
NUC1 13C
P1 8.80 use
P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

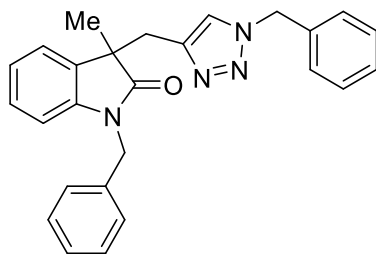
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NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

F2 - Processing parameters
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SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



¹H NMR (2a)

WB3-79

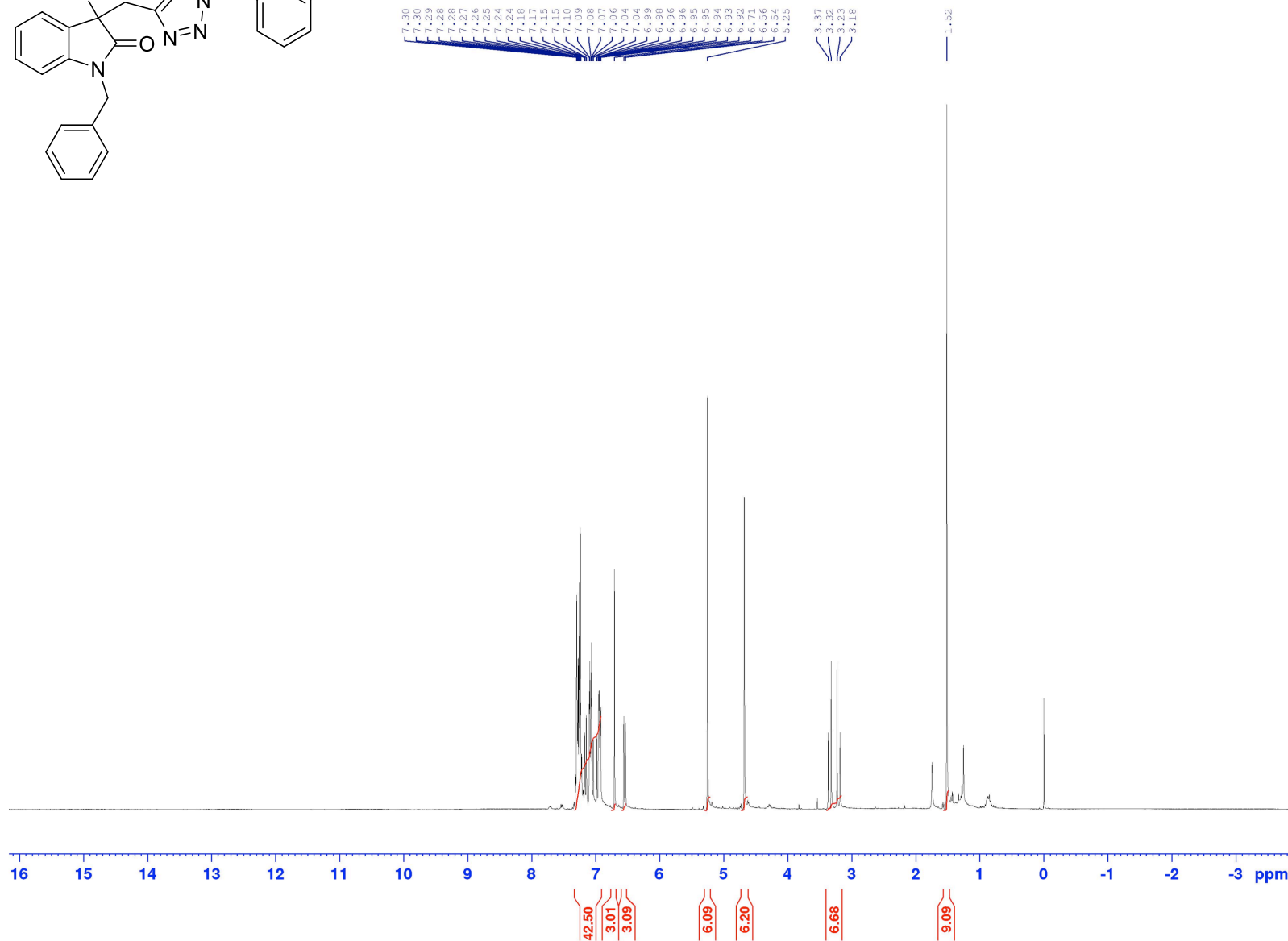


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EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150129
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INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 181
DW 83.200 use
DE 12.89 use
TE 294.3 K
D1 1.00000000 sec
TD0 1

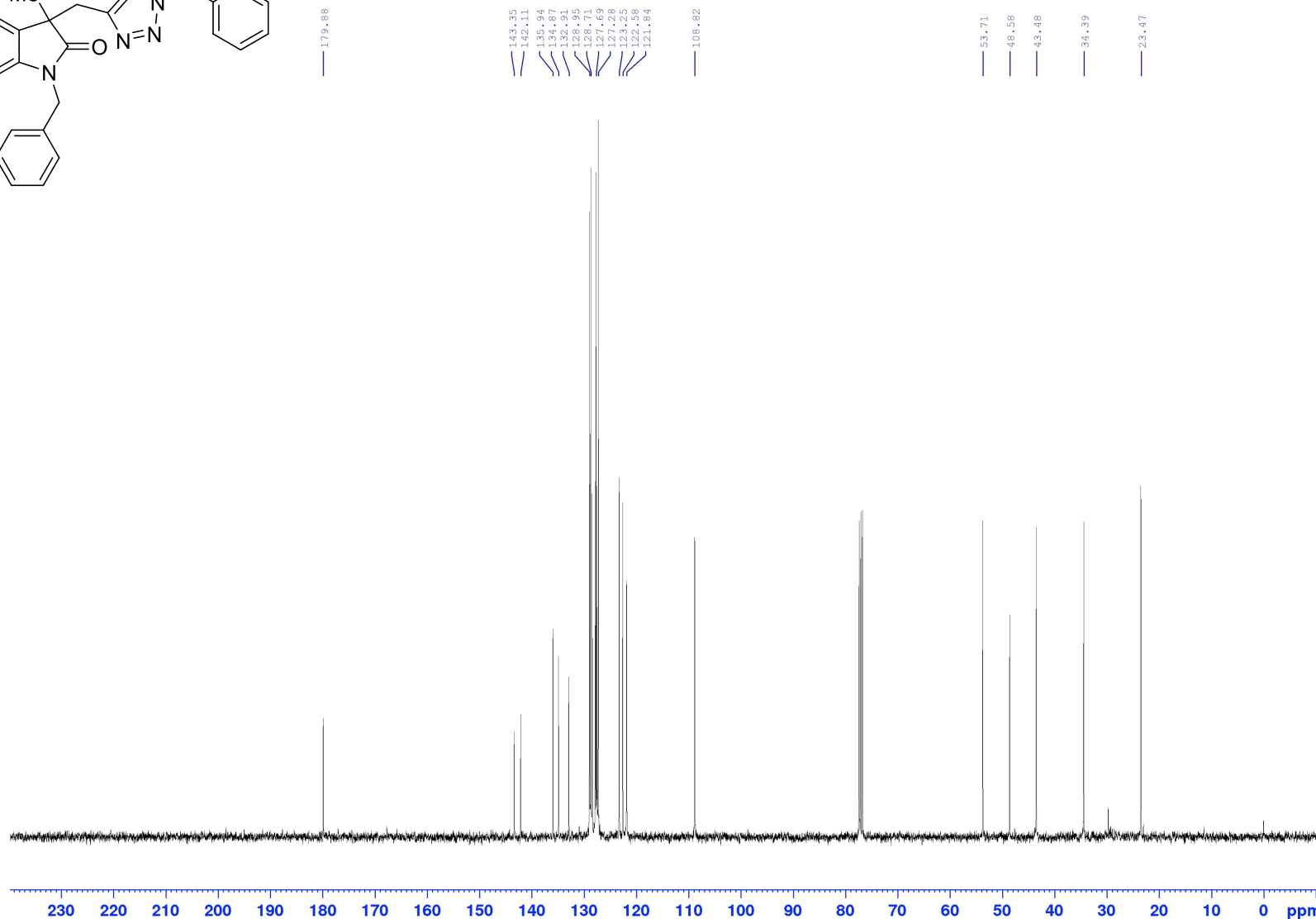
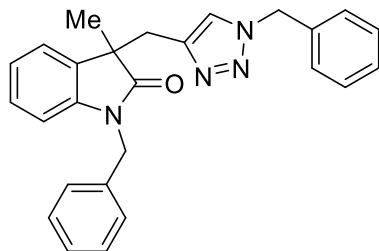
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SFO1 300.1318534 MHz
NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
SI 32768
SF 300.1300072 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR (2a)

WB3-79



Current Data Parameters
NAME 02-04-Fossey-9
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150204
Time 20.23
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG udeft
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.3 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

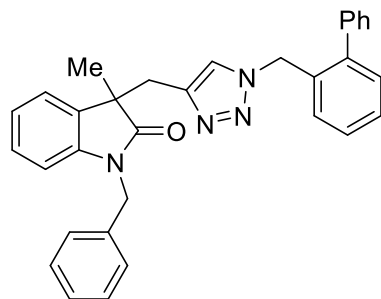
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P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

===== CHANNEL f2 =====
SFO2 400.1320000 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

F2 - Processing parameters
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SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹H NMR (2b)

WB C1-3

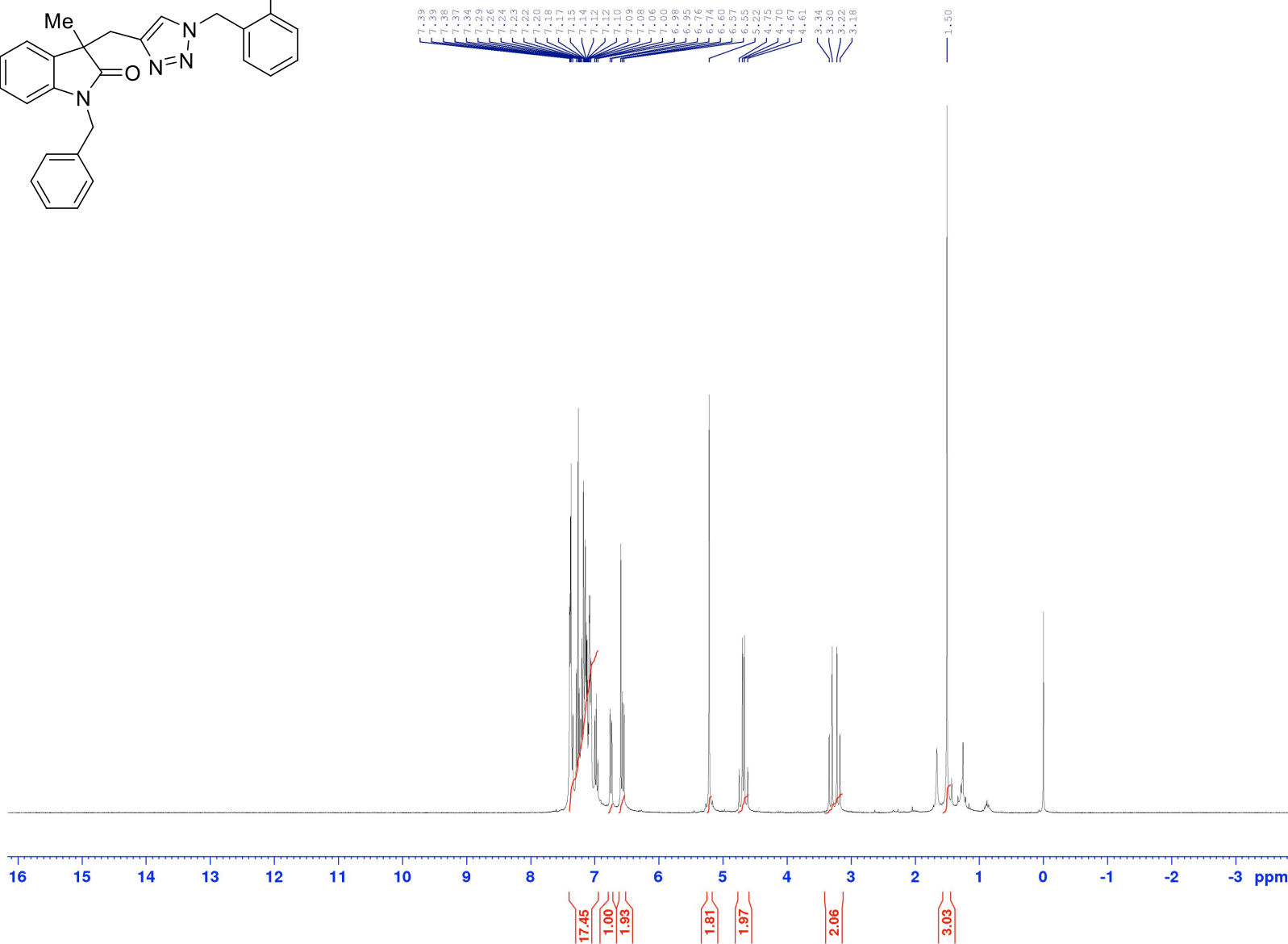


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EXPNO 10
PROCNO 1

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Time 10.40
INSTRUM spect
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PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 228
DW 83.200 use
DE 12.89 use
TE 294.2 K
D1 1.00000000 sec
TD0 1

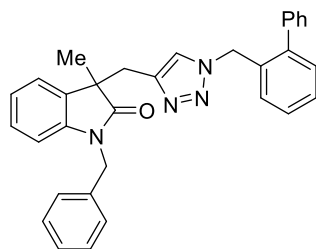
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NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR (2b)

WB C1-4-2



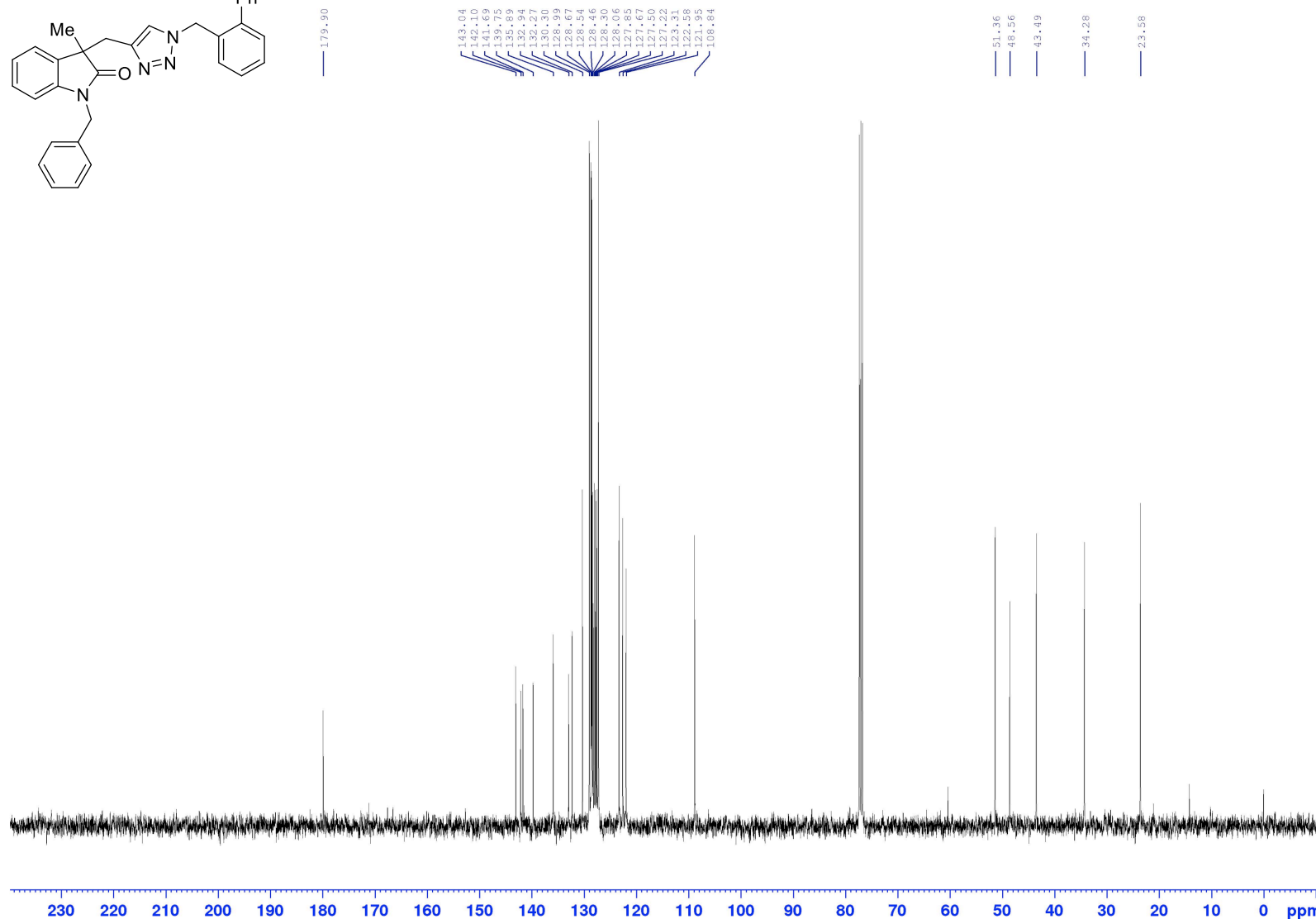
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NAME 02-10-Fossey-22
EXPNO 10
PROCNO 1

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INSTRUM spect
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PULPROG udeflt
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.1 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

===== CHANNEL f1 =====
SFO1 100.6242690 MHz
NUC1 13C
P1 8.80 use
P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

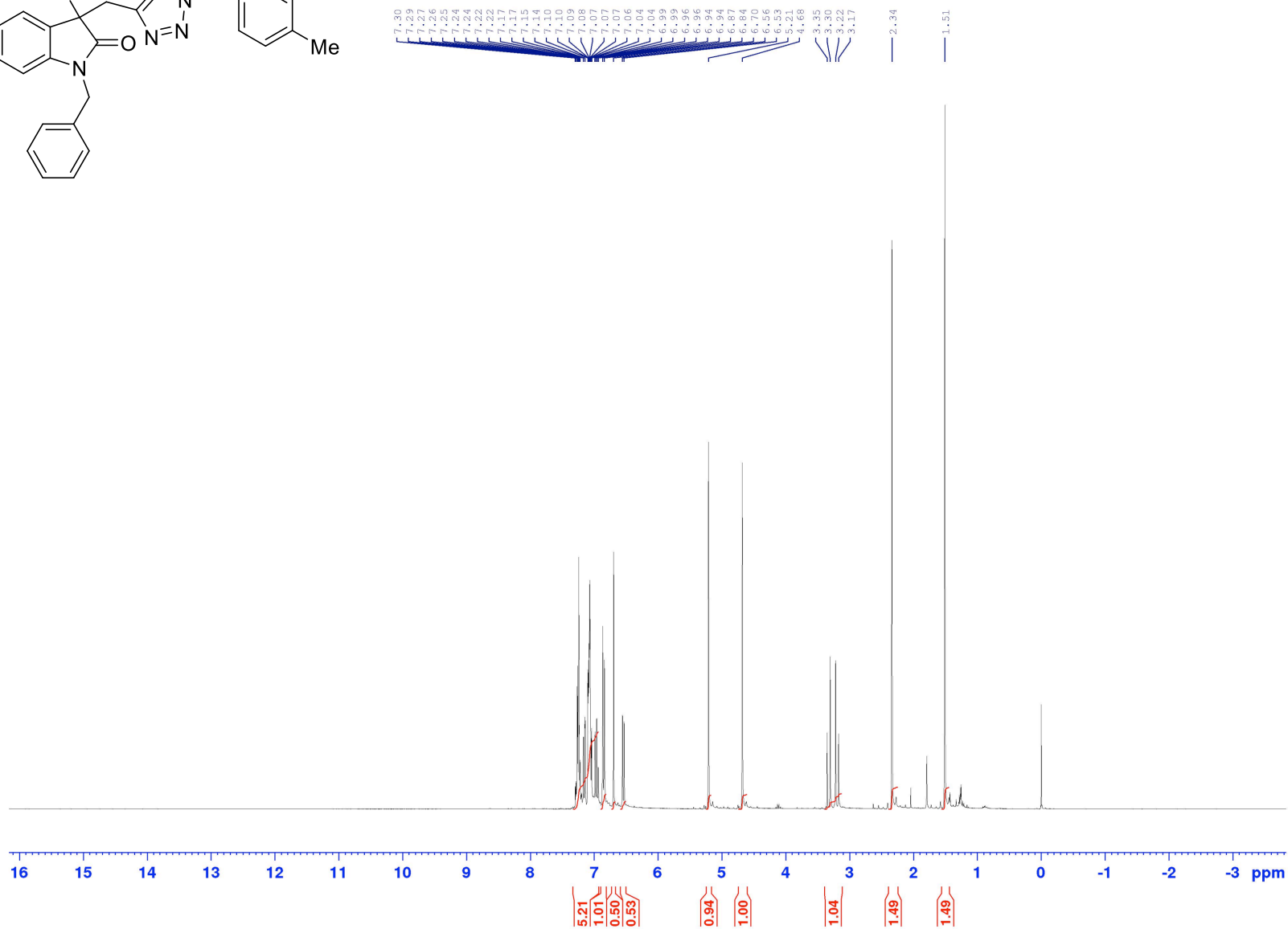
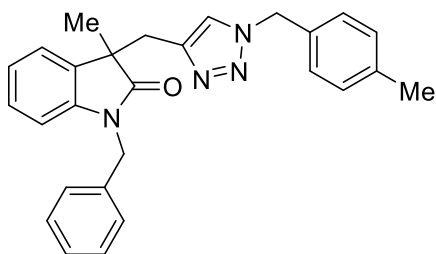
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NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



