

Supporting information

Direct synthesis of aryl-annulated [c]carbazoles by gold(I)-catalysed cascade reaction of azide-diynes and arenes

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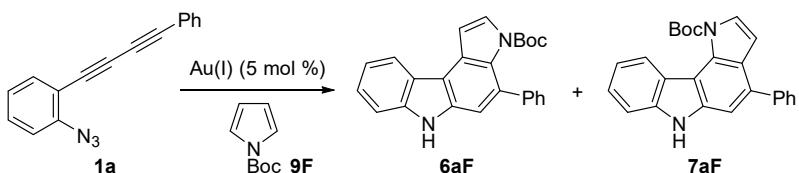
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Table of Contents

Reaction Optimisation Using <i>N</i> -Boc-pyrrole	S2
Experimental Section	S3
1. General Methods	S3
2. Preparation of the Cyclisation Precursors	S3
3. Gold-Catalysed Cascade Cyclisation	S11
4. Preparation of the Catalyst	S29
5. References	S29
NMR Spectra	S30

Table S1. Reaction Optimisation Using *N*-Boc-pyrrole ^a



Entry	Catalyst	Solvent	Temperature (°C)	Condition	Time (h)	Yield ^b (%)	Ratio ^c (6:7)
1	Ph ₃ PAuCl/AgNTf ₂	TCE	110		24	<5 ^d	87 : 13
2	IPrAuCl/AgNTf ₂	TCE	110		1	60	91 : 9
3	JohnPhosAuCl/AgNTf ₂	TCE	110		1	56	92 : 8
4	XPhosAuCl/AgNTf ₂	TCE	110		1	62	93 : 7
5	BrettPhosAuCl/AgNTf ₂	TCE	110		1	55	94 : 6
6	BrettPhosAuCl/AgSbF ₆	TCE	110		3	51	89 : 11
7	BrettPhosAuCl/AgOTf	TCE	110		20	<12 ^d	75 : 25
8	BrettPhosAu(MeCN)SbF₆	TCE	110	C	0.5	58	95 : 5
9	BrettPhosAuNTf ₂	TCE	110		0.5	58	95 : 5
10	BrettPhosAu(MeCN)SbF₆	DCE	80	D	1.5	60	92 : 8
11	BrettPhosAu(MeCN)SbF ₆	toluene	80		24	37	88 : 12
12	BrettPhosAu(MeCN)SbF ₆	MeCN	80		30	<48 ^d	87 : 13
13	BrettPhosAu(MeCN)SbF ₆	EtOH	reflux		24	<36 ^d	52 : 48
14	BrettPhosAu(MeCN)SbF ₆	AcOH	80		2	<49 ^d	53 : 47
15	BrettPhosAu(MeCN)SbF ₆	DCE	RT		72	59	72 : 28
16	BrettPhosAu(MeCN)SbF ₆	DCE	50		10	62	90 : 10
17	BrettPhosAu(MeCN)SbF ₆	TCE	140		0.25	51	91 : 9

^a Reaction conditions: **9F** (5 equiv.), gold catalyst (5 mol %). ^b Combined isolated yields. ^c Determined by ¹H NMR spectroscopy. ^d Contained small amounts of impurities.

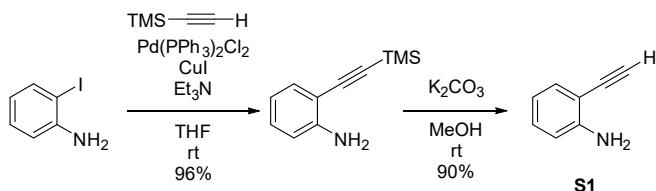
Experimental Section

1. General Methods

IR spectra were determined on a JASCO FT/IR-4100 spectrometer. Exact mass (HRMS) spectra were recorded on JMS-HX/HX 110A mass spectrometer or Shimadzu LC-ESI-IT-TOF-MS equipment. ¹H NMR spectra were recorded using a JEOL AL-400 or JEOL AL-500 spectrometer at 500 MHz frequency. Chemical shifts are reported in δ (ppm) relative to Me₄Si (in CDCl₃) as internal standard. ¹³C NMR spectra were recorded using a JEOL AL-500 and referenced to the residual solvent signal. Melting points were measured by a hot stage melting points apparatus (uncorrected). For column chromatography, silica gel (Wakogel C-200: Wako Pure Chemical Industries, Ltd), and amine silica gel (CHROMATOREX NH-DM1020: Fuji Silysa Chemical Ltd) were employed.

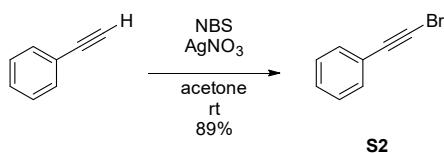
2. Preparation of the Cyclisation Precursors

2-Ethynylaniline (S1)^{1,2}



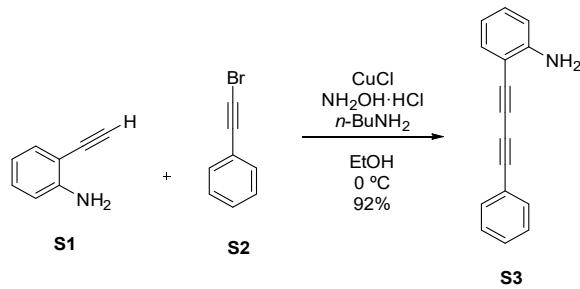
A mixture of 2-iodoaniline (4.38 g, 20.0 mmol), TMS acetylene (3.39 mL, 24.0 mmol), PdCl₂(PPh₃)₂ (337 mg, 0.48 mmol), CuI (91.4 mg, 0.48 mmol), and Et₃N (13.9 mL, 100 mmol) in THF (20 mL) was stirred at room temperature under Ar for 3 h. The mixture was filtered through a pad of Celite and concentrated in vacuo. The residue was purified by column chromatography on silica gel (hexane/EtOAc = 20/1) to give 2-[(trimethylsilyl)ethynyl]aniline² (3.65 g, 96%) as an orange oil. To a solution of this oil (1.70 g, 9.00 mmol) in MeOH (30 mL) was added K₂CO₃ (410 mg, 2.97 mmol). The reaction mixture was stirred at room temperature for 2 h and concentrated in vacuo. The residue was diluted with H₂O. The organic layer was washed with H₂O and brine, dried over MgSO₄, filtered, and concentrated in vacuo to give S1 (951 mg, 90%) as a brown oil. The spectral data were in good agreement with those previously reported.²

(Bromoethynyl)benzene (S2)^{1,3}



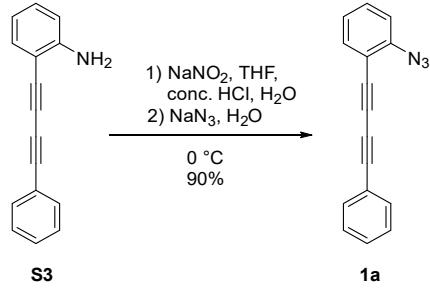
To a solution of ethynylbenzene (1.27 mL, 10.0 mmol) in acetone (50 mL) were added NBS (1.96 g, 11.0 mmol) and AgNO₃ (170 mg, 1.00 mmol). The mixture was stirred at room temperature for 7 h. The mixture was diluted with *n*-hexane and filtered through a pad of silica gel. The filtrate was concentrated in vacuo to give S2 (1.74 g, 89%) as a brown oil. The spectral data were in good agreement with those previously reported.³