¹H NMR (2c)

WB C1-2



Current Data Parameters
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EXPNO 10
PROCNO 1

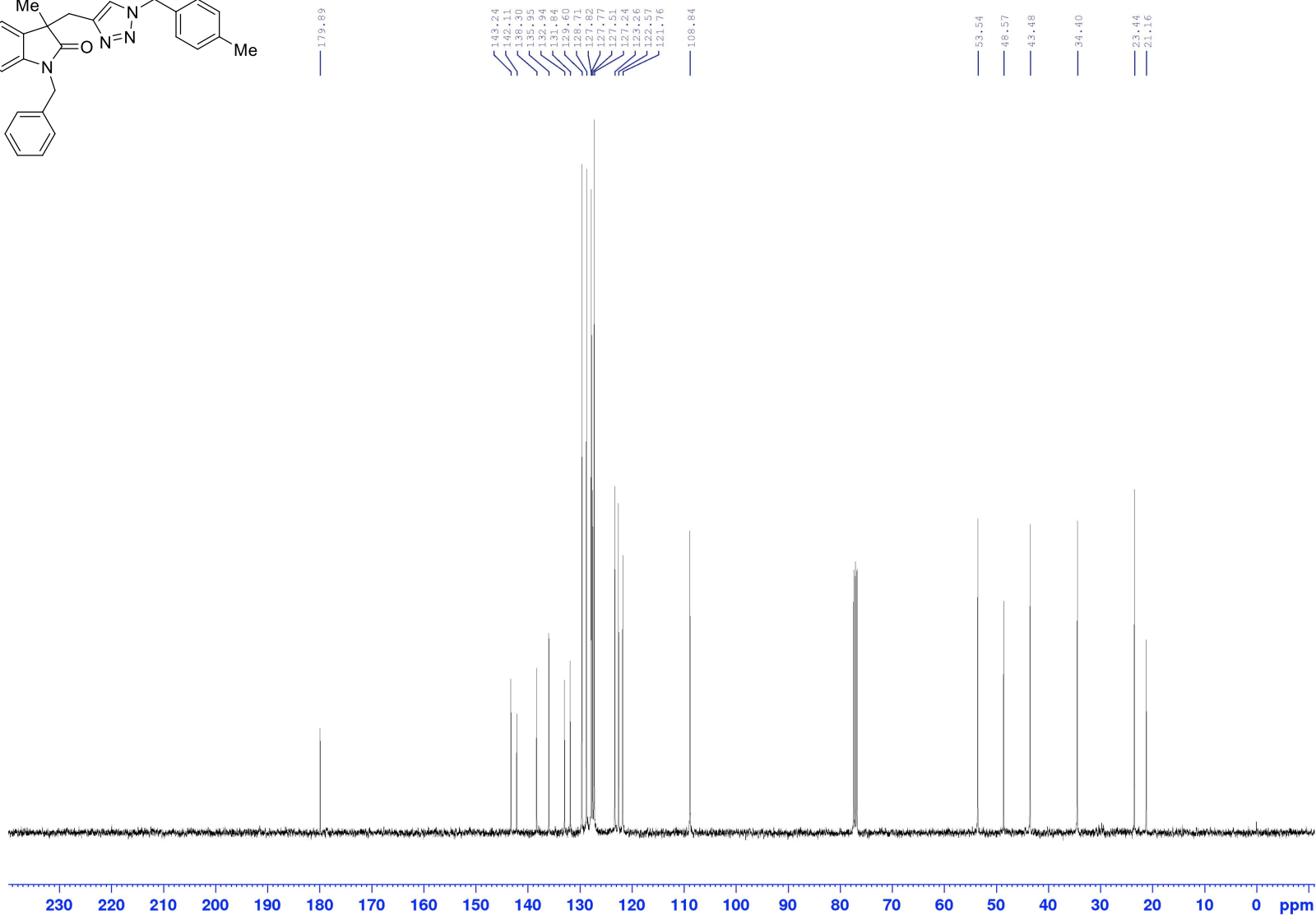
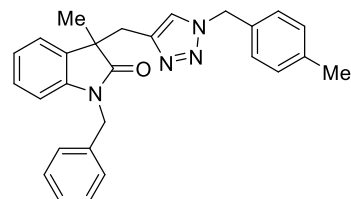
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Date_ 20150204
Time 16.19
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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 181
DW 83.200 use
DE 12.89 use
TE 293.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 300.1318534 MHz
NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
SI 32768
SF 300.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR (2c)

WB C1-2



Current Data Parameters
NAME 02-09-Fossey-2
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150209
Time 11.03
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG udeft
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.0 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

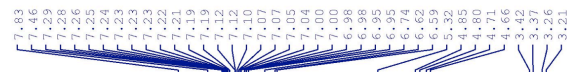
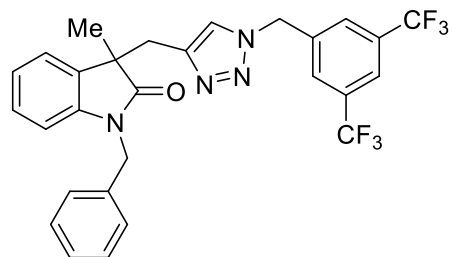
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P13 2000.00 use
P26 500.00 use
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SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

===== CHANNEL f2 =====
SFO2 400.1320000 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

F2 - Processing parameters
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SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹H NMR (2d)

WB C1-4

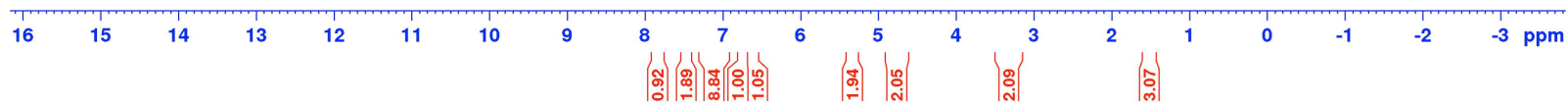


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EXPNO 10
PROCNO 1

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PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 181
DW 83.200 use
DE 12.89 use
TE 294.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 300.1318534 MHz
NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
SI 32768
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR (2d)



Current Data Parameters
NAME 02-09-Fossey-3
EXPNO 10
PROCNO 1

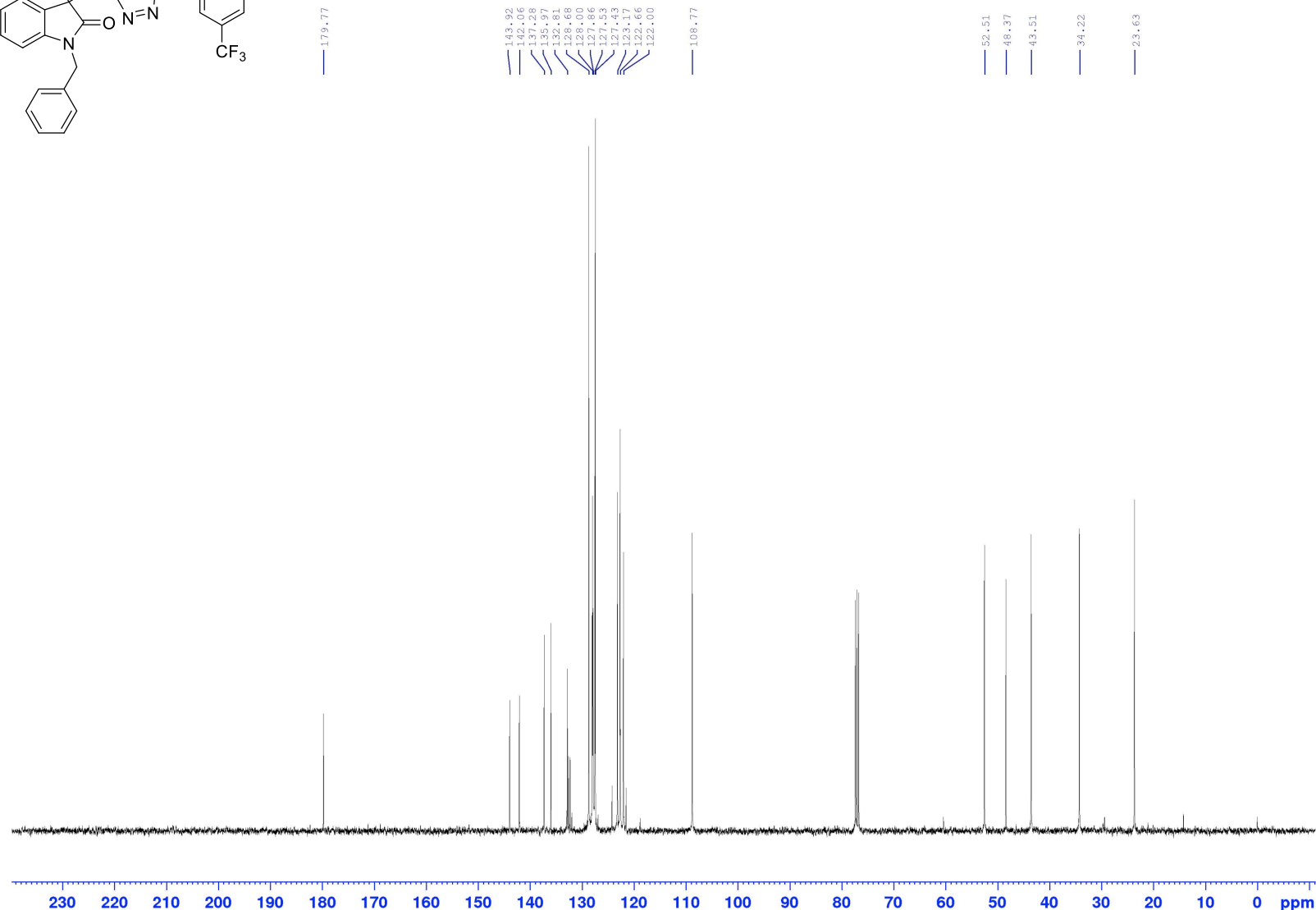
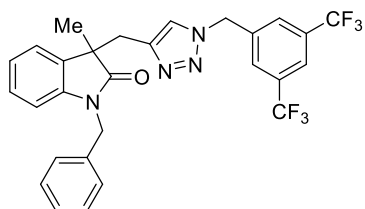
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Time 11.38
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG udeflt
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.3 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

===== CHANNEL f1 =====
SFO1 100.6242690 MHz
NUC1 13C
P1 8.80 use
P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

===== CHANNEL f2 =====
SFO2 400.1320000 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

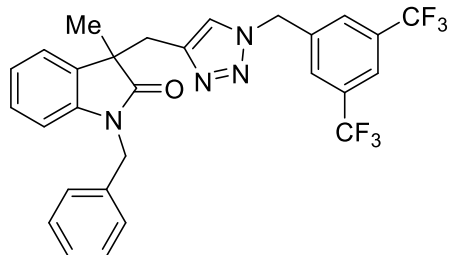
F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

WB C1-4



¹⁹F NMR (2d)

WBC1-4



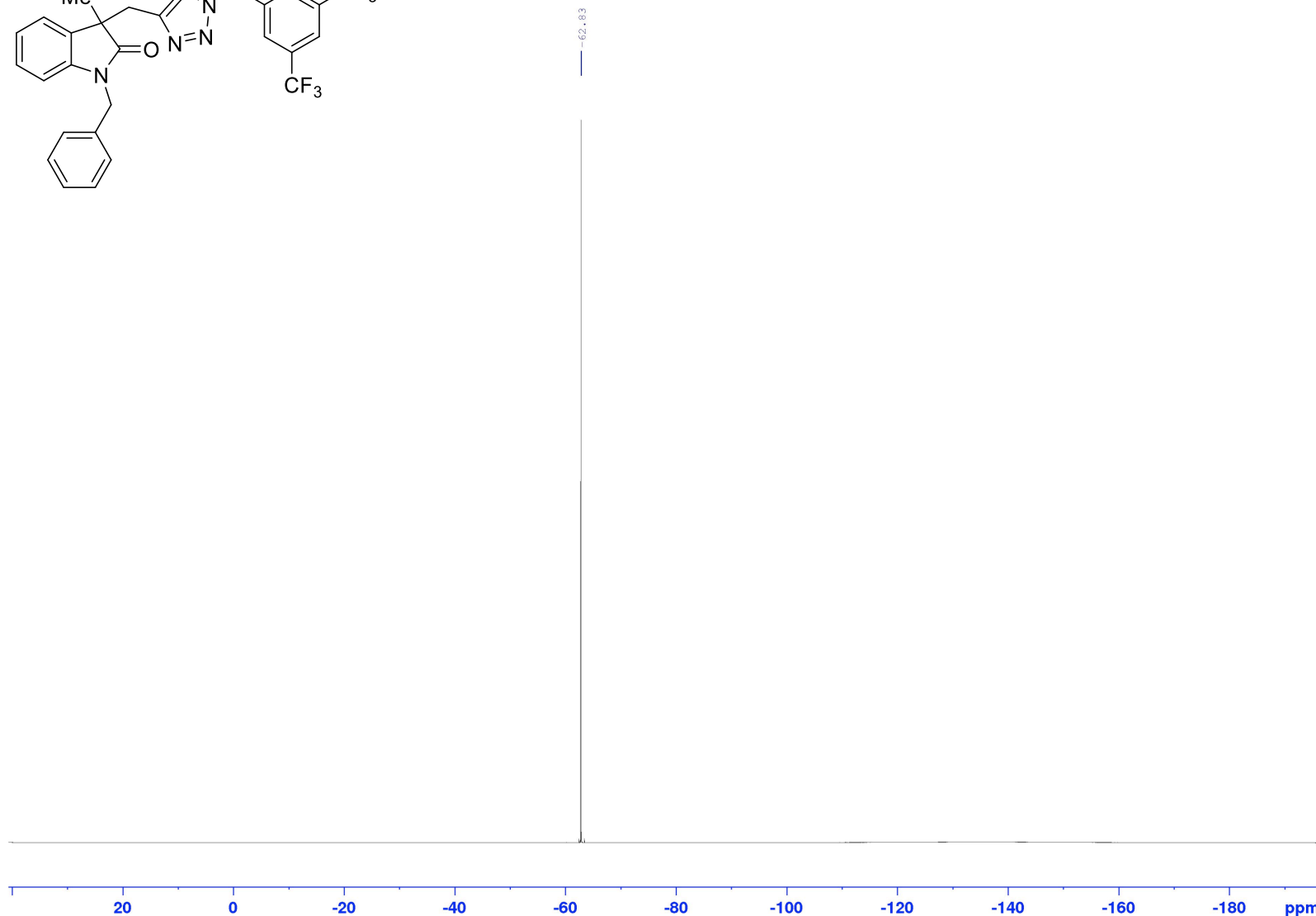
Current Data Parameters
NAME 05-05-Fossey-7
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150505
Time 11.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgig
TD 131072
SOLVENT CDCl₃
NS 32
DS 4
SWH 66964.289 Hz
FIDRES 0.510897 Hz
AQ 0.9786710 sec
RG 456
DW 7.467 use
DE 7.27 use
TE 294.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 282.3823550 MHz
NUC1 19F
P1 8.70 use
PLW1 30.58200073 W

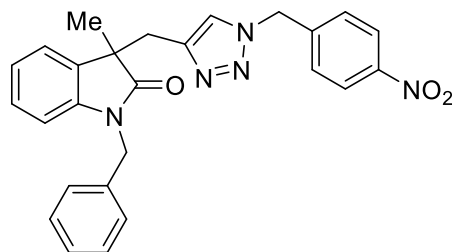
===== CHANNEL f2 =====
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 9.57730007 W
PLW12 0.19372000 W

F2 - Processing parameters
SI 131072
SF 282.4043550 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00



¹H NMR (2e)

WB C1-1

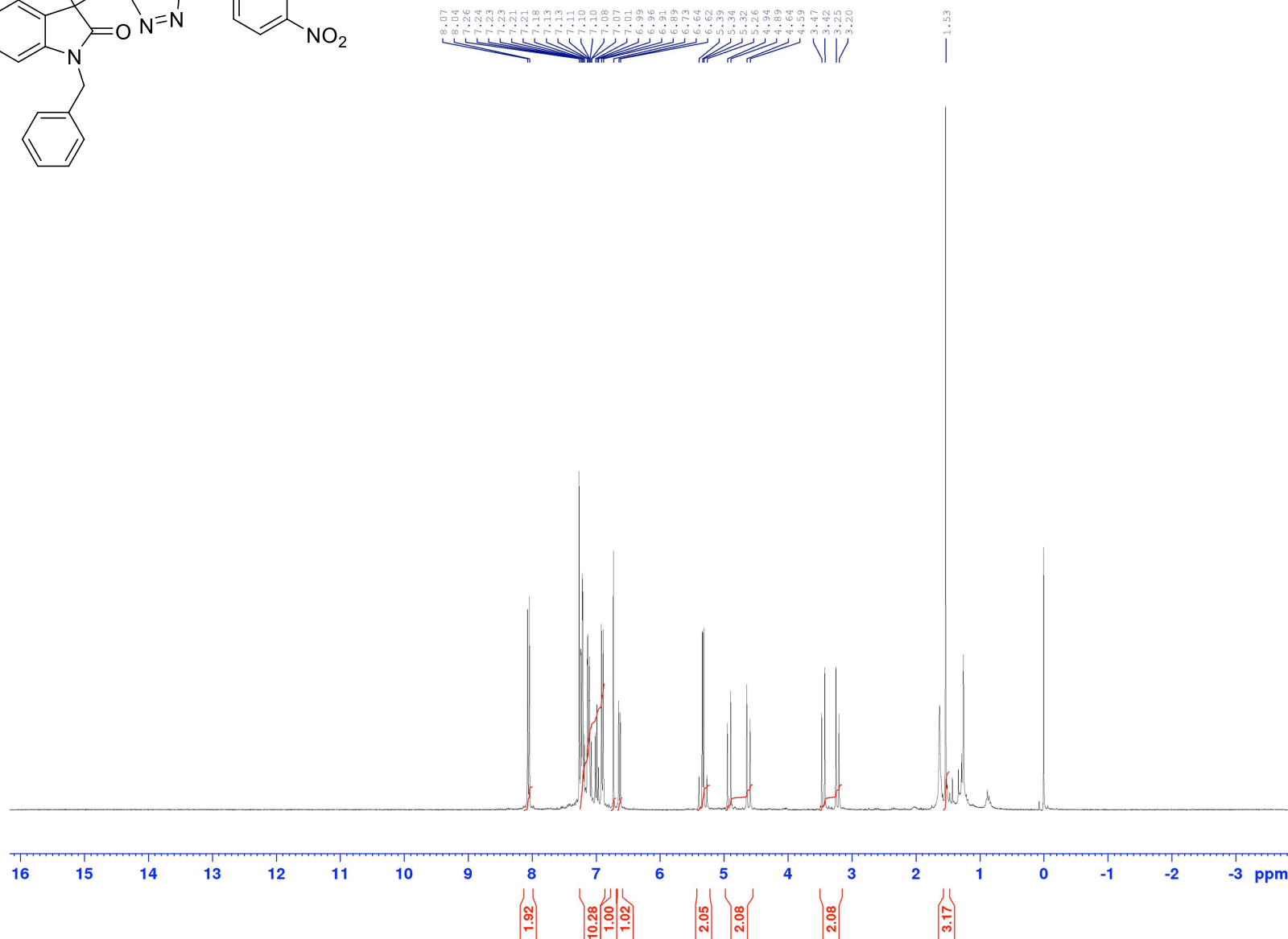


Current Data Parameters
NAME 02-19-Fossey-37
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150219
Time 15.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 456
DW 83.200 use
DE 12.89 use
TE 294.3 K
D1 1.00000000 sec
TD0 1