2-(Phenylbuta-1,3-diy-1-yl)aniline (S3**)^{1,4a}**



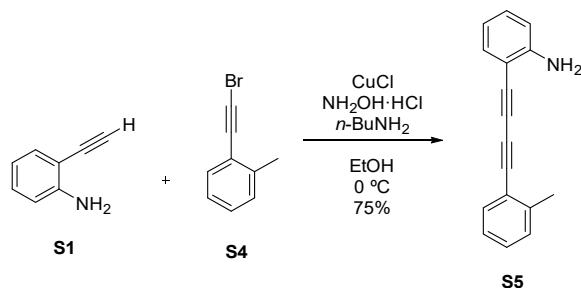
To a mixture of **S1** (1.06 g, 9.09 mmol), CuCl (45.0 mg, 0.455 mmol), NH₂OH·HCl (253 mg, 3.64 mmol), and *n*-BuNH₂ (2.26 mL, 22.8 mmol) in dry EtOH (22.8 mL) was added a solution of **S2** (2.14 g, 11.8 mmol) in dry EtOH (4.55 mL) via dropping funnel at 0 °C under Ar.^{4b} The mixture was stirred at room temperature for 1.5 h and concentrated in vacuo. The residue was diluted with Et₂O. The organic layer was washed with saturated aqueous NH₄Cl, H₂O, and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo. The filtrate was purified by column chromatography on silica gel (hexane/EtOAc = 10/1) to give **S3** (1.80 g, 92%) as a light yellow powder. The spectral data were in good agreement with those previously reported^{4a}: ¹H NMR (500 MHz, CDCl₃) δ: 4.32 (s, 2H), 6.69-6.70 (m, 2H), 7.16 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.32-7.39 (m, 4H), 7.52-7.54 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 73.9, 78.7, 78.9, 82.6, 105.7, 114.2, 117.7, 121.5, 128.3 (2C), 129.0, 130.5, 132.2 (2C), 132.8, 149.5.

1-Azido-2-(phenylbuta-1,3-diy-1-yl)benzene (1a**)^{1,5}**



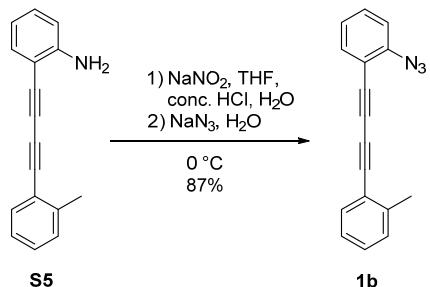
A solution of **S3** (1.09 g, 5.00 mmol) in THF/H₂O/conc. HCl (1/1/1, 10.0 mL) was cooled to 0 °C. To the solution was added NaNO₂ (690 mg, 10.0 mmol) in H₂O (10.0 mL) via dropping funnel at 0 °C. After the mixture was stirred at 0 °C for 15 min, NaN₃ (650 mg, 10.0 mmol) in H₂O (10.0 mL) was slowly added to the mixture at 0 °C,⁶ and the mixture was stirred for 3 h. The reaction mixture was quenched with H₂O. The resulting mixture was extracted with EtOAc twice. The combined organic layer was washed with brine, dried over MgSO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography on silica gel (hexane/EtOAc = 20/1) to give **1a** (1.09 g, 90%) as an light yellow powder: mp 93–96 °C; IR (neat) 2130, 2100; ¹H NMR (500 MHz, CDCl₃) δ: 7.11 (t, *J* = 7.0 Hz, 1H), 7.15 (d, *J* = 8.5 Hz, 1H), 7.33-7.41 (m, 4H), 7.50-7.54 (m, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 73.6, 77.0, 79.3, 83.3, 113.8, 118.5, 121.6, 124.6, 128.4 (2C), 129.3, 130.4 132.5 (2C), 134.4, 142.6; HRMS (ESI) calcd for C₁₆H₁₀N₃ [M+H]⁺: 244.0869, found 244.0867.