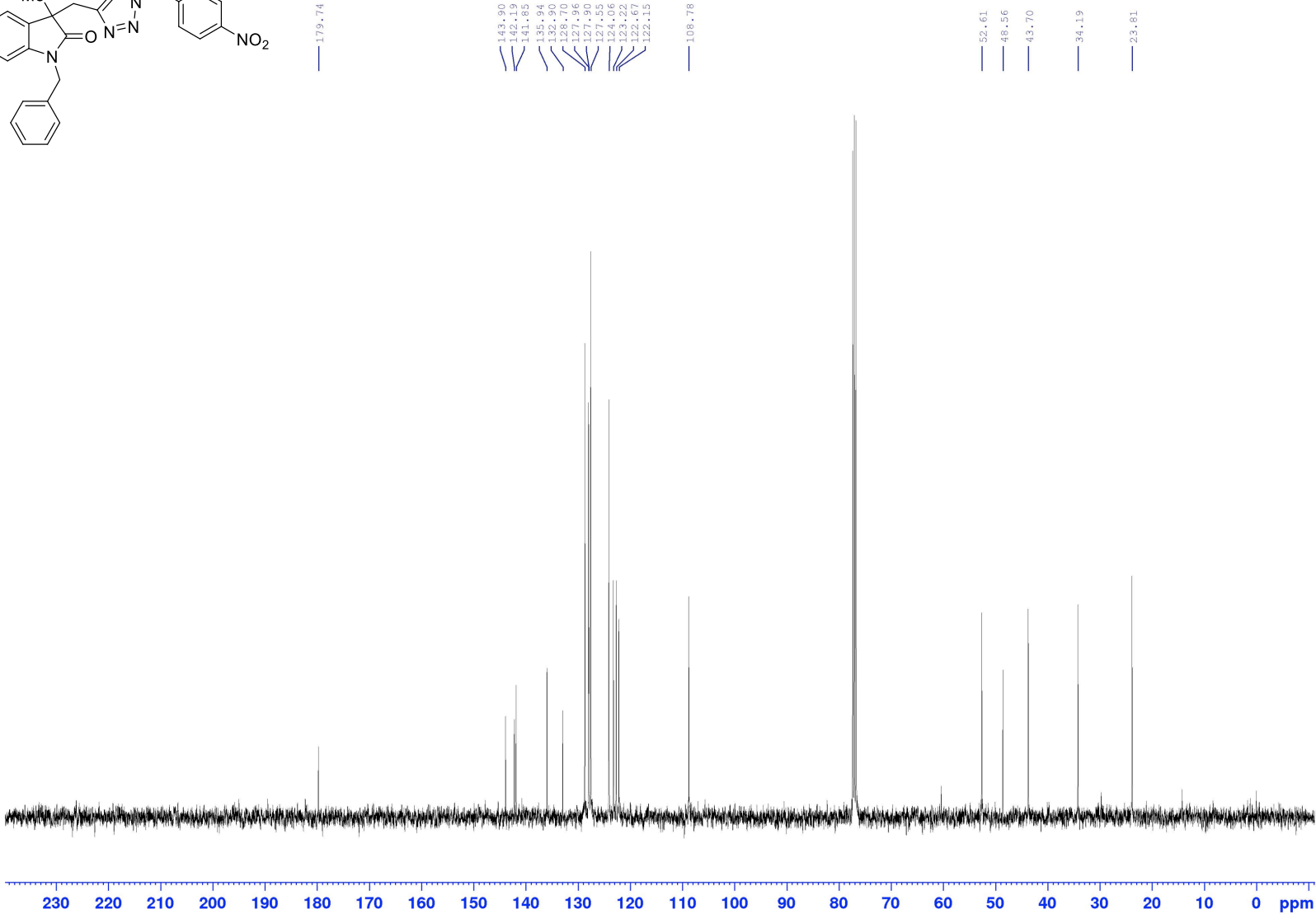
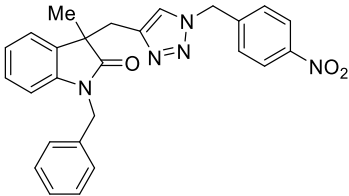
===== CHANNEL f1 =====
SFO1 300.1318534 MHz
NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
SI 32768
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



13C NMR (2e)

WB C1-1



Current Data Parameters
NAME 02-04-Fossey-10
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150204
Time 20.59
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG udeft
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.3 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

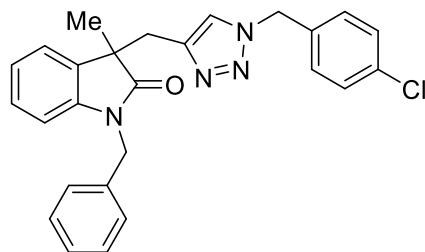
===== CHANNEL f1 =====
SFO1 100.6242690 MHz
NUC1 13C
P1 8.80 use
P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

===== CHANNEL f2 =====
SFO2 400.1320000 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹H NMR (2f)

WB C1-6

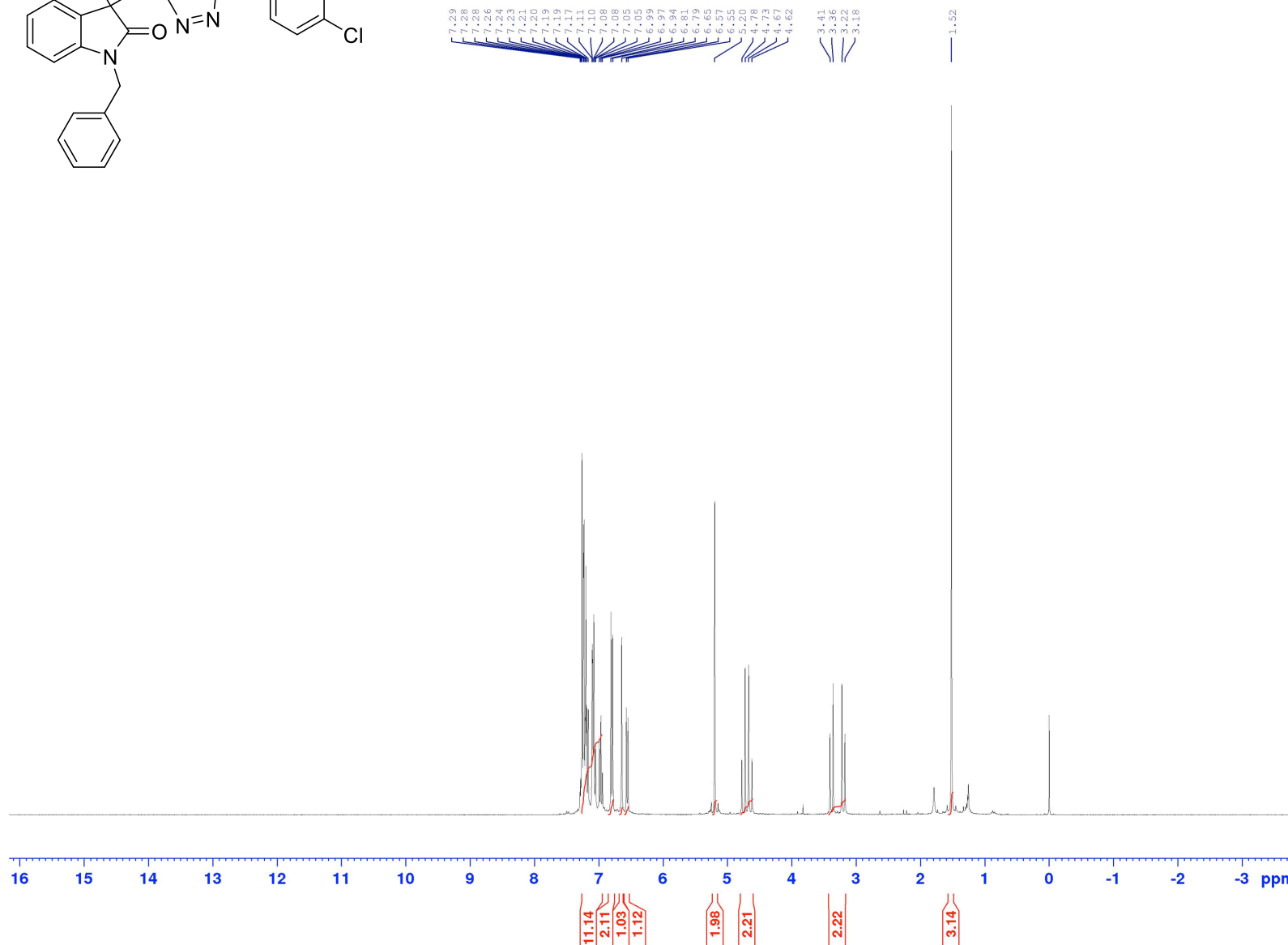


Current Data Parameters
NAME 02-20-Fossey-34
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150220
Time 14.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 161
DW 83.200 use
DE 12.89 use
TE 294.2 K
D1 1.00000000 sec
TD0 1

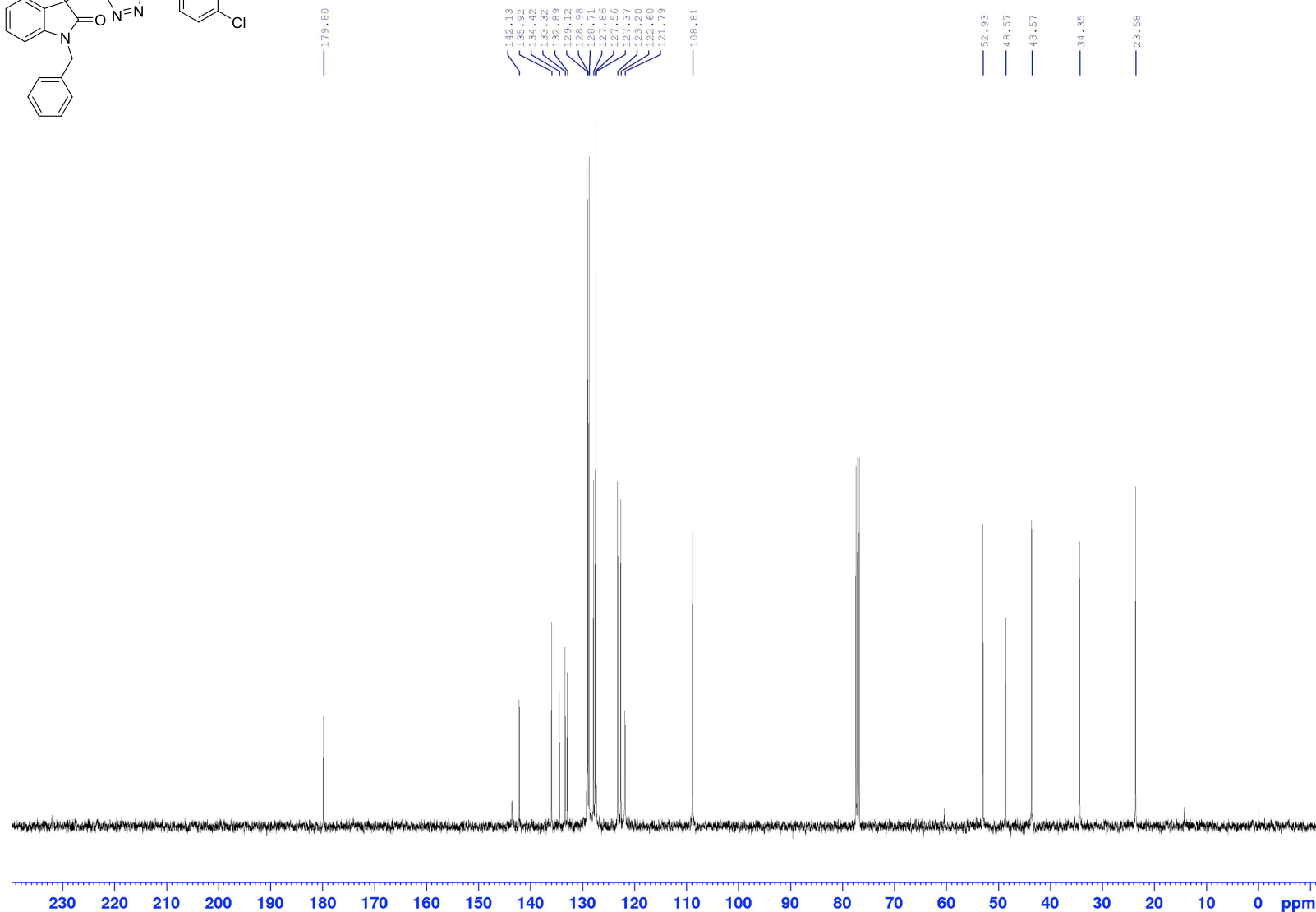
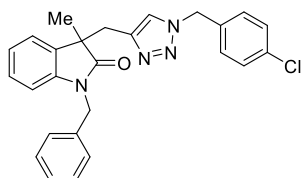
===== CHANNEL f1 =====
SFO1 300.1318534 MHz
NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
SI 32768
SF 300.1300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR (2f)

WB C1-6



Current Data Parameters
NAME 02-12-Fossey-13
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150212
Time 19.44
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG udeflt
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.2 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

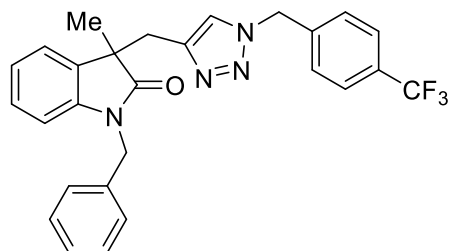
===== CHANNEL f1 =====
SFO1 100.6242690 MHz
NUC1 13C
P1 8.80 use
P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

===== CHANNEL f2 =====
SFO2 400.1320000 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

¹H NMR (2g)

WB C1-5

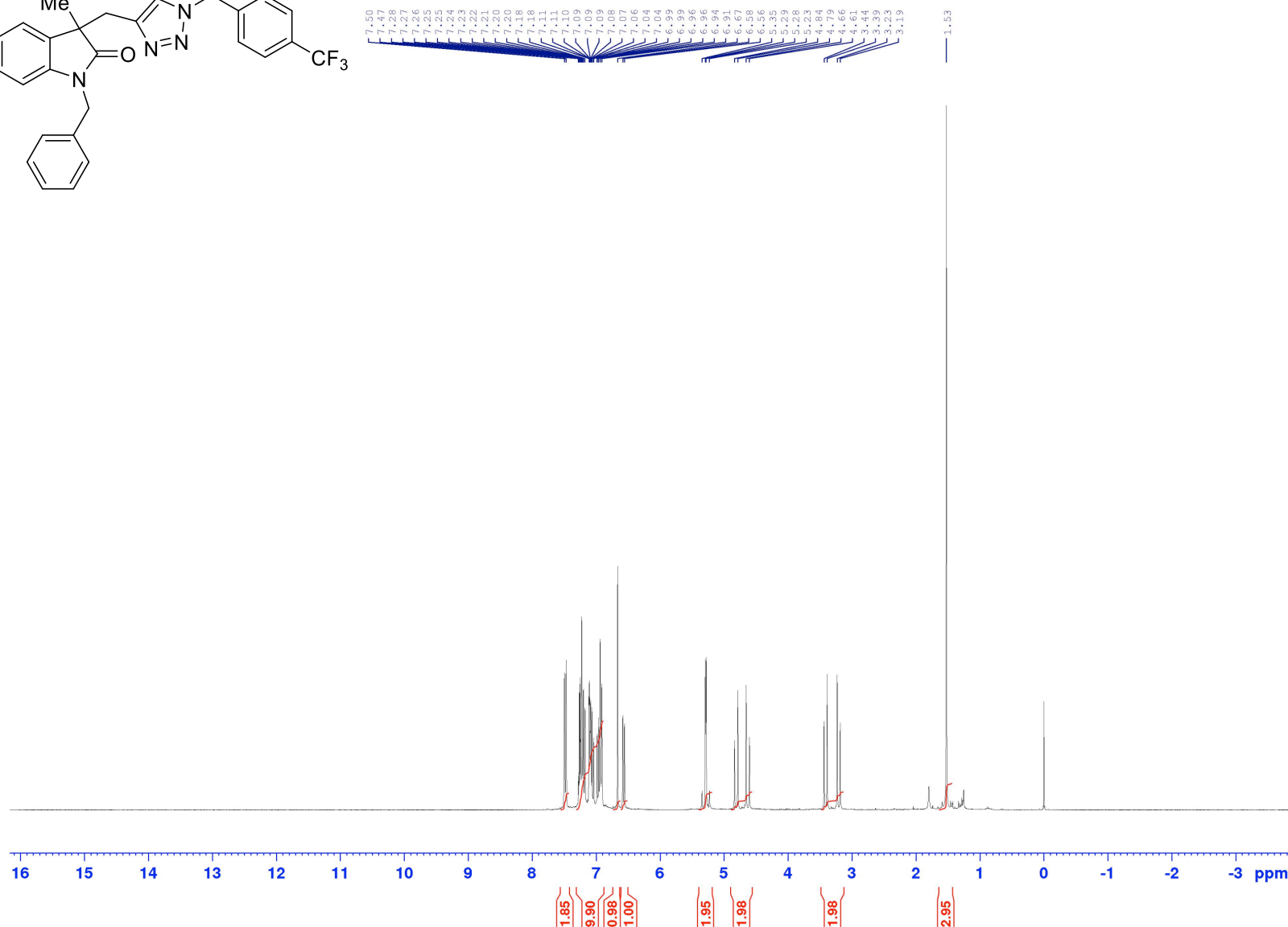


Current Data Parameters
NAME 02-09-Fossey-3-1H
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150209
Time 19.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 161
DW 83.200 use
DE 12.89 use
TE 294.2 K
D1 1.00000000 sec
TD0 1

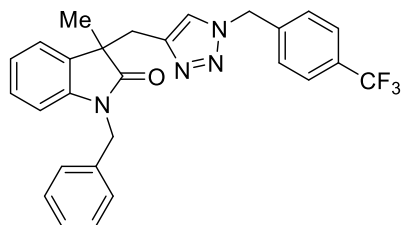
===== CHANNEL f1 =====
SF01 300.1318534 MHz
NUC1 1H
P1 12.80 use
PLW1 9.57730007 W

F2 - Processing parameters
SI 32768
SF 300.1300064 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹³C NMR (2g)

WB C1-5



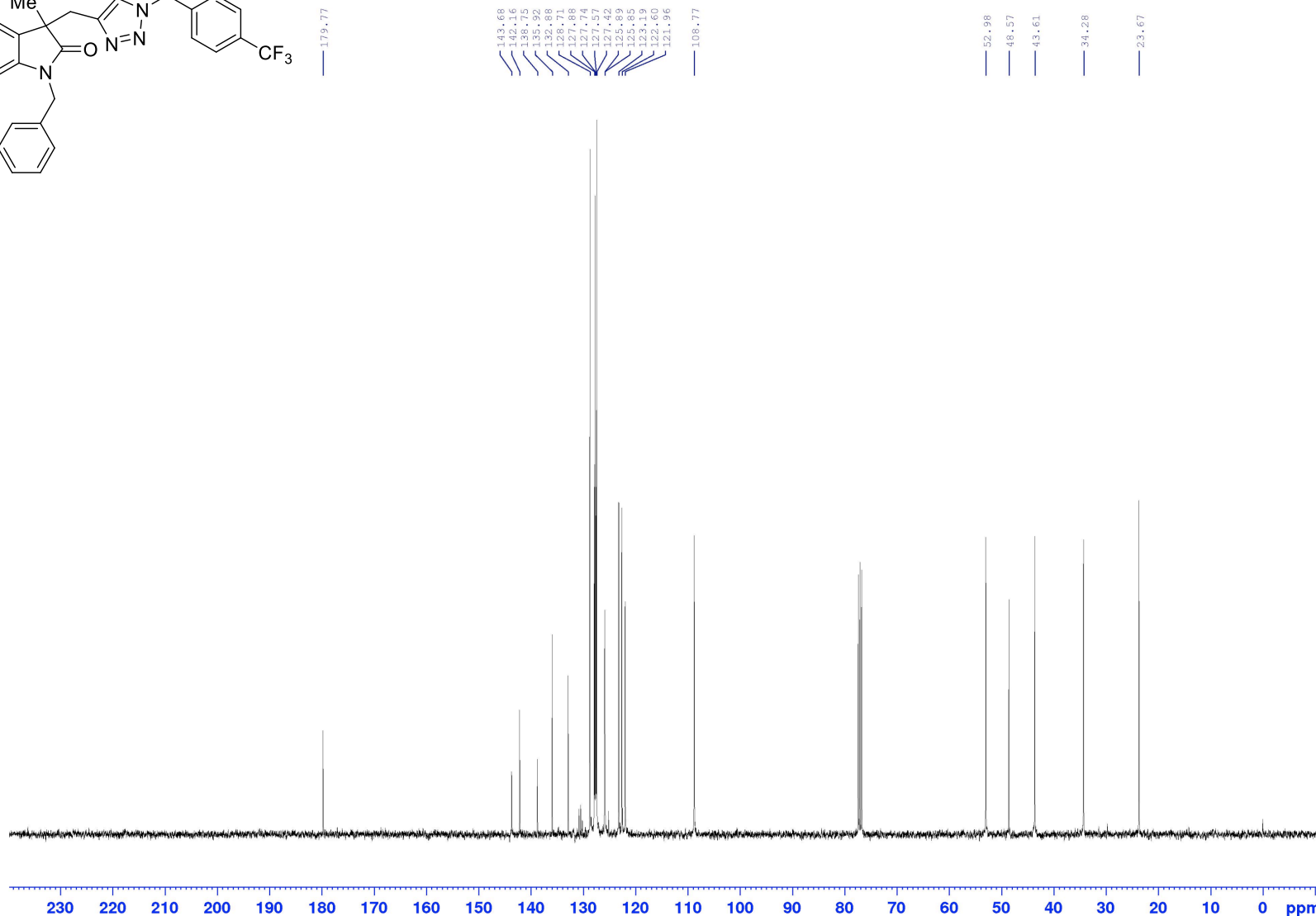
Current Data Parameters
NAME 02-10-Fossey-23
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150211
Time 3.01
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG udef
TD 18178
SOLVENT CDCl3
NS 380
DS 0
SWH 25252.525 Hz
FIDRES 1.389181 Hz
AQ 0.3599244 sec
RG 2050
DW 19.800 use
DE 8.20 use
TE 293.2 K
D1 3.00000000 sec
D11 0.03000000 sec
D12 0.00002000 sec
D20 200.00000000 sec
TD0 380

===== CHANNEL f1 =====
SFO1 100.6242690 MHz
NUC1 13C
P1 8.80 use
P13 2000.00 use
P26 500.00 use
PLW1 58.63899994 W
SPNAM[5] Crp60comp.4
SPOAL5 0.500
SPOFFS5 0 Hz
SPW5 6.93809986 W
SPNAM[8] Crp60,0.5,20.1
SPOAL8 0.500
SPOFFS8 0 Hz
SPW8 6.93809986 W