2-(*o*-Tolylbuta-1,3-diyn-1-yl)aniline (S5**)**



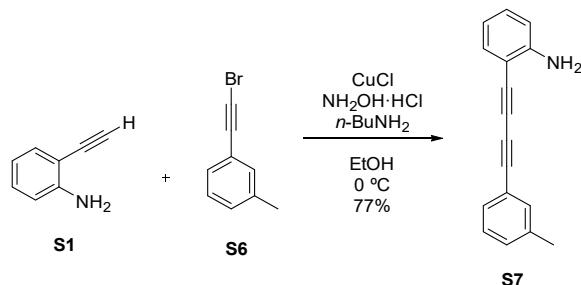
By a procedure identical with that described for the preparation of **S3**, **S1** (451 mg, 3.85 mmol) was converted to **S5** (671 mg, 75%) by the reaction with **S4** (983 mg, 5.04 mmol): yellow powder; mp 100–101.5 °C; IR (neat) 3479, 3392, 2211, 2137; ¹H NMR (400 MHz, CDCl₃) δ: 2.49 (s, 3H), 4.32 (s, 2H), 6.67–6.71 (m, 2H), 7.14–7.18 (m, 2H), 7.21–7.28 (m, 2H) 7.35 (d, *J* = 8.0 Hz, 1H), 7.49 (d, *J* = 7.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 20.7, 77.4, 79.1 (2C), 81.7, 106.2, 114.4, 117.9, 121.6, 125.7, 129.1, 129.6, 130.6, 132.8, 133.1, 141.6, 149.5; HRMS (ESI) calcd for C₁₇H₁₄N [M+H]⁺: 232.1126, found 232.1114

1-Azido-2-(*o*-tolylbuta-1,3-diyn-1-yl)benzene (1b**)**



By a procedure identical with that described for the preparation of **1a**, **S5** (550 mg, 2.38 mmol) was converted to **1b** (530 mg, 87%): light yellow powder; mp 61–63 °C; IR (neat) 2127, 2094; ¹H NMR (500 MHz, CDCl₃) δ: 2.49 (s, 3H), 7.09–7.17 (m, 3H), 7.20–7.28 (m, 2H), 7.38 (ddd, *J* = 7.9, 7.9, 1.5 Hz, 1H), 7.49–7.52 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 20.7, 77.1, 77.5, 79.4, 82.4, 113.9, 118.5, 121.4, 124.6, 125.6, 129.3, 129.6 (2C), 130.3, 134.4, 141.8, 142.5; HRMS (FAB) calcd for C₁₇H₁₂N₃ [M+H]⁺: 258.1031, found 258.1026.

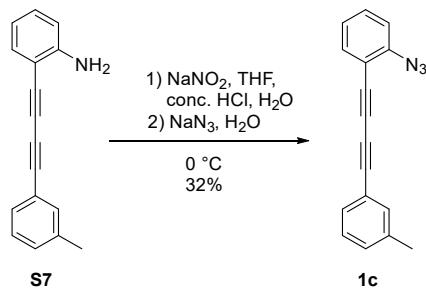
2-(*m*-Tolylbuta-1,3-diyn-1-yl)aniline (S7**)**



By a procedure identical with that described for the preparation of **S3**, **S1** (421 mg, 3.59 mmol) was converted to **S7** (637 mg, 77%) by the reaction with **S6** (912 mg, 4.67 mmol): yellow powder; mp 67–69 °C; IR (neat) 3476, 3378, 2207, 2136; ¹H NMR (500 MHz, CDCl₃) δ: 2.34 (s, 3H), 4.32 (s, 2H), 6.66–6.70 (m, 2H), 7.13–7.26 (m, 3H),

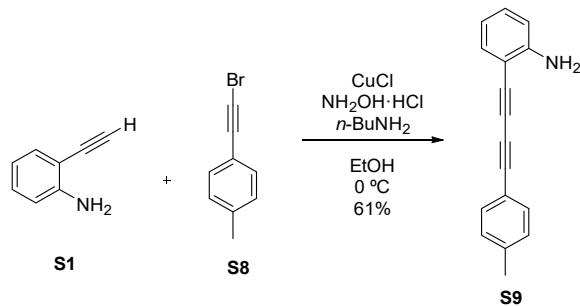
7.32–7.35 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.2, 73.5, 78.4, 79.1, 82.9, 106.2, 114.4, 117.9, 121.6, 128.3, 129.5, 130.1, 130.6, 133.0, 133.1, 138.2, 149.5; HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{14}\text{N}$ [$\text{M}+\text{H}]^+$: 232.1126, found 232.1127

1-Azido-2-(*m*-tolylbuta-1,3-diyne-1-yl)benzene (**1c**)



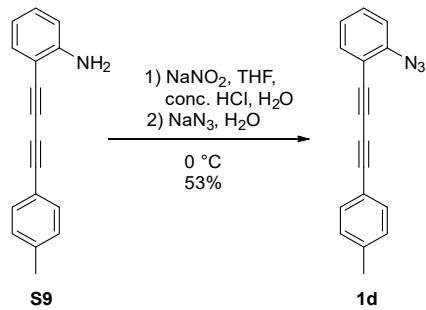
By a procedure identical with that described for the preparation of **1a**, **S7** (500 mg, 2.16 mmol) was converted to **1c** (180 mg, 32%): beige powder; mp 70–71 °C; IR (neat) 2126, 2094; ^1H -NMR (CDCl_3) δ : 2.34 (s, 3H), 7.09–7.12 (m, 1H), 7.15 (d, J = 7.5 Hz, 1H), 7.19 (d, J = 8.0 Hz, 1H), 7.21 (dd, J = 8.0, 8.0 Hz, 1H), 7.33–7.35 (m, 2H), 7.38 (ddd, J = 8.0, 8.0, 1.5 Hz, 1H), 7.50 (dd, J = 8.0, 1.5 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.7, 73.0, 76.6, 79.5, 83.6, 114.0, 118.5 (2C), 124.7, 129.2 (2C), 130.3, 132.4 (2C), 134.4, 139.8, 142.5; HRMS (FAB) calcd for $\text{C}_{17}\text{H}_{12}\text{N}_3$ [$\text{M}+\text{H}]^+$: 258.1031, found 258.1037.

2-(*p*-Tolylbuta-1,3-diyne-1-yl)aniline (**S9**)^{4a}



By a procedure identical with that described for the preparation of **S3**, **S1** (805 mg, 6.87 mmol) was converted to **S9** (966 mg, 61%) by the reaction with **S8** (2.14 g, 11.8 mmol): yellow powder. The spectral data were in good agreement with those previously reported.^{4a}

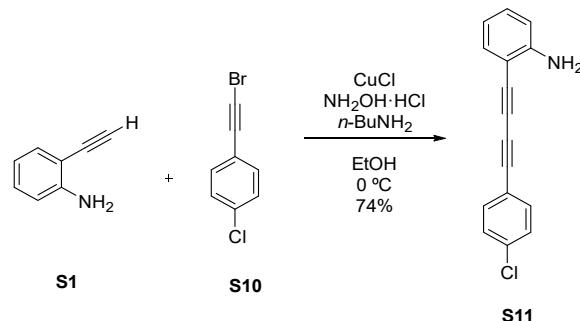
1-Azido-2-(*p*-tolylbuta-1,3-diyne-1-yl)benzene (**1d**)



By a procedure identical with that described for the preparation of **1a**, **S9** (966 mg, 4.18 mmol) was converted to **1d** (570 mg, 53%): yellow powder; mp 100–102 °C; IR (neat) 2116, 2092; ^1H NMR (500 MHz, CDCl_3) δ : 2.37 (s,