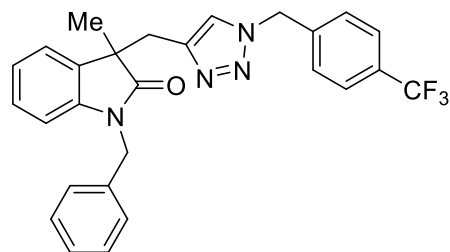
===== CHANNEL f2 =====
SFO2 400.1320000 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 24.29199982 W
PLW12 0.28218001 W

F2 - Processing parameters
SI 65536
SF 100.6127690 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00



¹⁹F NMR (2g)

WBC1-4



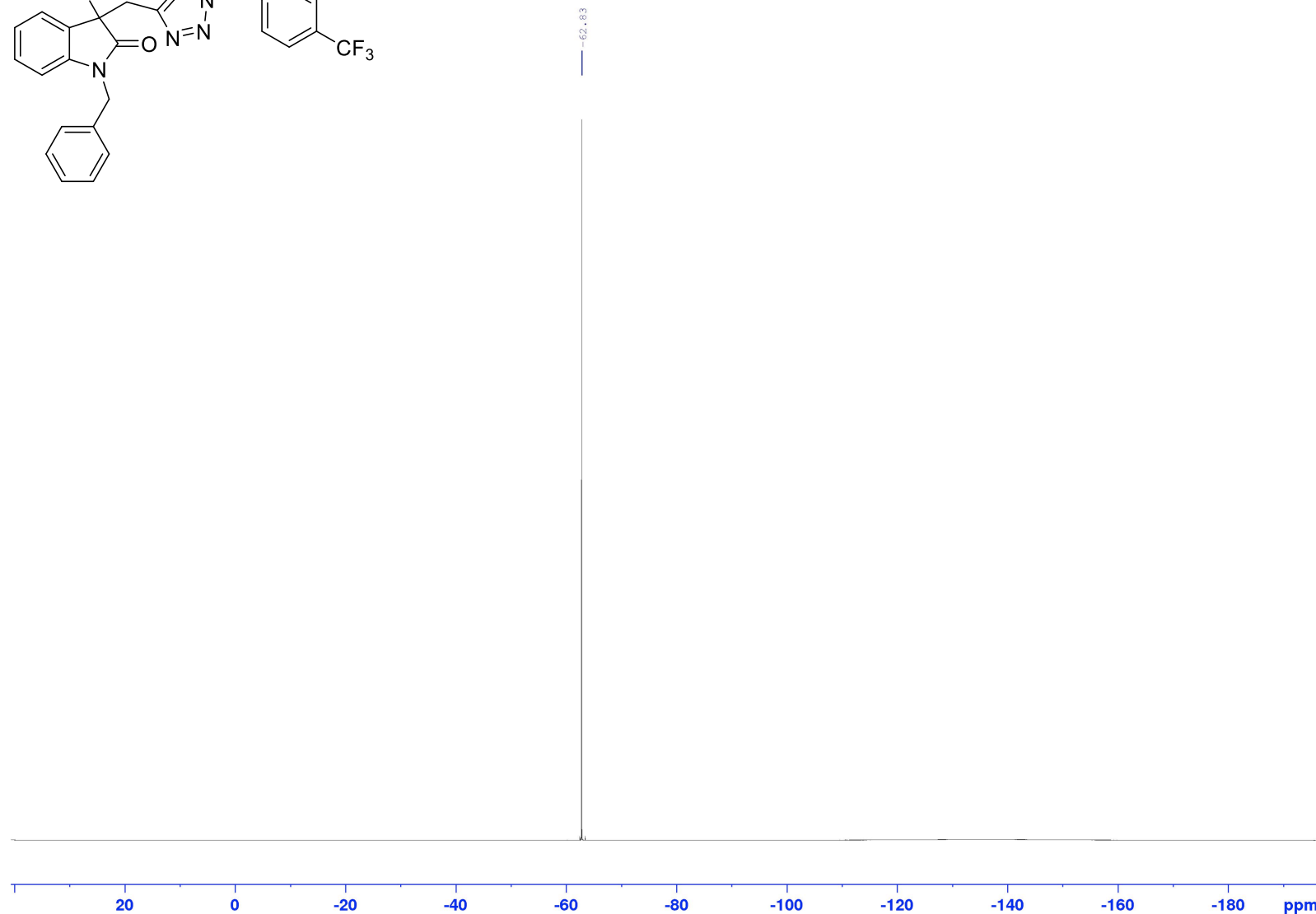
Current Data Parameters
NAME 05-05-Fossey-7
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150505
Time 11.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgig
TD 131072
SOLVENT CDCl₃
NS 32
DS 4
SWH 66964.289 Hz
FIDRES 0.510897 Hz
AQ 0.9786710 sec
RG 456
DW 7.467 use
DE 7.27 use
TE 294.2 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 282.3823550 MHz
NUC1 19F
P1 8.70 use
PLW1 30.58200073 W

===== CHANNEL f2 =====
SFO2 300.1312005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 90.00 use
PLW2 9.57730007 W
PLW12 0.19372000 W

F2 - Processing parameters
SI 131072
SF 282.4043550 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00



HPLC Traces

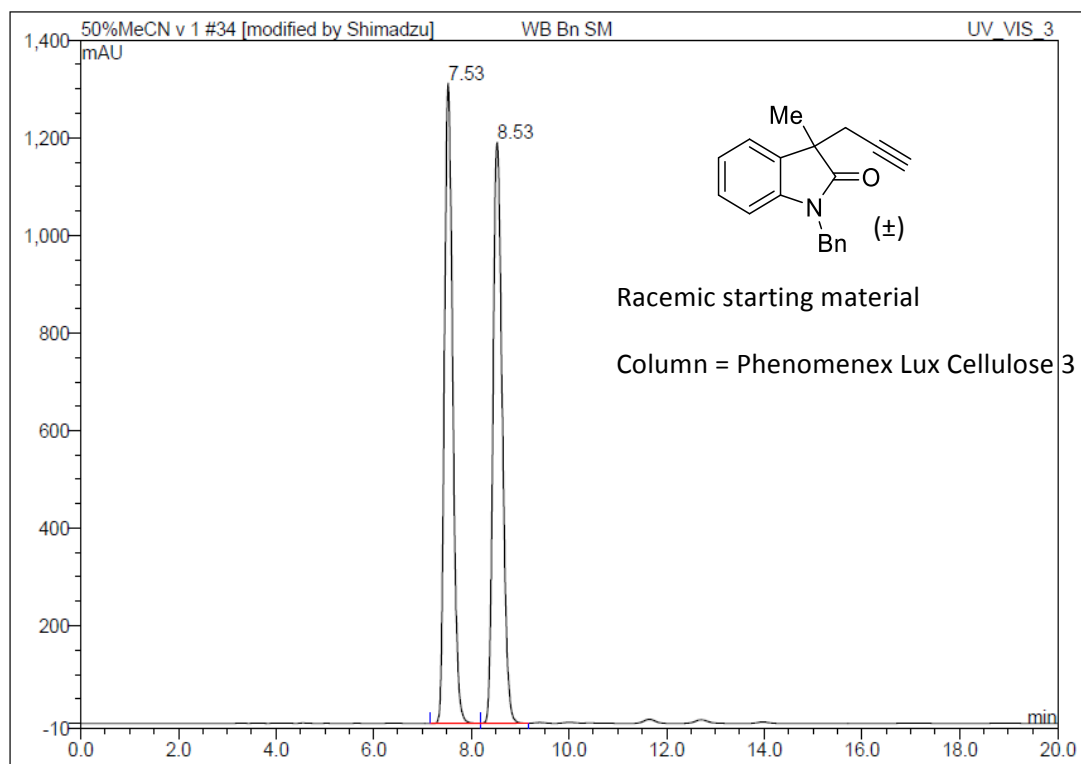
Operator:Shimadzu Timebase:LC_System1 Sequence:50%MeCN v 1

Page 1-1
18/2/2015 3:48 PM

34 WB Bn SM

Cell-3 50% MeCN 50% water, 1ml/min

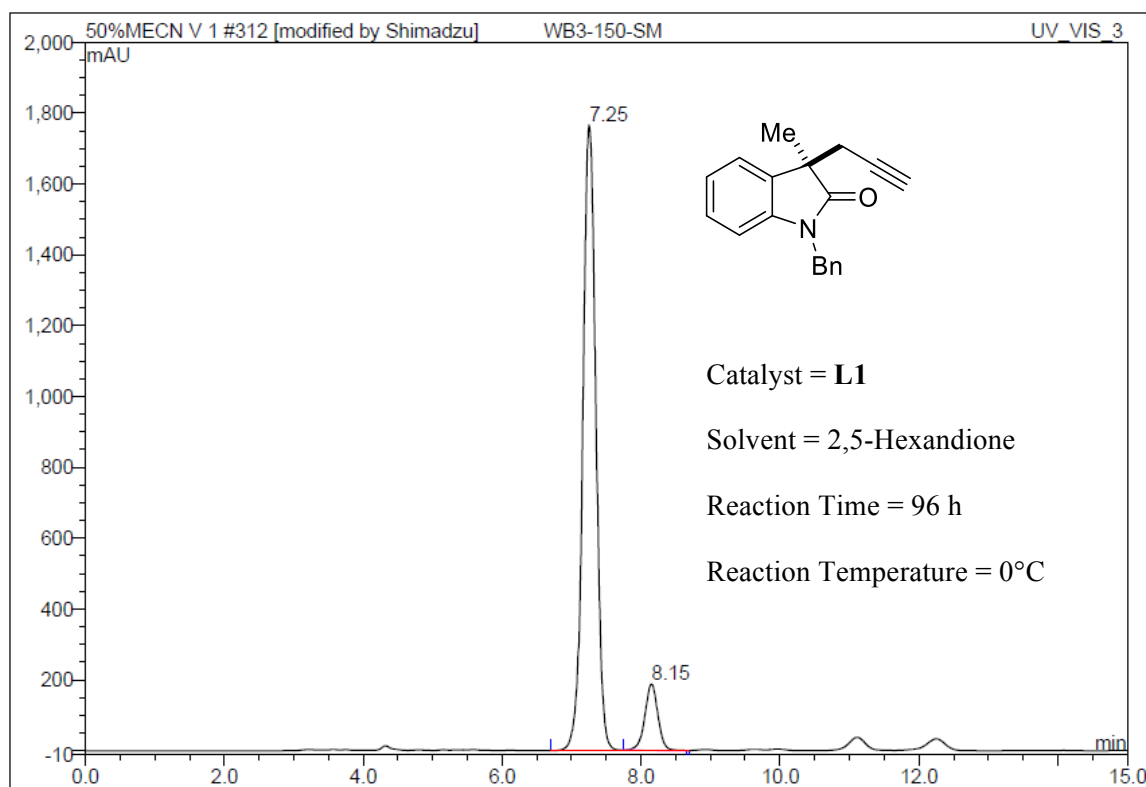
Sample Name:	WB Bn SM	Injection Volume:	10.0
Vial Number:	1_2	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 13:32	Sample Weight:	1.0000
Run Time (min):	20.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.53	n.a.	1311.427	259.060	49.82	n.a.	BM
2	8.53	n.a.	1189.898	260.893	50.18	n.a.	MB
Total:			2501.325	519.952	100.00	0.000	

312 WB3-150-SM**50% MeCN 50% Water Cell-3, 1mL/min**

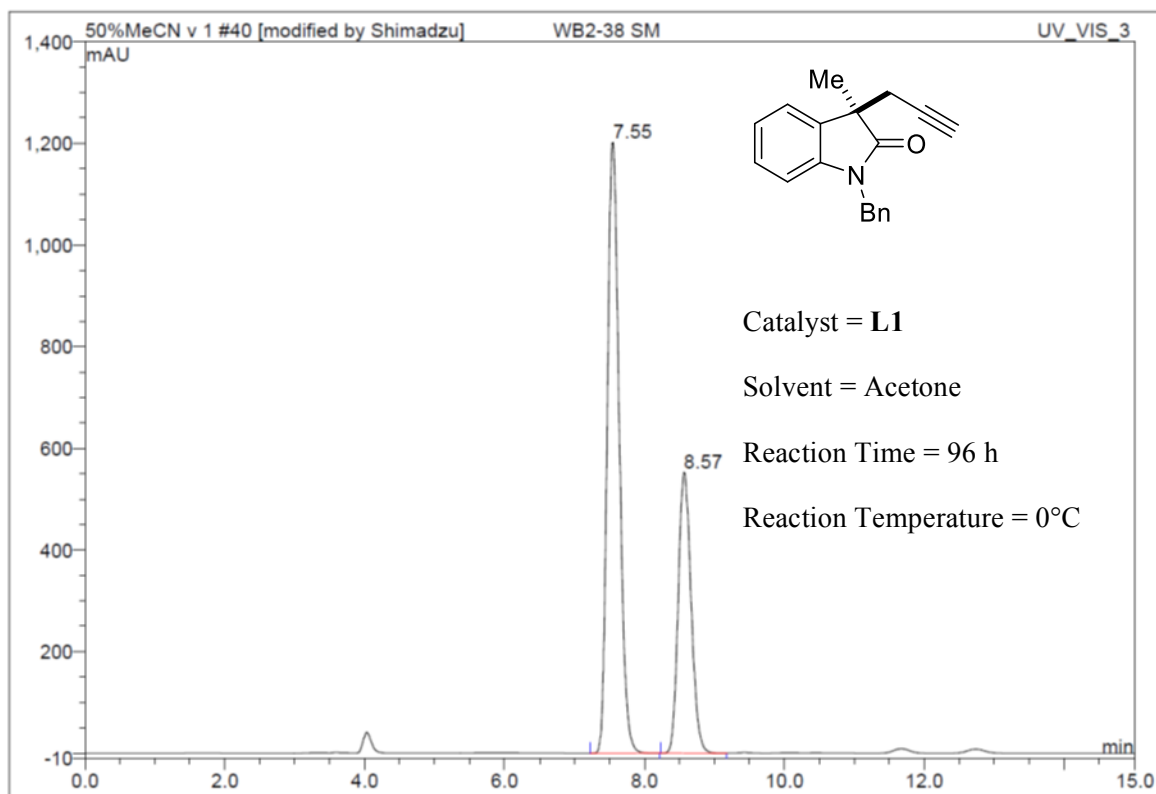
Sample Name:	WB3-150-SM	Injection Volume:	10.0
Vial Number:	1_6	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	21/3/2015 19:35	Sample Weight:	1.0000
Run Time (min):	30.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.25	n.a.	1765.969	383.333	90.40	n.a.	BMB
2	8.15	n.a.	186.806	40.689	9.60	n.a.	Rd
Total:			1952.775	424.023	100.00	0.000	

40 WB2-38 SM**Cell-3 50% MeCN 50% water, 1ml/min**

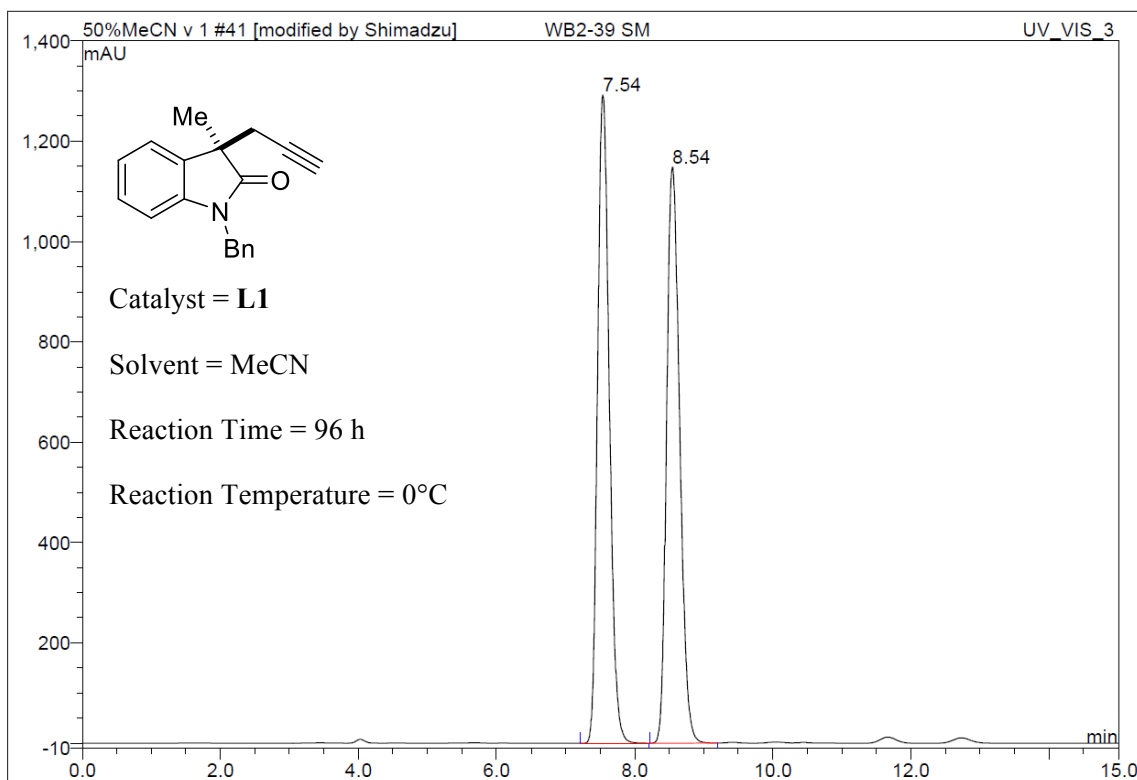
Sample Name:	WB2-38 SM	Injection Volume:	10.0
Vial Number:	1_7	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 17:27	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.55	n.a.	1201.675	237.272	66.78	n.a.	BMB
2	8.57	n.a.	552.682	118.011	33.22	n.a.	BMB
Total:			1754.357	355.283	100.00	0.000	

41 WB2-39 SM**Cell-3 50% MeCN 50% water, 1ml/min**

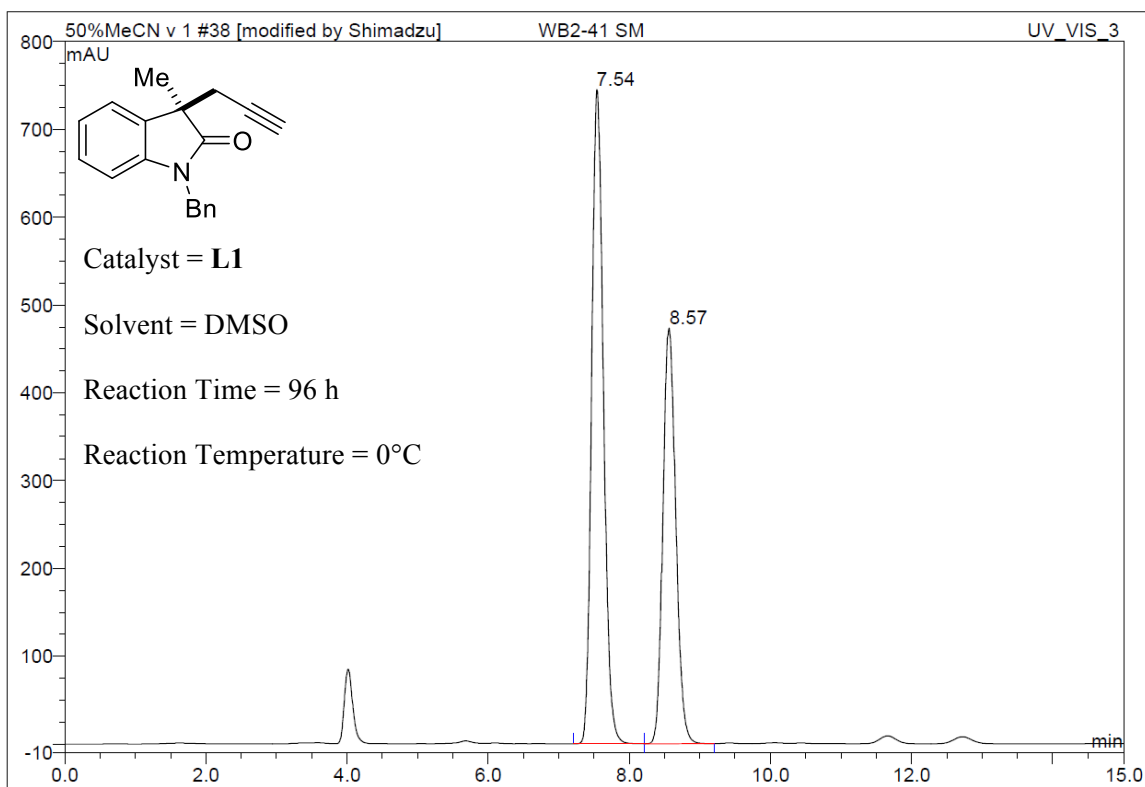
Sample Name:	WB2-39 SM	Injection Volume:	10.0
Vial Number:	1_8	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 17:42	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	1292.113	255.822	50.39	n.a.	BMB
2	8.54	n.a.	1148.354	251.902	49.61	n.a.	BMB
Total:			2440.467	507.724	100.00	0.000	