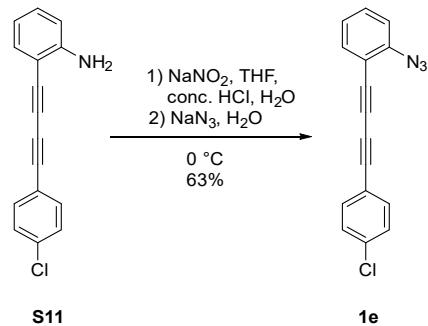
3H), 7.08-7.15 (m, 1H), 7.13-7.15 (m, 3H), 7.36-7.39 (m, 1H), 7.42-7.44 (m, 2H), 7.50 (dd, $J = 7.7, 1.4$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.6, 73.0, 76.6, 79.5, 83.6, 114.0, 118.5 (2C), 124.6, 129.2 (3C), 130.2, 134.4 (2C), 139.8, 142.5; HRMS (FAB) calcd for $\text{C}_{17}\text{H}_{12}\text{N}_3$ $[\text{M}+\text{H}]^+$: 258.1031, found 258.1026.

2-[(4-Chlorophenyl)buta-1,3-diyn-1-yl]aniline (S11)



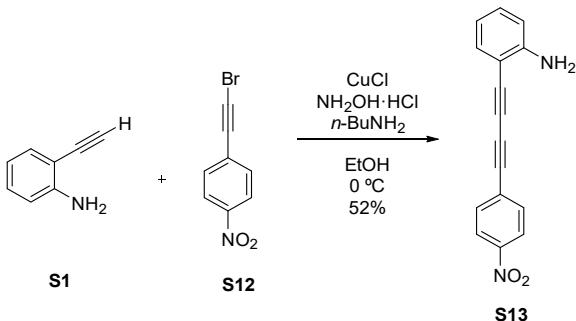
By a procedure identical with that described for the preparation of **S3**, **S1** (389 mg, 3.32 mmol) was converted to **S11** (617 mg, 74%) by the reaction with **S10** (932 mg, 4.32 mmol): yellow powder; mp 134-136 °C; IR (neat) 3477, 3378, 2206, 2139; ^1H NMR (400 MHz, CDCl_3) δ : 4.31 (s, 2H), 6.67-6.71 (m, 2H), 7.17 (dd, $J = 8.8, 8.0$ Hz, 1H), 7.31-7.35 (m, 3H), 7.45 (dd, $J = 6.7, 2.0$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 74.9, 78.8, 79.2, 81.4, 105.9, 114.4, 118.0, 120.4, 128.9 (2C), 130.8, 133.1 (2C), 133.5, 135.3, 149.6; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{11}\text{ClN}$ $[\text{M}+\text{H}]^+$: 252.0580, found 252.0575.

1-Azido-2-[(4-chlorophenyl)buta-1,3-diyn-1-yl]benzene (1e)



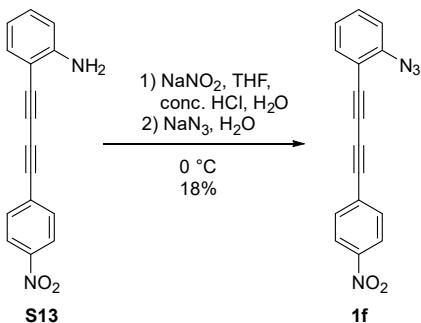
By a procedure identical with that described for the preparation of **1a**, **S11** (500 mg, 1.99 mmol) was converted to **1e** (351 mg, 63%): white powder; mp 144–145 °C; IR (neat) 2129, 2097; ^1H NMR (500 MHz, CDCl_3) δ : 7.09-7.16 (m, 2H), 7.31 (d, $J = 8.6$ Hz, 2H), 7.37-7.41 (m, 1H), 7.45 (d, $J = 8.6$ Hz, 2H), 7.50 (dd, $J = 8.0, 1.1$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 74.6, 77.5, 79.0, 82.0, 113.6, 118.5, 120.1, 124.7, 128.8 (2C), 130.5, 133.7 (2C), 134.4, 135.5, 142.7; HRMS (FAB) calcd for $\text{C}_{16}\text{H}_9\text{ClN}_3$ MH^+ : 278.0485, found 278.0481.

2-[(4-Nitrophenyl)buta-1,3-diyn-1-yl]aniline (S13)



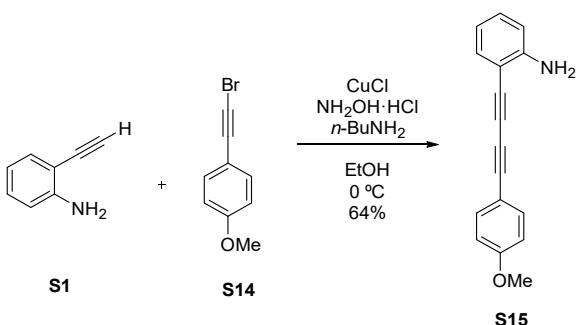
By a procedure identical with that described for the preparation of **S3**, **S1** (562 mg, 4.80 mmol) was converted to **S13** (653 mg, 52%) by the reaction with **S12** (1.42 g, 6.24 mmol): orange powder; mp 215–217 °C; IR (neat) 3499, 3397, 2127, 2097, 1591; ¹H NMR (500 MHz, CDCl₃) δ: 4.34 (s, 2H), 6.69–6.72 (m, 2H), 7.21 (ddd, *J* = 7.8, 7.8, 1.0 Hz, 1H), 7.36 (d, *J* = 6.9 Hz, 1H), 7.66 (d, *J* = 8.5 Hz, 2H), 8.23 (d, *J* = 8.5 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 78.4, 79.0, 80.4, 91.2, 105.3, 114.5 (2C), 118.1, 123.7, 128.9, 131.3, 133.0 (2C), 133.2, 133.4, 149.9; HRMS (FAB⁺) calcd for C₁₆H₁₁N₂O₂ [M+H]⁺: 263.0821, found 263.0825.

1-Azido-2-[(4-nitrophenyl)buta-1,3-diyn-1-yl]benzene (1f)



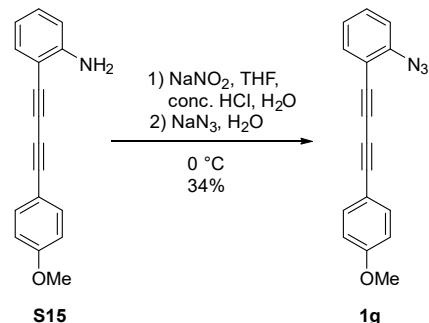
By a procedure identical with that described for the preparation of **1a**, **S13** (550 mg, 2.10 mmol) was converted to **1f** (108 mg, 18%): yellow powder; mp 168–171 °C; IR (neat) 2131, 2100, 1518; ¹H NMR (500 MHz, CDCl₃) δ: 7.14 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.41–7.45 (m, 1H), 7.53 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.66–7.71 (m, 2H), 8.20–8.25 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 78.4, 78.6, 79.9, 80.7, 113.1, 118.6, 123.7 (2C), 124.7, 128.6, 131.0, 133.2, 134.5 (2C), 143.0, 147.5; HRMS (FAB) calcd for C₁₆H₉N₄O₂ [M+H]⁺: 289.0726, found 289.0722.