38 WB2-41 SM**Cell-3 50% MeCN 50% water, 1ml/min**

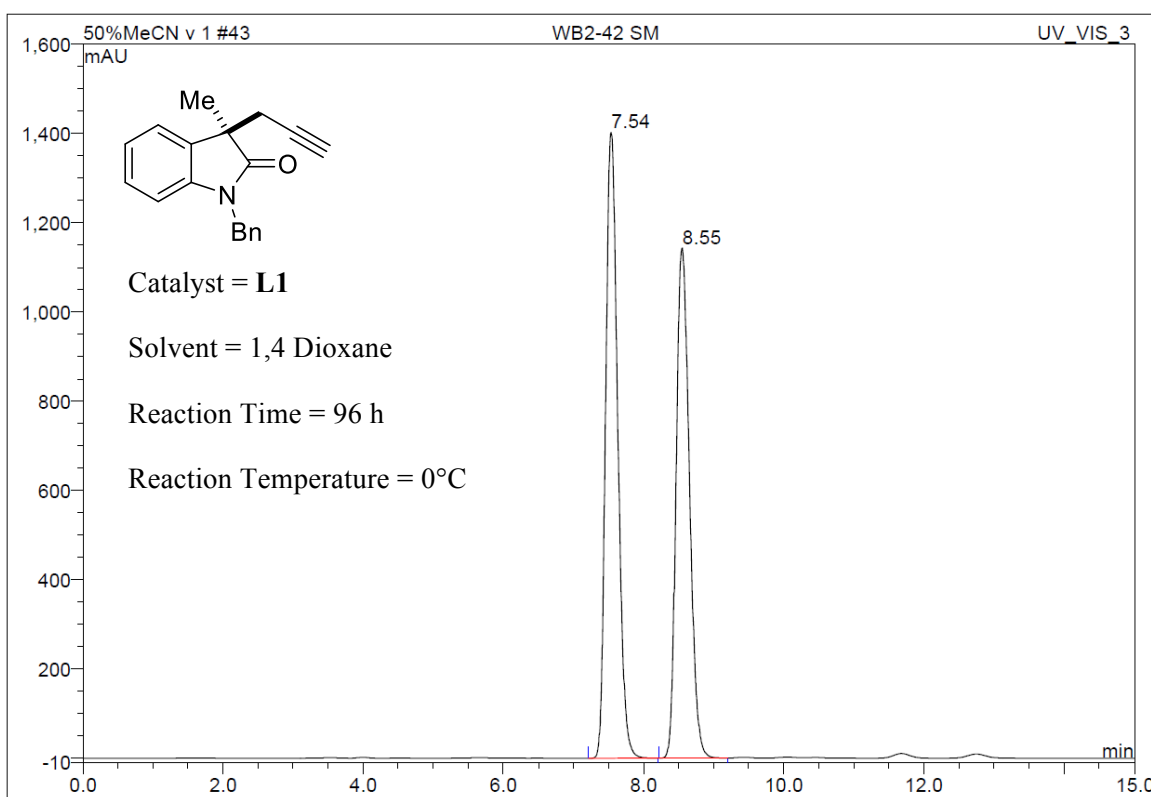
Sample Name:	WB2-41 SM	Injection Volume:	10.0
Vial Number:	1_5	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 16:56	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	744.701	144.062	58.88	n.a.	BMB
2	8.57	n.a.	473.317	100.598	41.12	n.a.	BMB
Total:			1218.018	244.660	100.00	0.000	

43 WB2-42 SM**Cell-3 50% MeCN 50% water, 1ml/min**

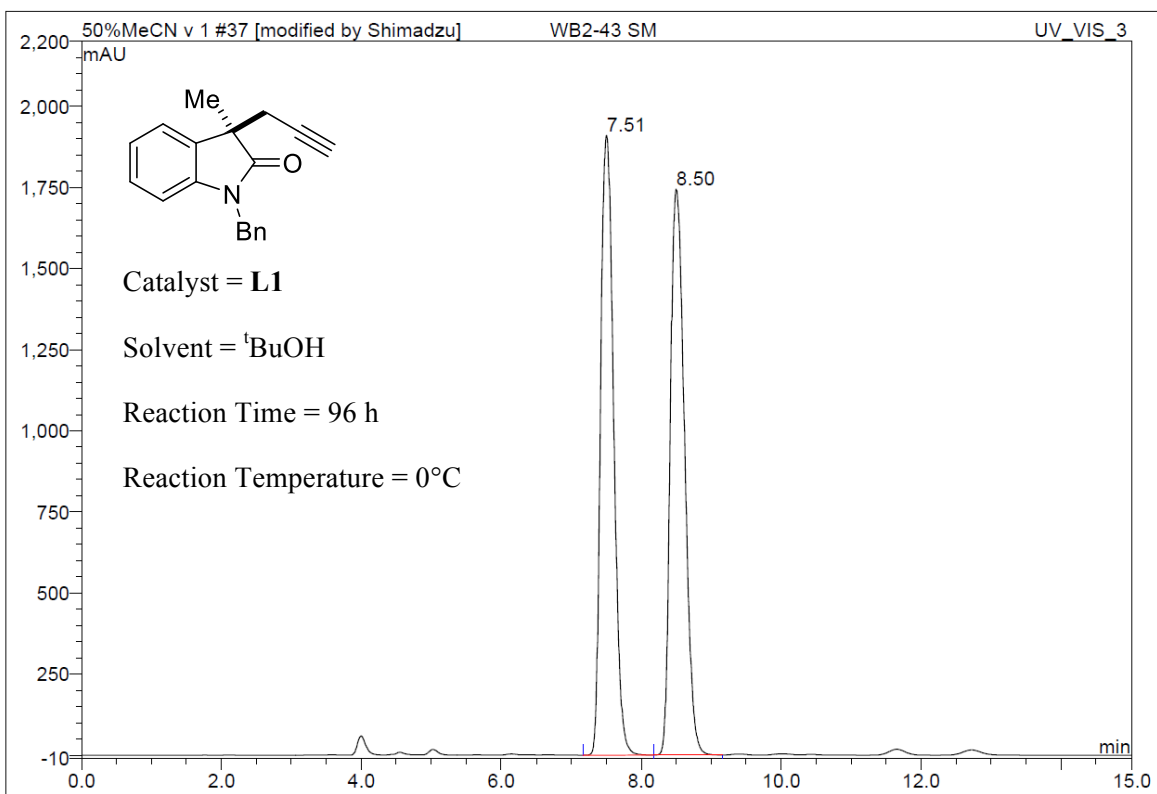
Sample Name:	WB2-42 SM	Injection Volume:	10.0
Vial Number:	1_10	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 18:13	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	1402.586	279.975	52.77	n.a.	BMB
2	8.55	n.a.	1143.103	250.542	47.23	n.a.	BMB
Total:			2545.689	530.518	100.00	0.000	

37 WB2-43 SM**Cell-3 50% MeCN 50% water, 1ml/min**

Sample Name:	WB2-43 SM	Injection Volume:	10.0
Vial Number:	1_4	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 16:40	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000

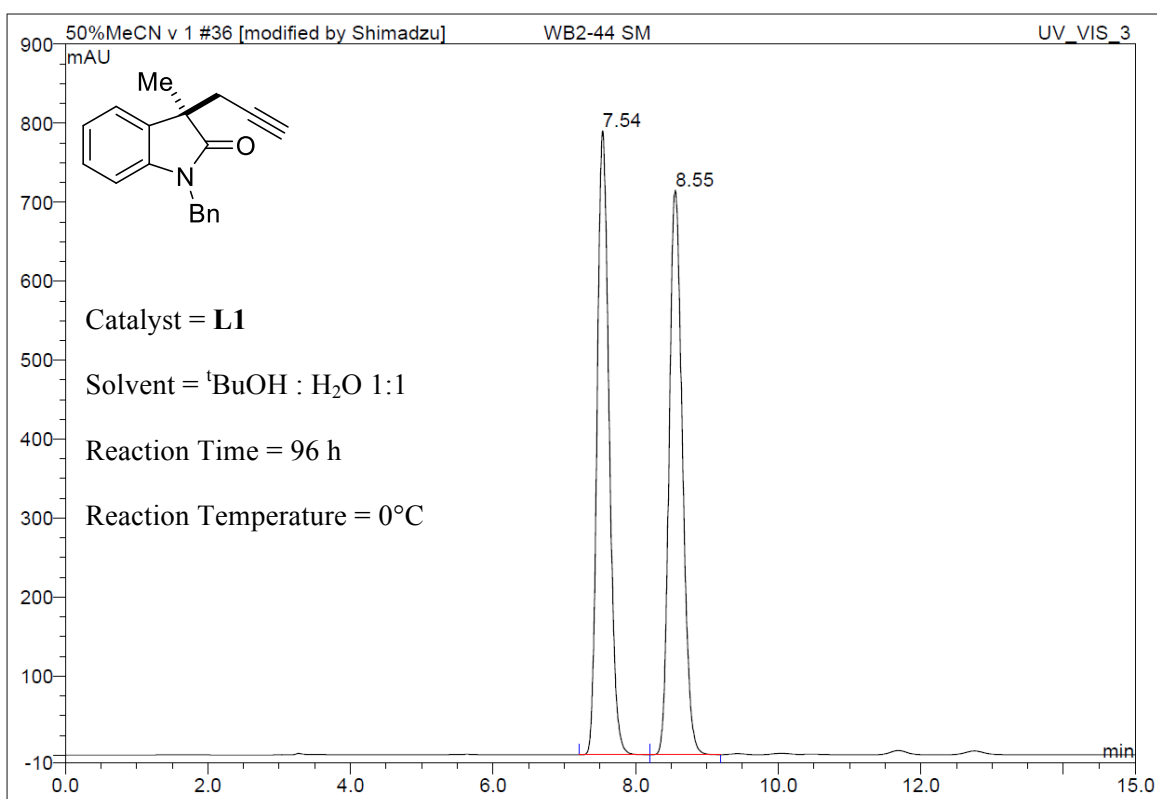


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.51	n.a.	1909.535	410.489	49.99	n.a.	BMB
2	8.50	n.a.	1743.787	410.719	50.01	n.a.	BMB
Total:			3653.322	821.208	100.00	0.000	

36 WB2-44 SM**Cell-3 50% MeCN 50% water, 1ml/min**

Sample Name: **WB2-44 SM**
Vial Number: **1_3**
Sample Type: **unknown**
Control Program: **50% MeCN v 1**
Quantif. Method: **50% MeCN v 1**
Recording Time: **28/8/2014 16:25**
Run Time (min): **15.01**

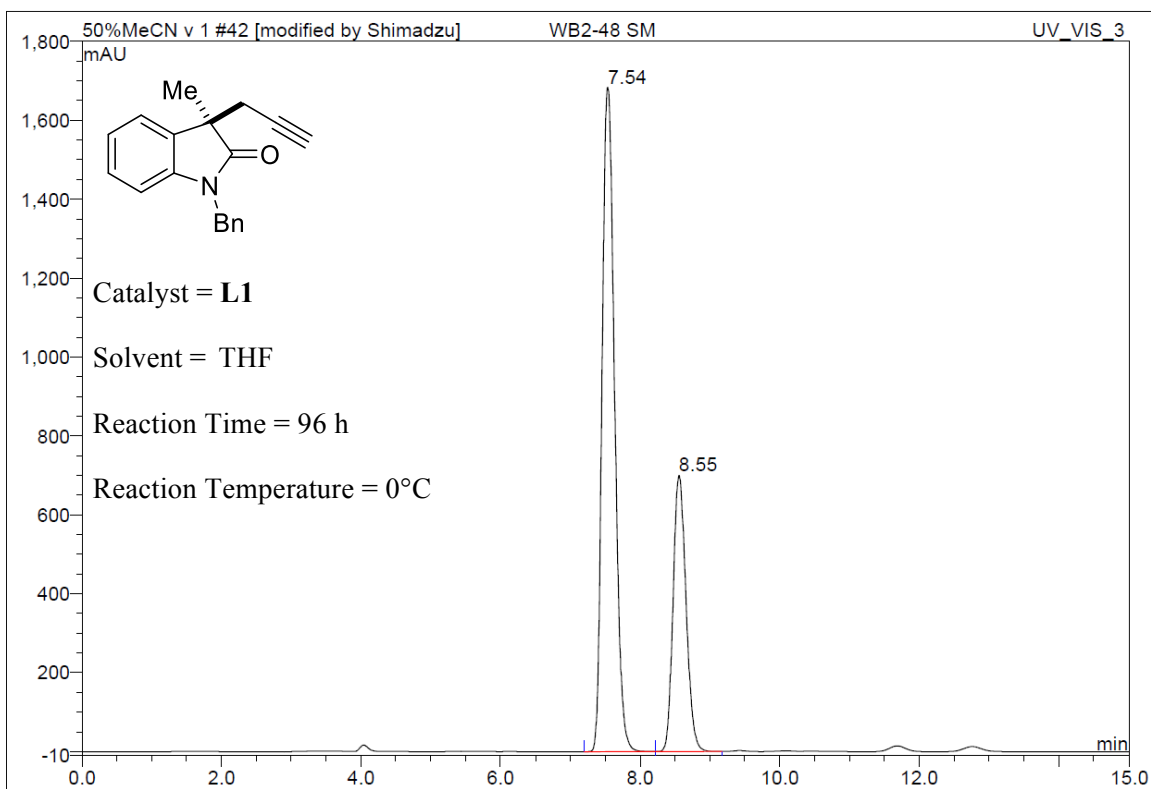
Injection Volume: **10.0**
Channel: **UV_VIS_3**
Wavelength: **n.a.**
Bandwidth: **n.a.**
Dilution Factor: **1.0000**
Sample Weight: **1.0000**
Sample Amount: **1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	789.896	153.679	49.93	n.a.	BM
2	8.55	n.a.	714.878	154.083	50.07	n.a.	MB
Total:			1504.774	307.762	100.00	0.000	

42 WB2-48 SM**Cell-3 50% MeCN 50% water, 1ml/min**

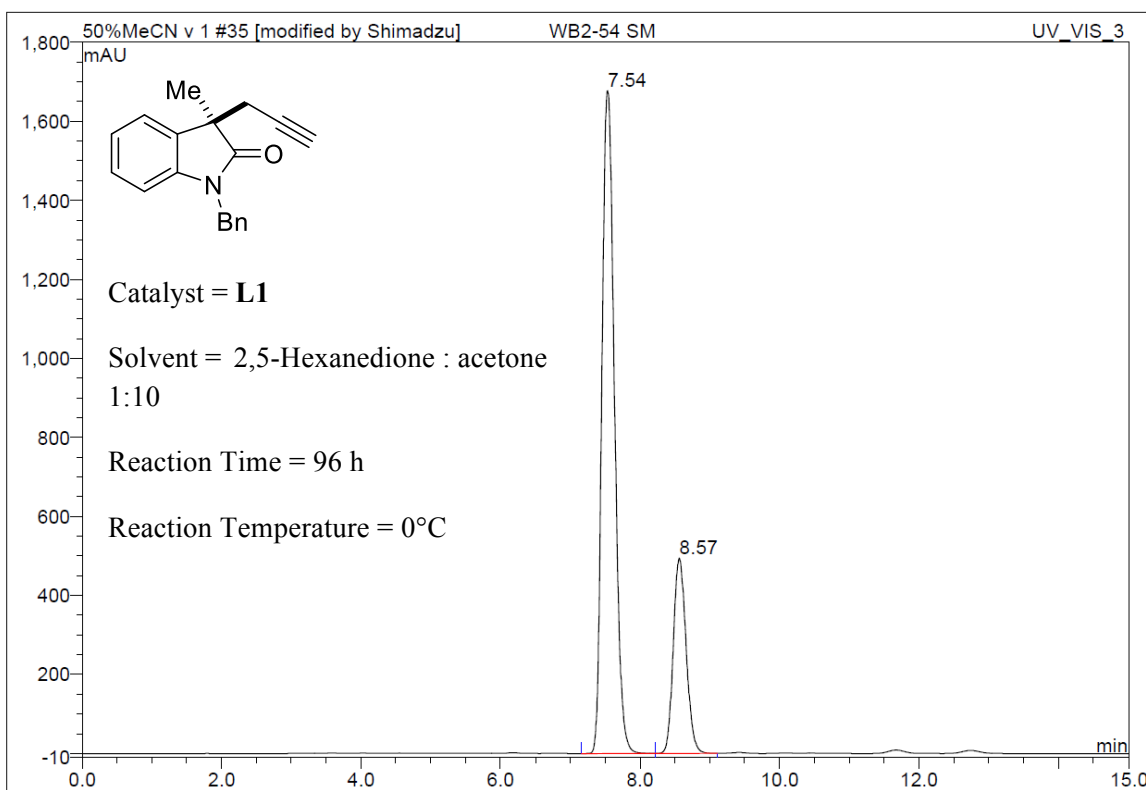
Sample Name:	WB2-48 SM	Injection Volume:	10.0
Vial Number:	1_9	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 17:58	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	1683.354	347.246	69.75	n.a.	BM
2	8.55	n.a.	699.790	150.579	30.25	n.a.	MB
Total:			2383.144	497.824	100.00	0.000	

35 WB2-54 SM**Cell-3 50% MeCN 50% water, 1ml/min**

Sample Name:	WB2-54 SM	Injection Volume:	10.0
Vial Number:	1_2	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 16:09	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000

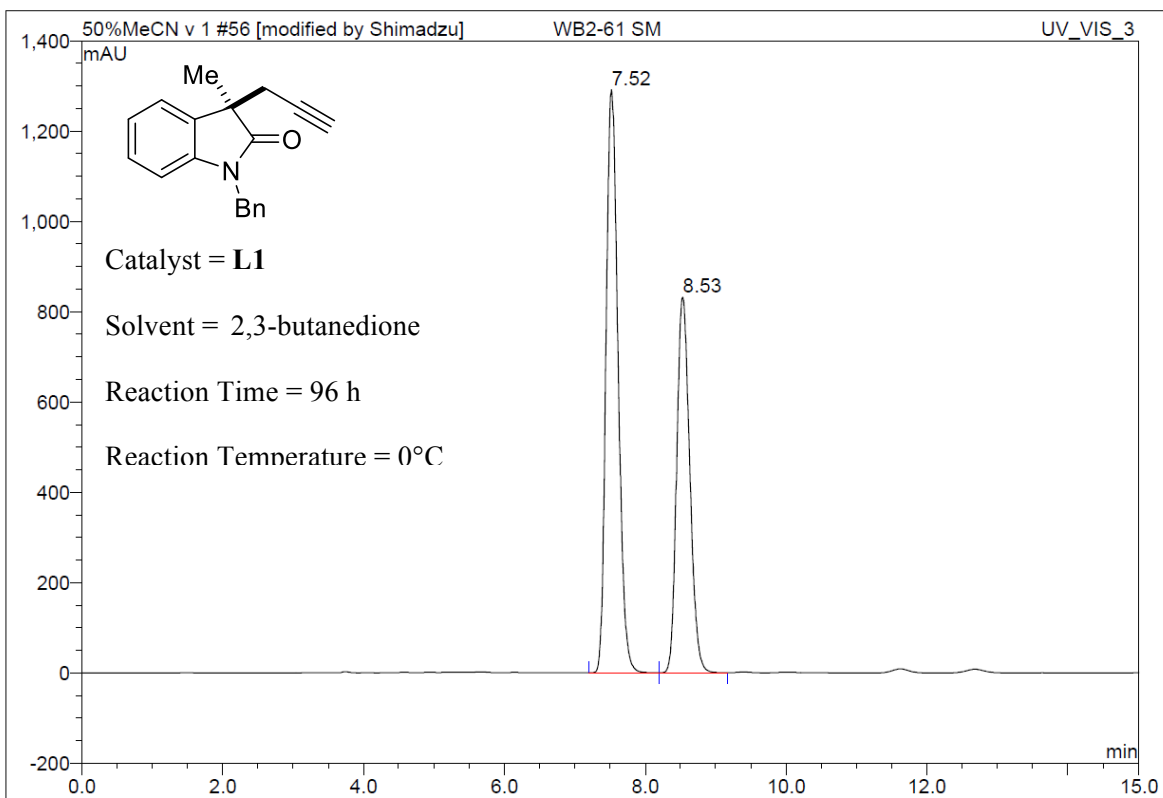


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	1677.147	346.774	76.67	n.a.	BM
2	8.57	n.a.	494.037	105.495	23.33	n.a.	MB
Total:			2171.184	452.270	100.00	0.000	

56 WB2-61 SM**Cell-3 50% MeCN 50% water, 1ml/min**

Sample Name: **WB2-61 SM**
Vial Number: **1_23**
Sample Type: **unknown**
Control Program: **50% MeCN v 1**
Quantif. Method: **50% MeCN v 1**
Recording Time: **28/8/2014 21:34**
Run Time (min): **15.01**

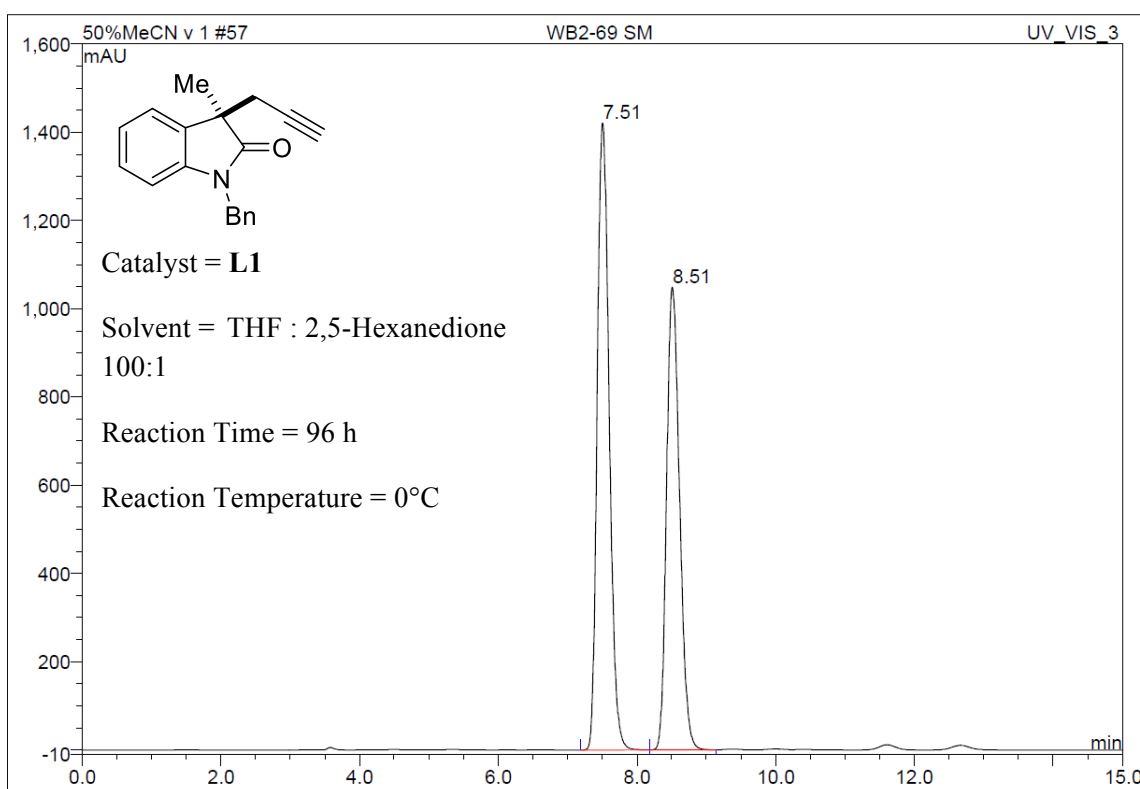
Injection Volume: **10.0**
Channel: **UV_VIS_3**
Wavelength: **n.a.**
Bandwidth: **n.a.**
Dilution Factor: **1.0000**
Sample Weight: **1.0000**
Sample Amount: **1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.52	n.a.	1291.017	254.967	58.77	n.a.	BMB
2	8.53	n.a.	832.046	178.870	41.23	n.a.	BMB
Total:			2123.063	433.837	100.00	0.000	

57 WB2-69 SM**Cell-3 50% MeCN 50% water, 1ml/min**

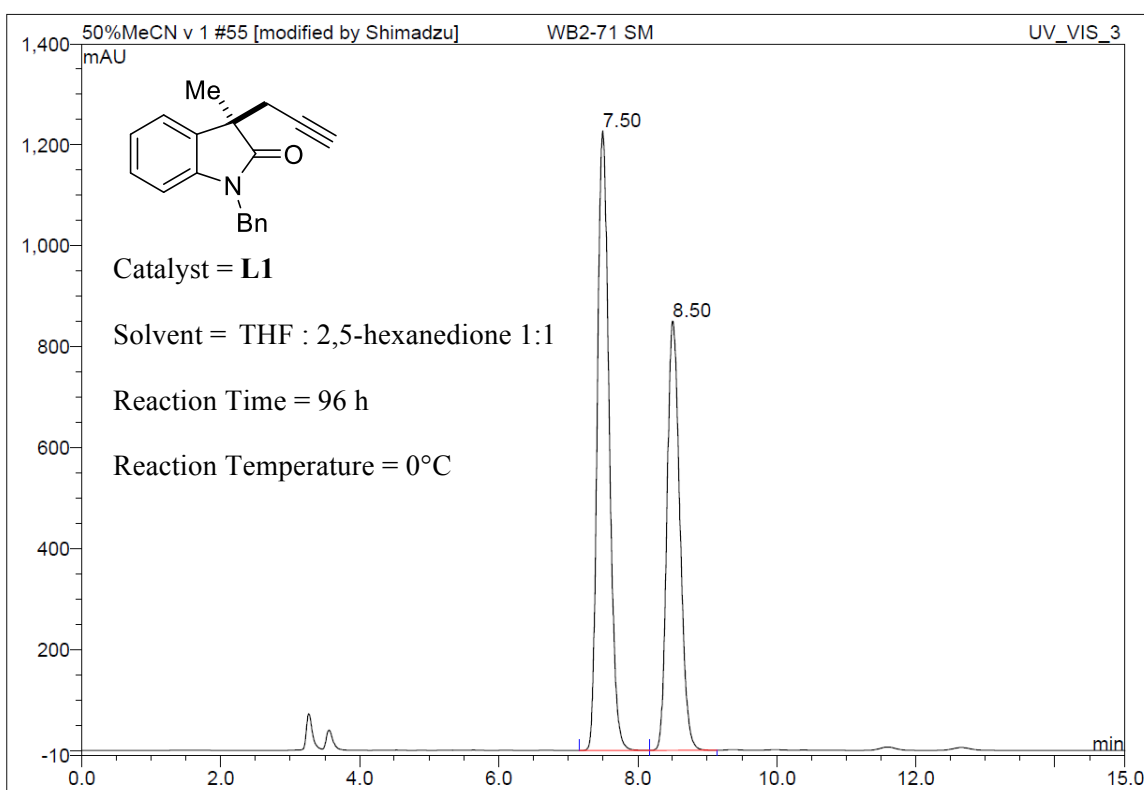
Sample Name:	WB2-69 SM	Injection Volume:	10.0
Vial Number:	1_24	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 21:49	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.51	n.a.	1421.375	283.815	55.45	n.a.	BM
2	8.51	n.a.	1049.278	228.023	44.55	n.a.	MB
Total:			2470.653	511.838	100.00	0.000	

55 WB2-71 SM**Cell-3 50% MeCN 50% water, 1ml/min**

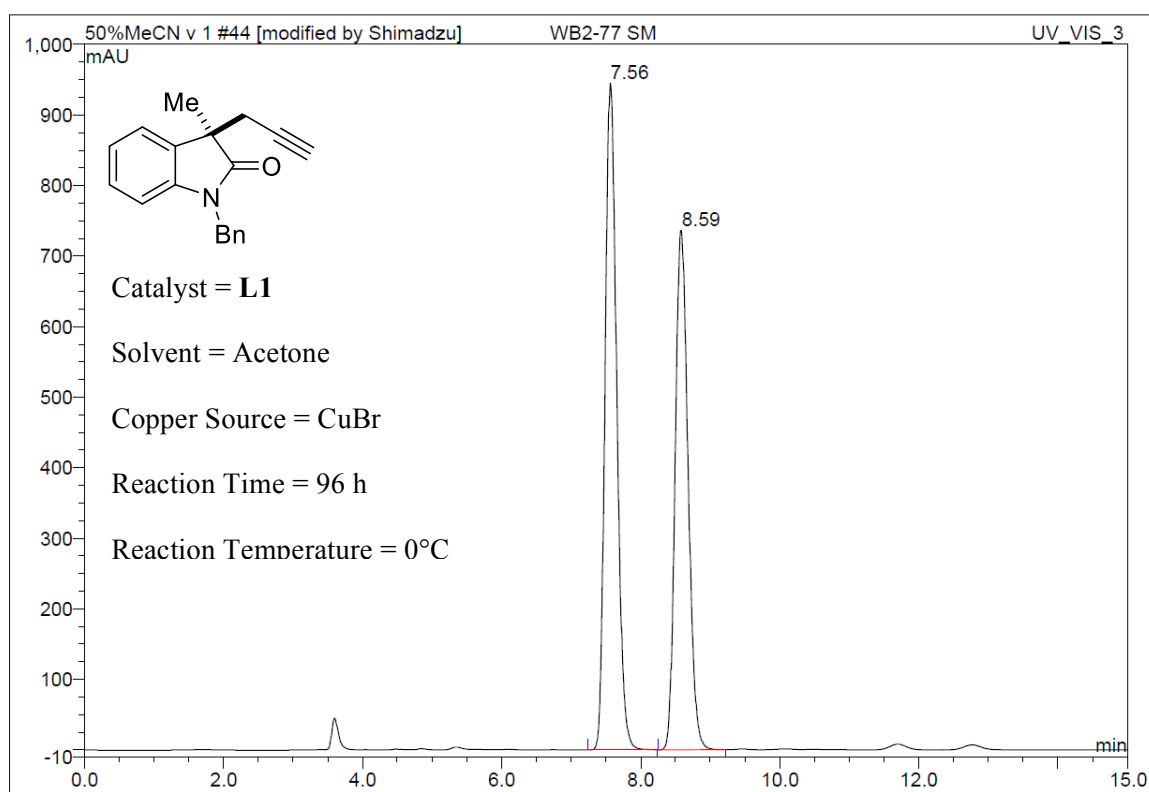
Sample Name:	WB2-71 SM	Injection Volume:	10.0
Vial Number:	1_22	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 21:18	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.50	n.a.	1227.367	241.604	56.90	n.a.	BM
2	8.50	n.a.	850.574	182.997	43.10	n.a.	MB
Total:			2077.941	424.601	100.00	0.000	

44 WB2-77 SM**Cell-3 50% MeCN 50% water, 1ml/min**

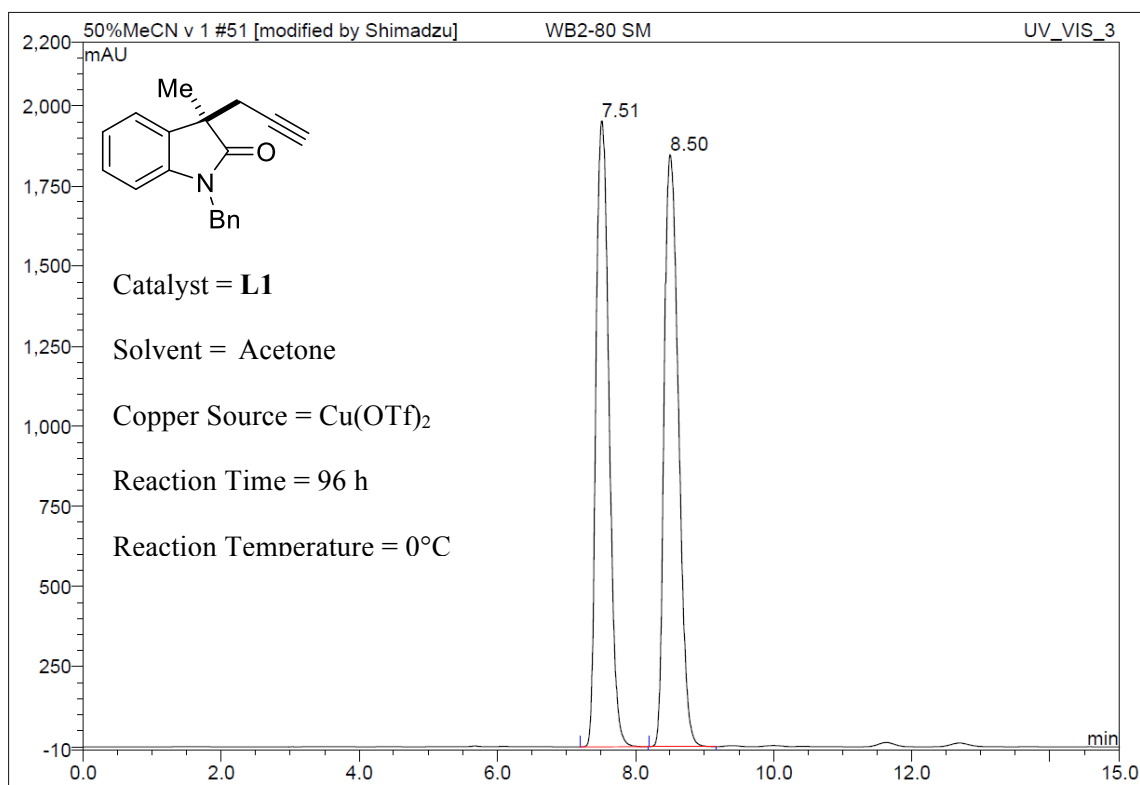
Sample Name:	WB2-77 SM	Injection Volume:	10.0
Vial Number:	1_11	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 18:28	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.56	n.a.	944.956	184.013	53.77	n.a.	BMB
2	8.59	n.a.	736.171	158.200	46.23	n.a.	BMB
Total:			1681.127	342.213	100.00	0.000	

51 WB2-80 SM**Cell-3 50% MeCN 50% water, 1ml/min**

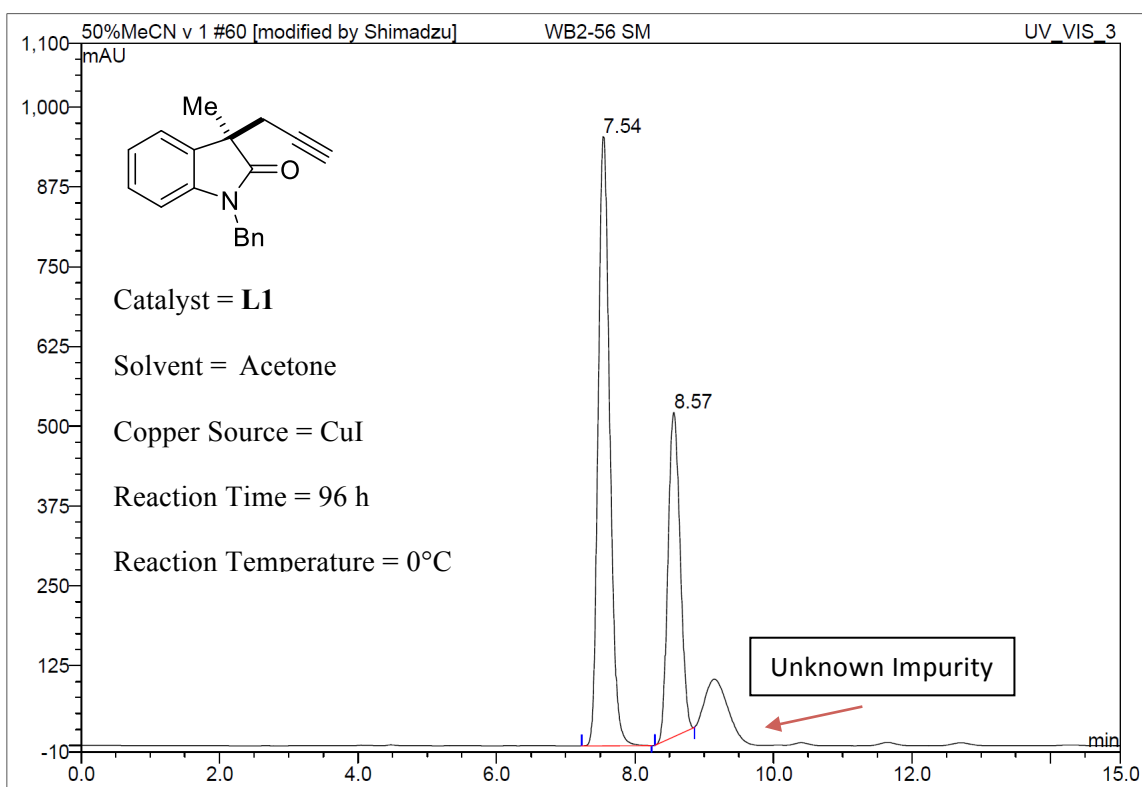
Sample Name:	WB2-80 SM	Injection Volume:	10.0
Vial Number:	1_18	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 20:17	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.51	n.a.	1952.662	425.449	48.84	n.a.	BMB
2	8.50	n.a.	1846.541	445.603	51.16	n.a.	BMB
Total:			3799.203	871.052	100.00	0.000	

60 WB2-56 SM**Cell-3 50% MeCN 50% water, 1ml/min**

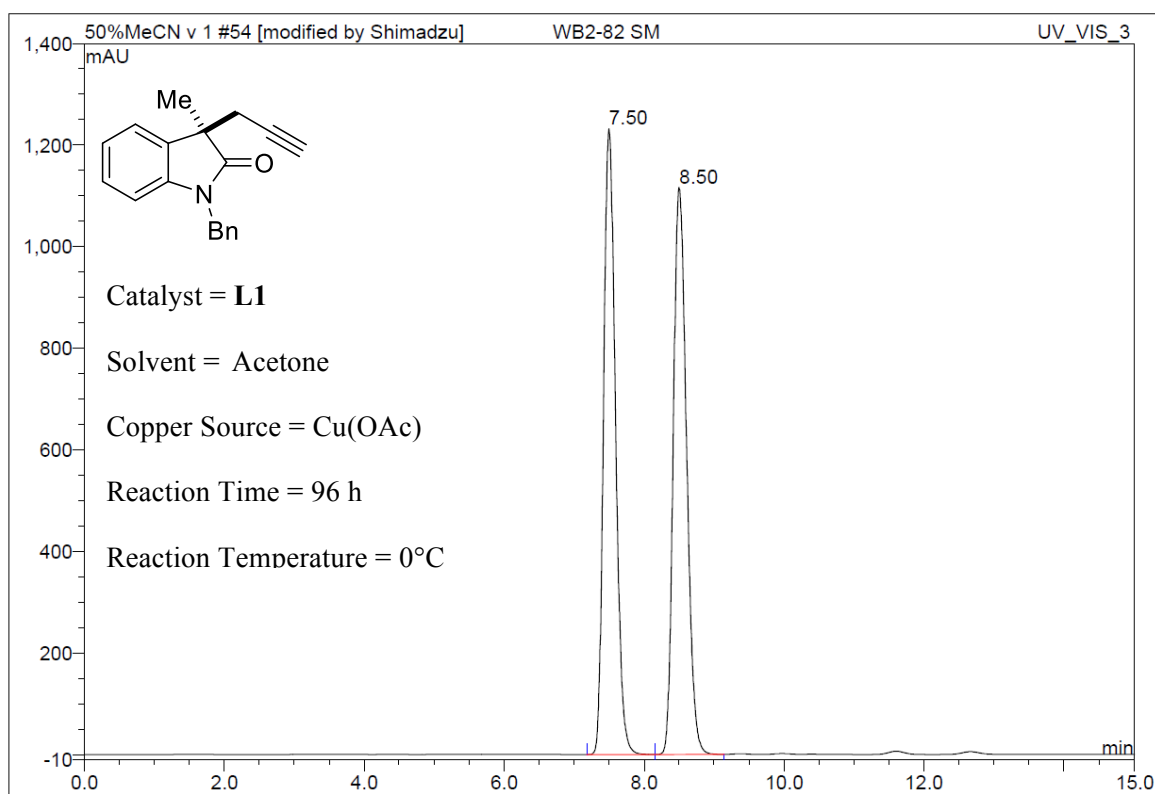
Sample Name:	WB2-56 SM	Injection Volume:	10.0
Vial Number:	1_9	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	29/8/2014 15:52	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	954.444	185.554	64.23	n.a.	BMB
2	8.57	n.a.	508.033	103.341	35.77	n.a.	BMB*
Total:			1462.477	288.895	100.00	0.000	

54 WB2-82 SM**Cell-3 50% MeCN 50% water, 1ml/min**

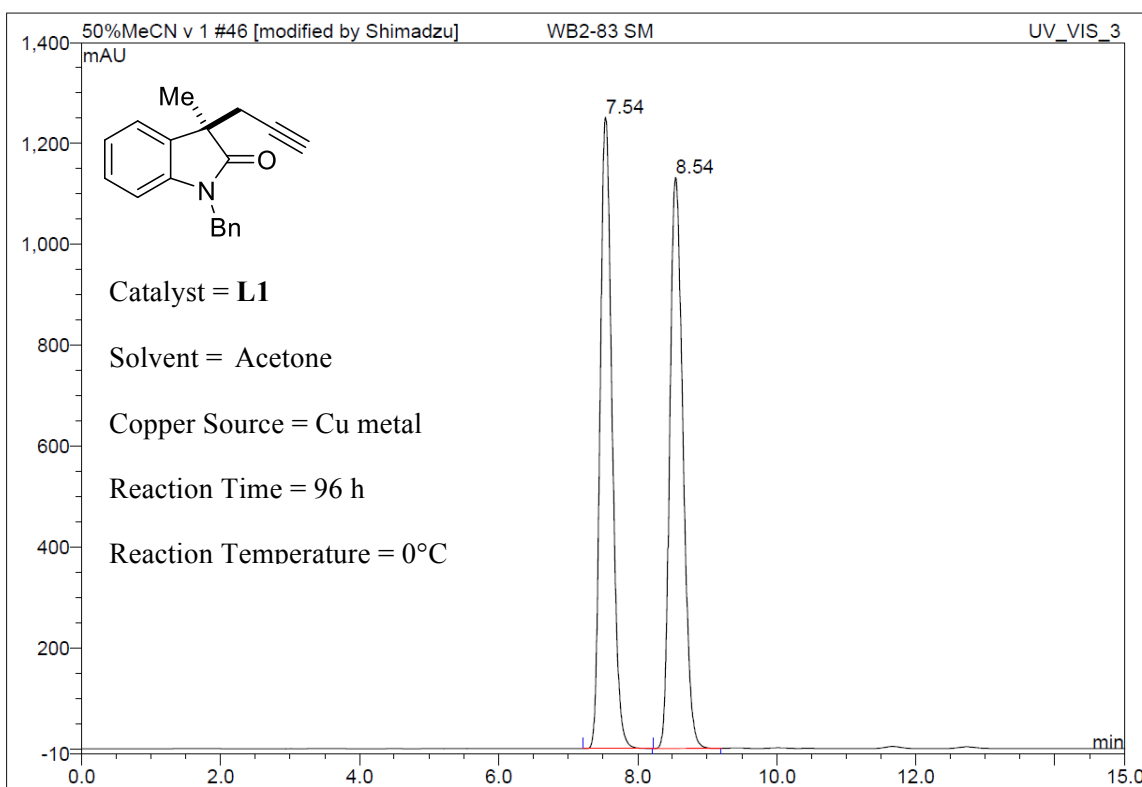
Sample Name:	WB2-82 SM	Injection Volume:	10.0
Vial Number:	1_21	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 21:03	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.50	n.a.	1231.918	242.361	49.84	n.a.	BM
2	8.50	n.a.	1116.686	243.884	50.16	n.a.	MB
Total:			2348.604	486.245	100.00	0.000	

46 WB2-83 SM**Cell-3 50% MeCN 50% water, 1ml/min**

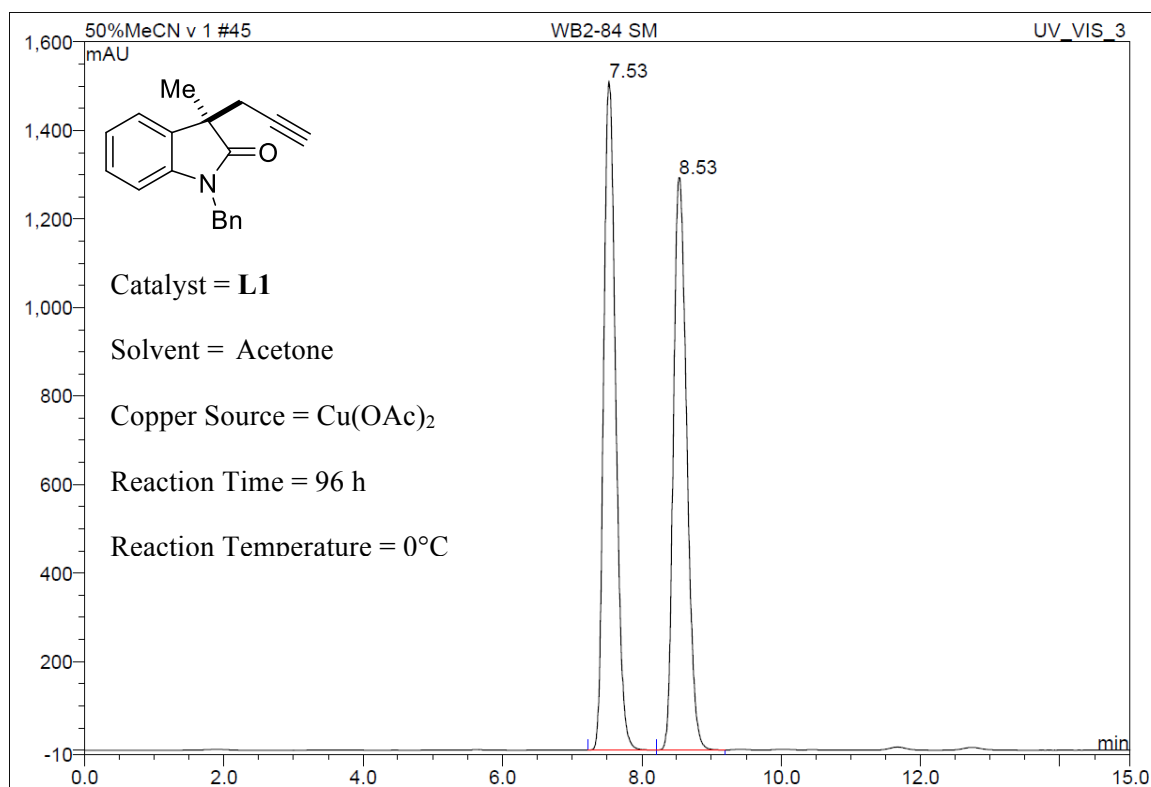
Sample Name:	WB2-83 SM	Injection Volume:	10.0
Vial Number:	1_13	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 18:59	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.54	n.a.	1251.774	247.277	49.94	n.a.	BMB
2	8.54	n.a.	1131.994	247.847	50.06	n.a.	BMB
Total:			2383.768	495.125	100.00	0.000	

45 WB2-84 SM**Cell-3 50% MeCN 50% water, 1ml/min**

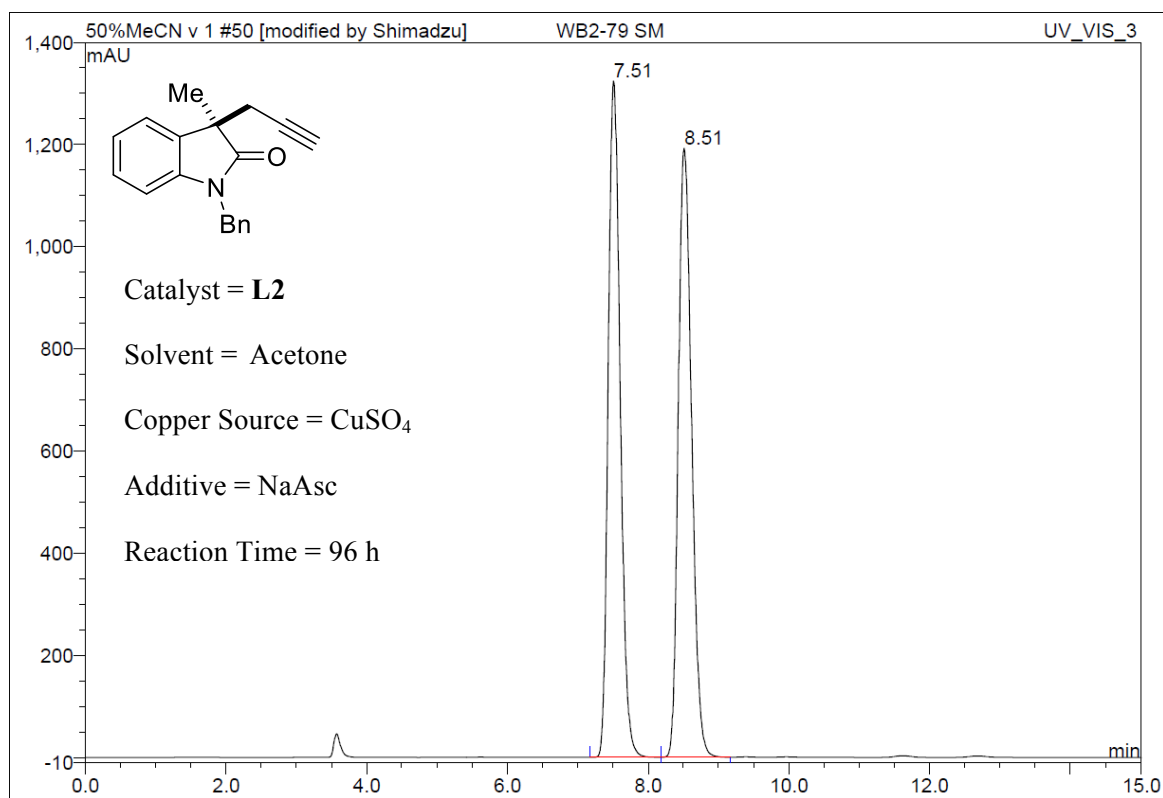
Sample Name:	WB2-84 SM	Injection Volume:	10.0
Vial Number:	1_12	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 18:44	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.53	n.a.	1511.468	303.294	51.37	n.a.	BMB
2	8.53	n.a.	1293.097	287.101	48.63	n.a.	BMB
Total:			2804.565	590.395	100.00	0.000	

50 WB2-79 SM**Cell-3 50% MeCN 50% water, 1ml/min**

Sample Name:	WB2-79 SM	Injection Volume:	10.0
Vial Number:	1_17	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	28/8/2014 20:01	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000

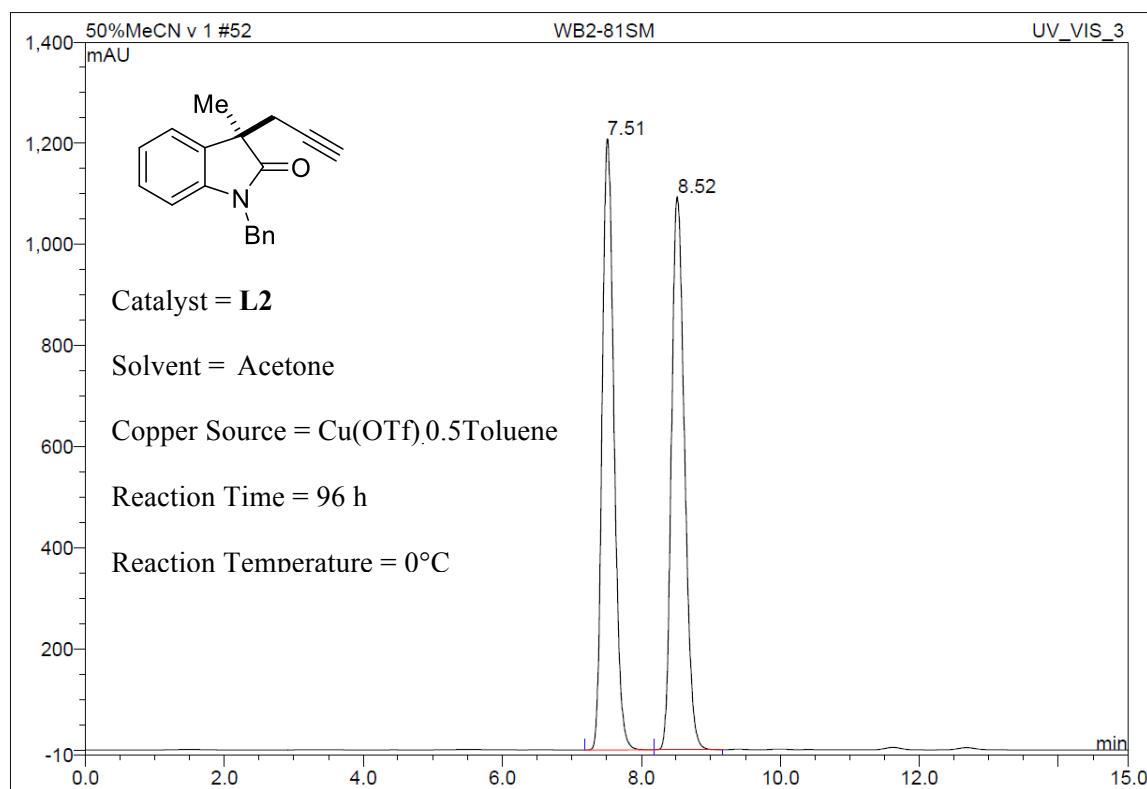


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.51	n.a.	1324.159	262.107	50.03	n.a.	BM
2	8.51	n.a.	1192.548	261.744	49.97	n.a.	MB
Total:			2516.707	523.851	100.00	0.000	

52 WB2-81SM**Cell-3 50% MeCN 50% water, 1ml/min**

Sample Name: **WB2-81SM**
Vial Number: **1_19**
Sample Type: **unknown**
Control Program: **50% MeCN v 1**
Quantif. Method: **50% MeCN v 1**
Recording Time: **28/8/2014 20:32**
Run Time (min): **15.01**

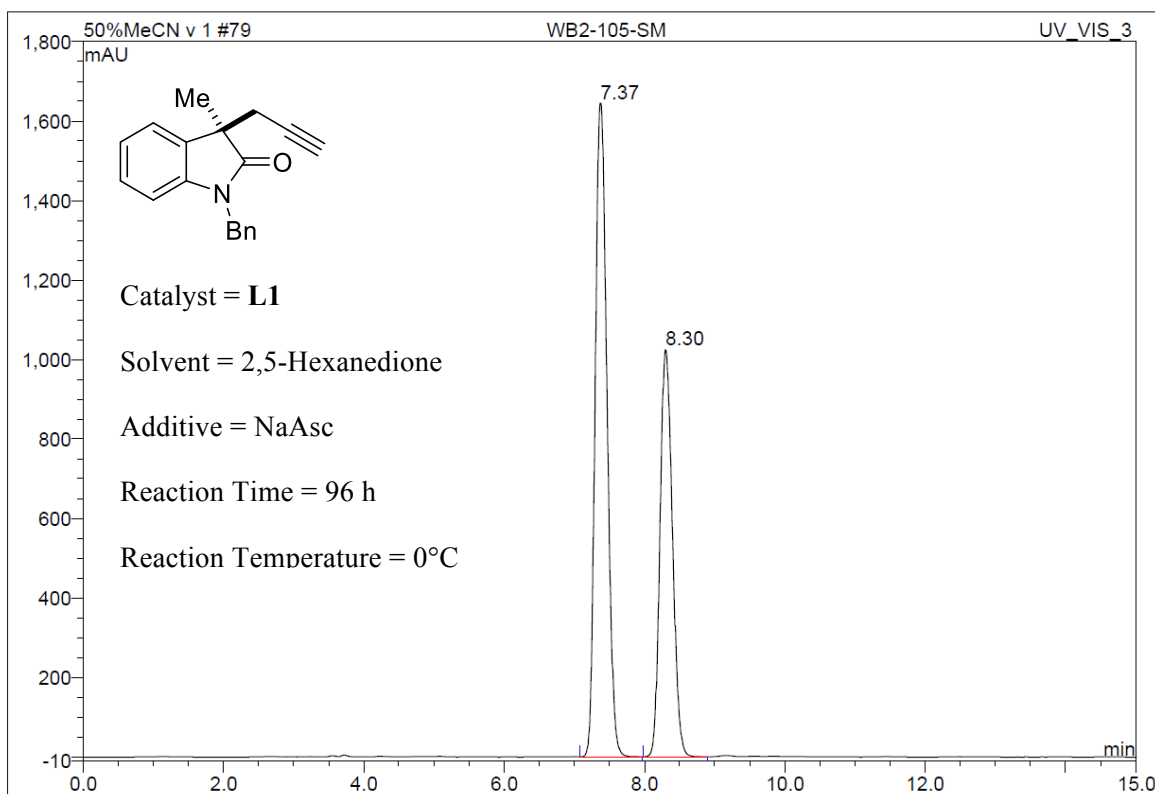
Injection Volume: **10.0**
Channel: **UV_VIS_3**
Wavelength: **n.a.**
Bandwidth: **n.a.**
Dilution Factor: **1.0000**
Sample Weight: **1.0000**
Sample Amount: **1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.51	n.a.	1208.048	237.848	49.91	n.a.	BMB
2	8.52	n.a.	1093.990	238.717	50.09	n.a.	BMB
Total:			2302.038	476.565	100.00	0.000	

79 WB2-105-SM**Cell-3 50% MeCN 50% water, 1ml/min**

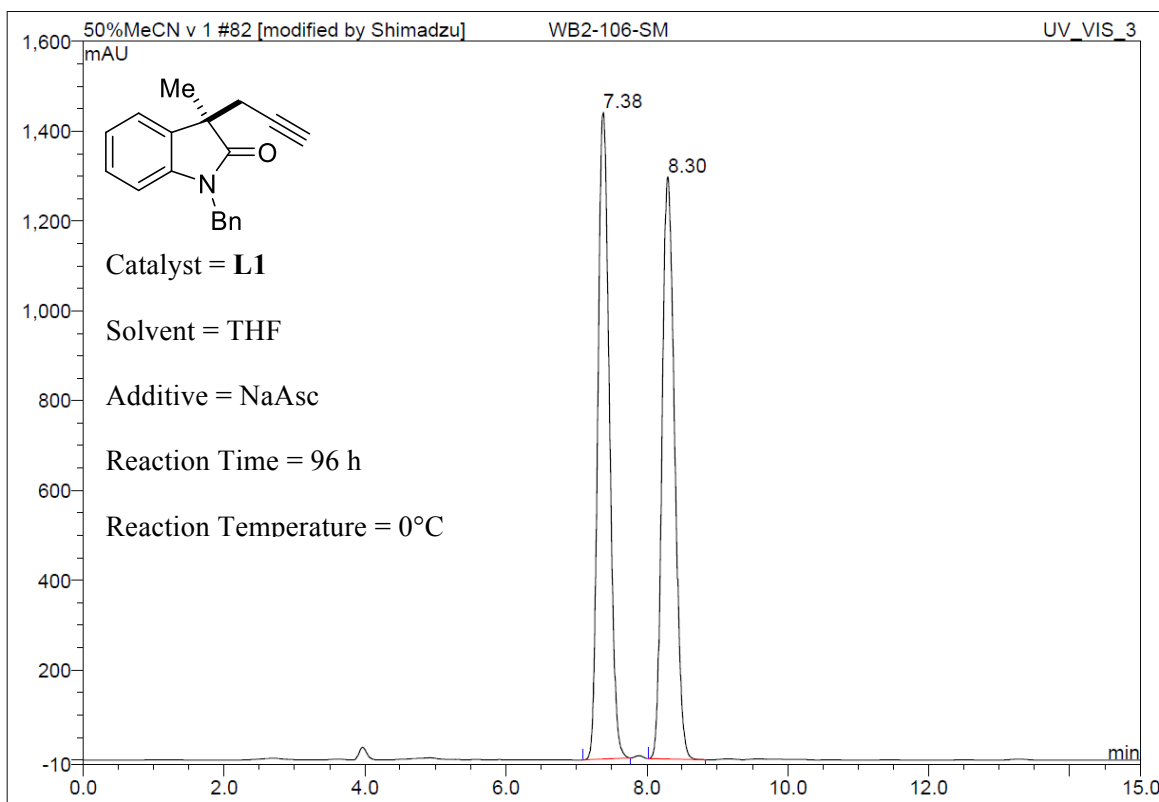
Sample Name:	WB2-105-SM	Injection Volume:	10.0
Vial Number:	1_6	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	25/9/2014 13:11	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.37	n.a.	1645.146	319.812	60.38	n.a.	BMB
2	8.30	n.a.	1024.592	209.880	39.62	n.a.	BMB
Total:			2669.738	529.692	100.00	0.000	

82 WB2-106-SM**Cell-3 50% MeCN 50% water, 1ml/min**

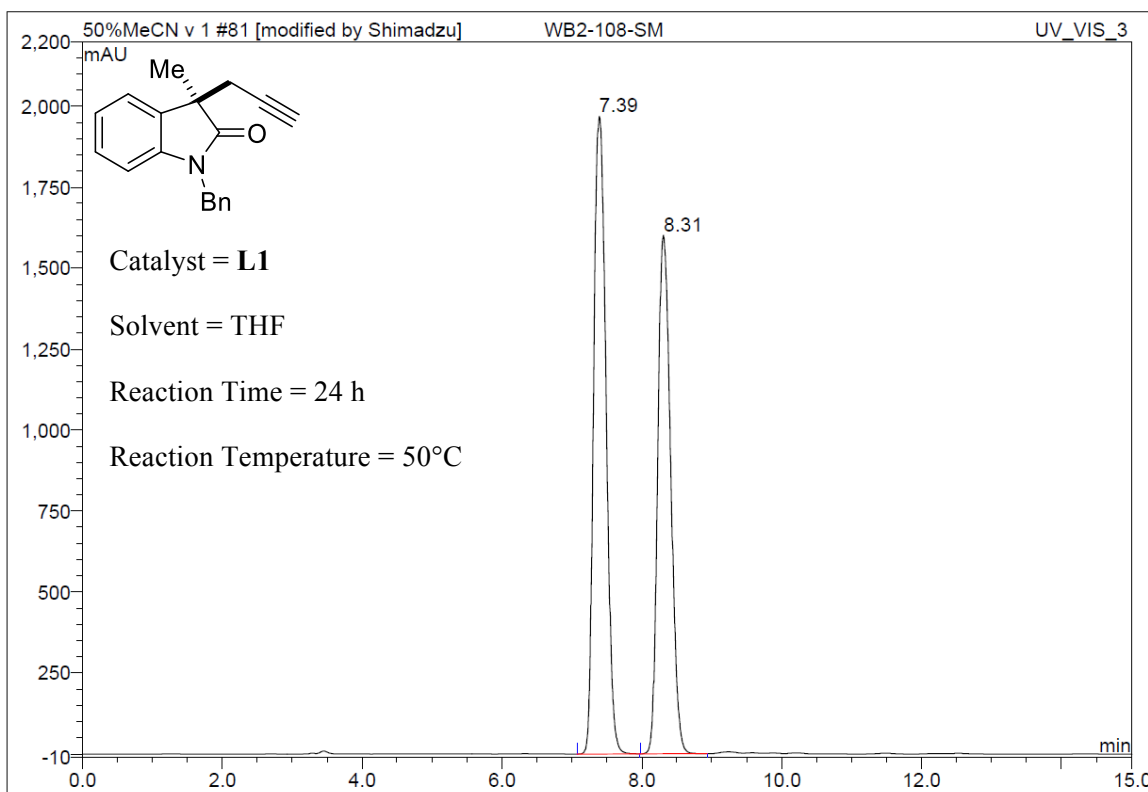
Sample Name:	WB2-106-SM	Injection Volume:	10.0
Vial Number:	1_9	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	25/9/2014 13:57	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.38	n.a.	1440.567	271.121	50.34	n.a.	BMB
2	8.30	n.a.	1296.554	267.461	49.66	n.a.	BMB
Total:			2737.121	538.581	100.00	0.000	

81 WB2-108-SM**Cell-3 50% MeCN 50% water, 1ml/min**

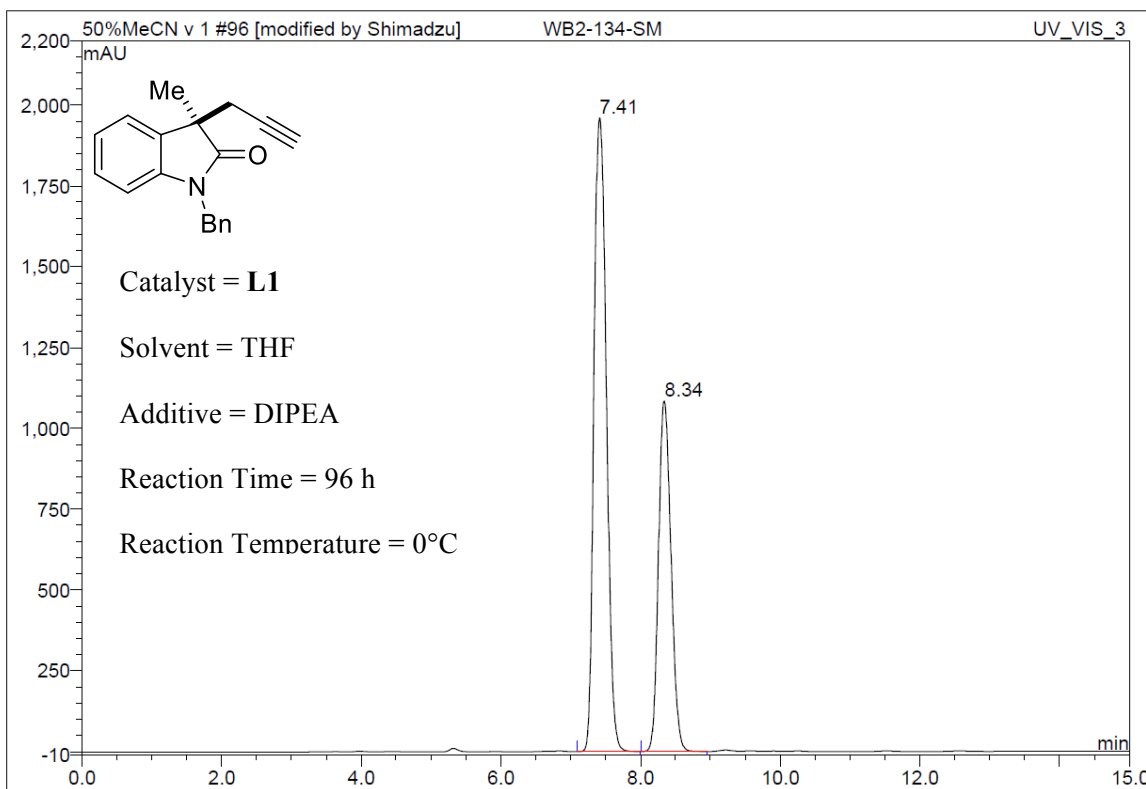
Sample Name:	WB2-108-SM	Injection Volume:	10.0
Vial Number:	1_8	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	25/9/2014 13:42	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.39	n.a.	1968.651	401.712	53.91	n.a.	BMB
2	8.31	n.a.	1600.731	343.426	46.09	n.a.	BMB
Total:			3569.382	745.138	100.00	0.000	

96 WB2-134-SM**Cell-3 50% MeCN 50% water, 1ml/min,**

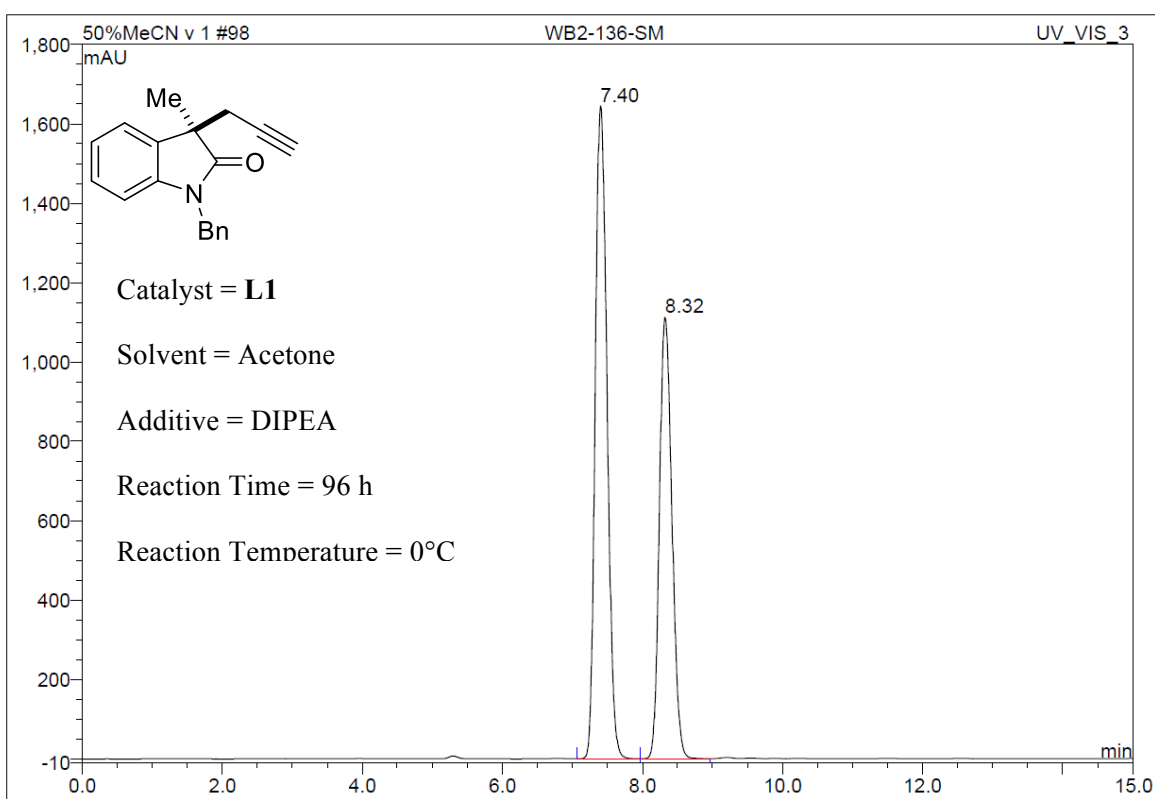
Sample Name:	WB2-134-SM	Injection Volume:	10.0
Vial Number:	1_2	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	14/10/2014 17:28	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.41	n.a.	1959.693	404.546	64.31	n.a.	BMB
2	8.34	n.a.	1084.948	224.517	35.69	n.a.	BMB
Total:			3044.641	629.063	100.00	0.000	

98 WB2-136-SM**Cell-3 50% MeCN 50% water, 1ml/min,**

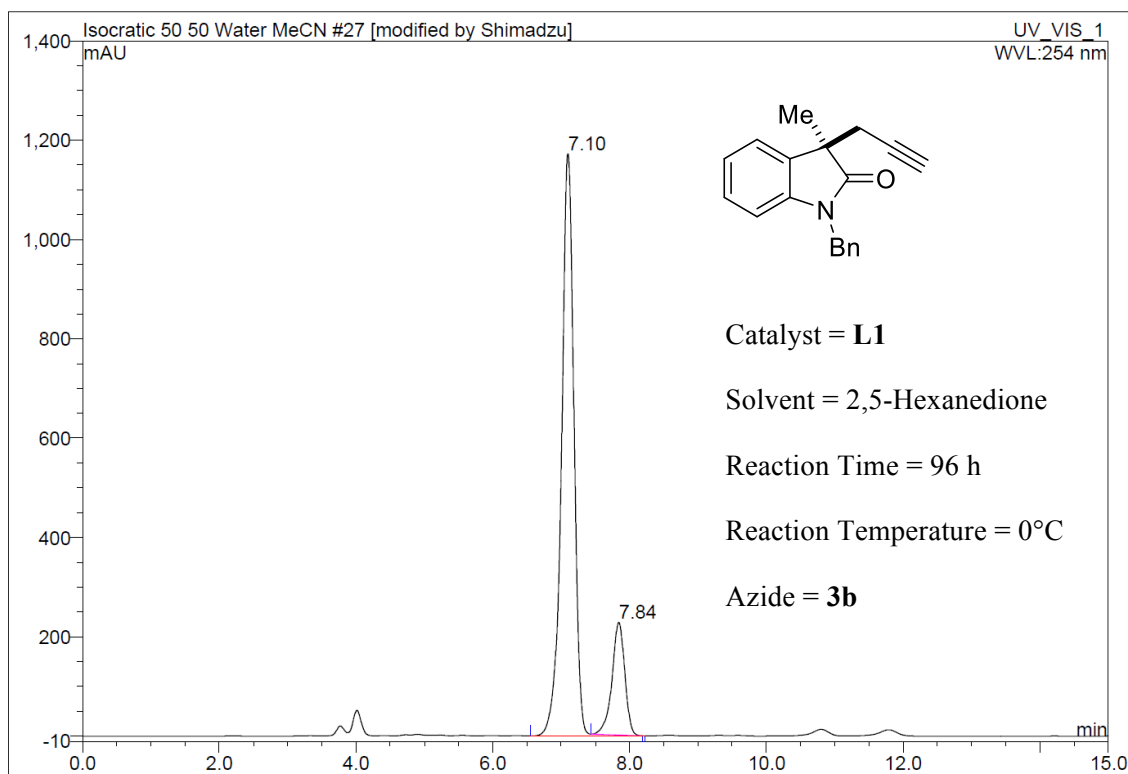
Sample Name:	WB2-136-SM	Injection Volume:	10.0
Vial Number:	1_4	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	n.a.
Control Program:	50% MeCN v 1	Bandwidth:	n.a.
Quantif. Method:	50% MeCN v 1	Dilution Factor:	1.0000
Recording Time:	14/10/2014 17:59	Sample Weight:	1.0000
Run Time (min):	15.01	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.40	n.a.	1645.227	320.375	58.18	n.a.	BM
2	8.32	n.a.	1112.746	230.268	41.82	n.a.	MB
Total:			2757.973	550.643	100.00	0.000	

27 WB3-178 SM**Cell 3, 50% MeCN 50% Water**

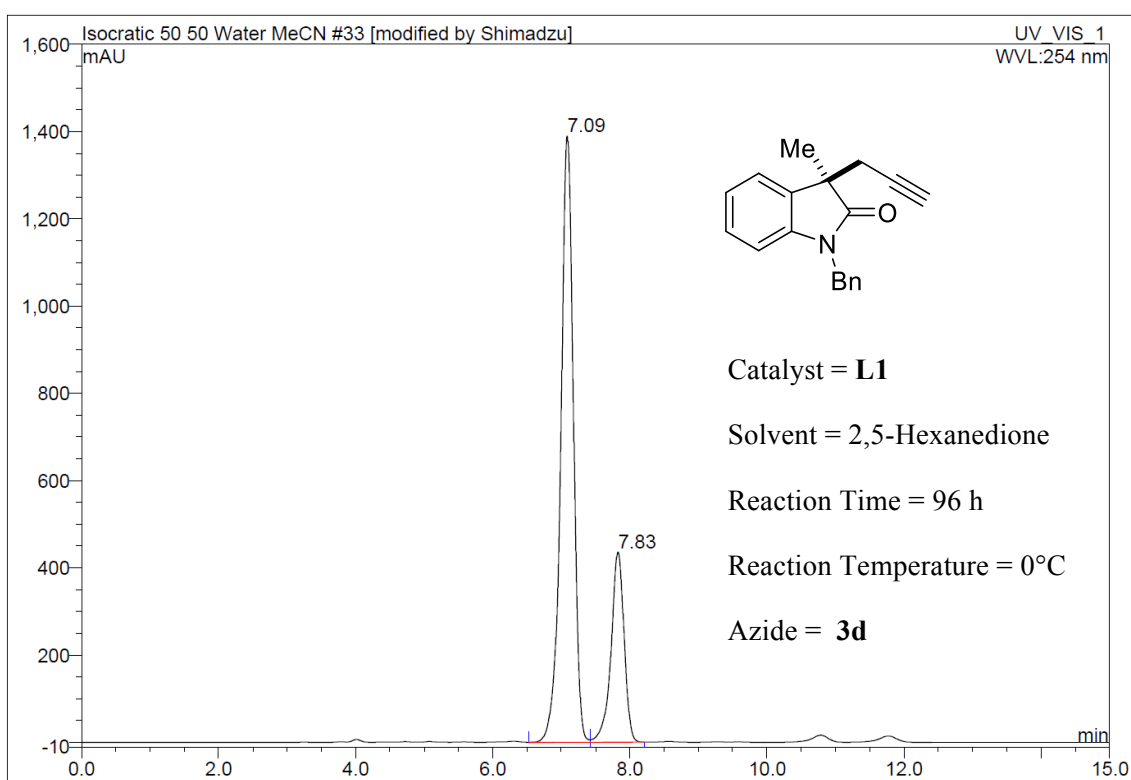
Sample Name:	WB3-178 SM	Injection Volume:	10.0
Vial Number:	1_6	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	254
Control Program:	Isocratic 50 50 water MeCN	Bandwidth:	n.a.
Quantif. Method:	Isocratic 50 50 water MeCN	Dilution Factor:	1.0000
Recording Time:	17/4/2015 18:57	Sample Weight:	1.0000
Run Time (min):	30.00	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.10	n.a.	1171.835	247.375	83.43	n.a.	BMB
2	7.84	n.a.	226.589	49.139	16.57	n.a.	Rd
Total:			1398.424	296.514	100.00	0.000	

33 WB3-184 SM**Cell 3, 50% MeCN 50% Water**

Sample Name:	WB3-184 SM	Injection Volume:	10.0
Vial Number:	1_12	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	254
Control Program:	Isocratic 50 50 water MeCN	Bandwidth:	n.a.
Quantif. Method:	Isocratic 50 50 water MeCN	Dilution Factor:	1.0000
Recording Time:	17/4/2015 21:31	Sample Weight:	1.0000
Run Time (min):	30.00	Sample Amount:	1.0000

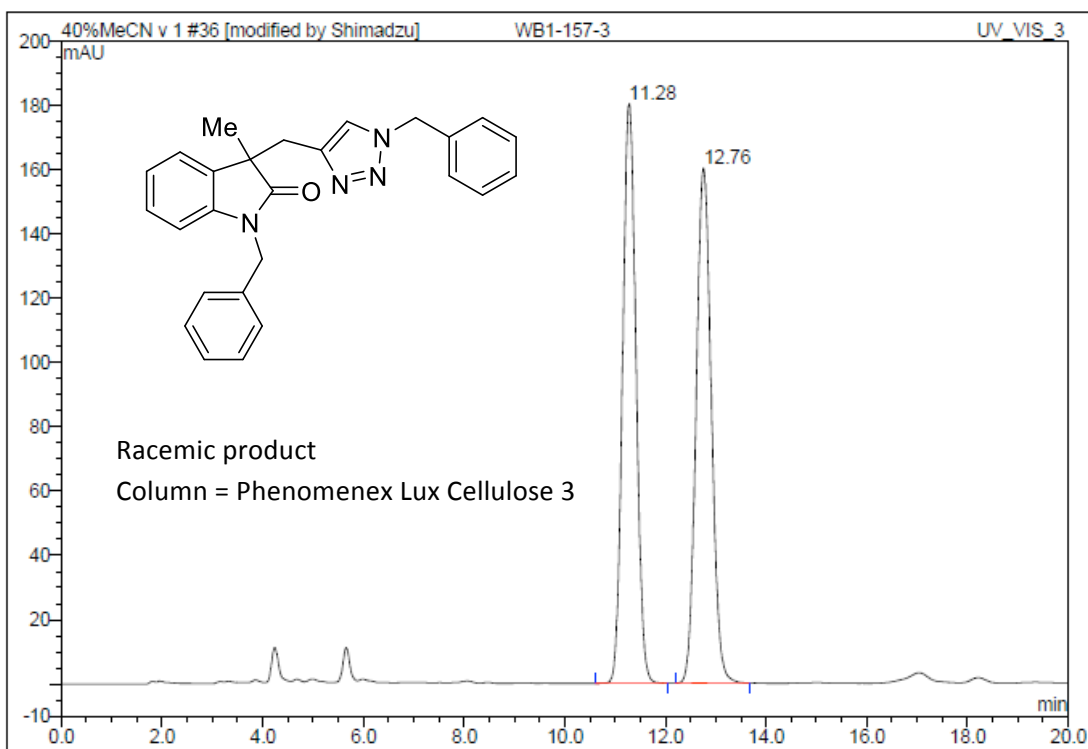


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	7.09	n.a.	1389.235	295.485	75.38	n.a.	BM
2	7.83	n.a.	435.236	96.528	24.62	n.a.	MB
Total:			1824.471	392.012	100.00	0.000	

36 WB1-157-3**Cell-3 40% MeCN 60% water, 1ml/min**

Sample Name: **WB1-157-3**
Vial Number: **1_5**
Sample Type: **unknown**
Control Program: **40% MeCN v 1**
Quantif. Method: **40% MeCN v 1**
Recording Time: **28/8/2014 15:00**
Run Time (min): **27.18**

Injection Volume: **10.0**
Channel: **UV_VIS_3**
Wavelength: **n.a.**
Bandwidth: **n.a.**
Dilution Factor: **1.0000**
Sample Weight: **1.0000**
Sample Amount: **1.0000**

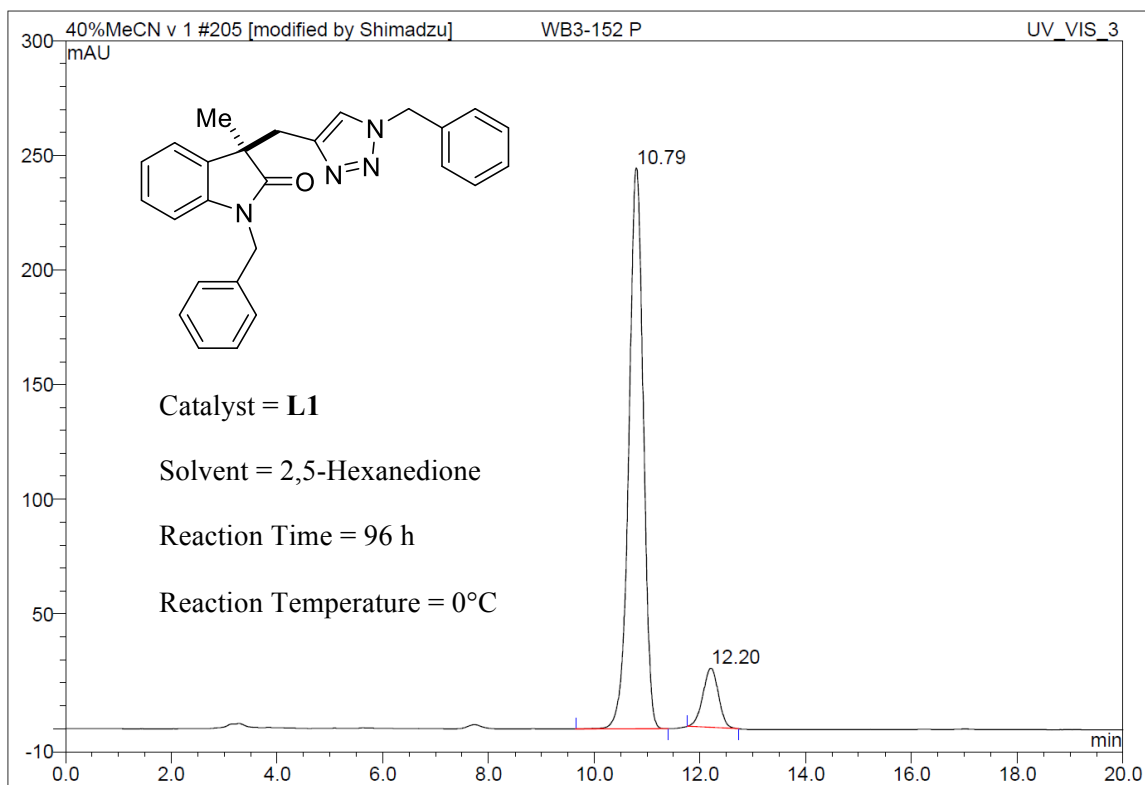


No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	11.28	n.a.	180.202	55.610	49.83	n.a.	BMB
2	12.76	n.a.	160.133	55.988	50.17	n.a.	BMB
Total:			340.335	111.598	100.00	0.000	

205 WB3-152 P**40% MeCN 60% water Cell-3, 1ml/min**

Sample Name: **WB3-152 P**
Vial Number: **1_9**
Sample Type: **unknown**
Control Program: **40% MeCN v 1**
Quantif. Method: **40% MeCN v 1**
Recording Time: **31/3/2015 15:12**
Run Time (min): **30.01**

Injection Volume: **10.0**
Channel: **UV_VIS_3**
Wavelength: **n.a.**
Bandwidth: **n.a.**
Dilution Factor: **1.0000**
Sample Weight: **1.0000**
Sample Amount: **1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	10.79	n.a.	244.634	77.375	89.78	n.a.	BMB
2	12.20	n.a.	25.753	8.810	10.22	n.a.	BMB*
Total:			270.387	86.184	100.00	0.000	

References

- (1) Rossy, C.; Majimel, J.; Delapierre, M. T.; Fouquet, E.; Felpin, F.-X. *J. Organomet. Chem.* **2014**, 755, 78.
- (2) Kulkarni, S. S.; Hu, X.; Manetsch, R. *Chem. Commun.* **2013**, 49, 1193.
- (3) Pramanik, S.; Ghorai, P. *Org. Lett.* **2014**, 16, 2104.