2-[(4-Methoxyphenyl)buta-1,3-diyn-1-yl]aniline (S15)^{4a}



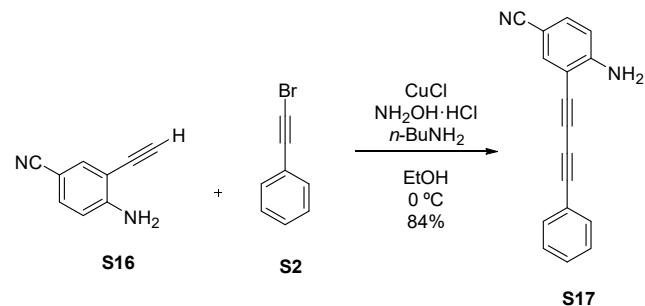
By a procedure identical with that described for the preparation of **S3**, **S1** (972 mg, 8.30 mmol) was converted to **S15** (1.31 g, 64%) by the reaction with **S14** (2.61 g, 12.4 mmol). The spectral data were in good agreement with those previously reported.^{4a}

1-Azido-2-[(4-methoxyphenyl)buta-1,3-diyn-1-yl]benzene (**1g**)



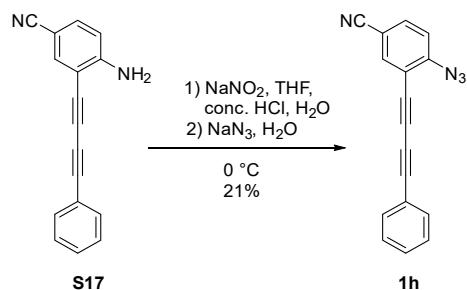
By a procedure identical with that described for the preparation of **1a**, **S15** (495 mg, 2.00 mmol) was converted to **1g** (188 mg, 34%): orange solid: mp 89–90 °C; IR (neat) 2127, 2096; ¹H NMR (500 MHz, CDCl₃) δ: 3.83 (s, 3H), 6.86 (d, *J* = 7.6 Hz, 2H), 7.08–7.15 (m, 2H), 7.37 (ddd, *J* = 7.9, 7.9, 1.7 Hz, 1H), 7.46–7.51 (m, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 55.3, 72.5, 76.4, 77.2, 79.6, 83.6, 113.5, 114.1, 118.5 (2C), 124.6, 130.2, 134.2, 134.3 (2C), 142.5, 160.4; HRMS (FAB) calcd for C₁₇H₁₂N₃O [M+H]⁺: 274.0980, found 274.0981.

4-Amino-3-(phenylbuta-1,3-diyn-1-yl)benzonitrile (**S17**)



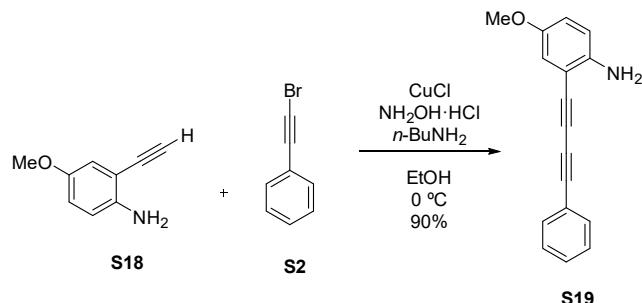
By a procedure identical with that described for the preparation of **S3**, **S16** (325 mg, 2.29 mmol) was converted to **S17** (463 mg, 84%) by the reaction with **S2** (538 mg, 2.97 mmol): white solid: mp 181–182 °C; IR (neat) 3473, 3355, 2216, 2127, 2097; ¹H NMR (500 MHz, CDCl₃) δ: 4.83 (br s, 2H), 6.70 (d, *J* = 8.6 Hz, 1H), 7.34–7.41 (m, 4H), 7.54 (dd, *J* = 8.6, 1.7 Hz, 2H), 7.62 (d, *J* = 1.7 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 73.2, 75.6, 80.5, 83.8, 100.4, 114.1, 119.0, 121.3, 128.5 (3C), 129.6, 132.5 (2C), 133.9, 137.4, 152.4; HRMS (FAB⁺) calcd for C₁₇H₁₁N₂ [M+H]⁺: 243.0922, found 243.0919.

4-Azido-3-(phenylbuta-1,3-diyn-1-yl)benzonitrile (1h)



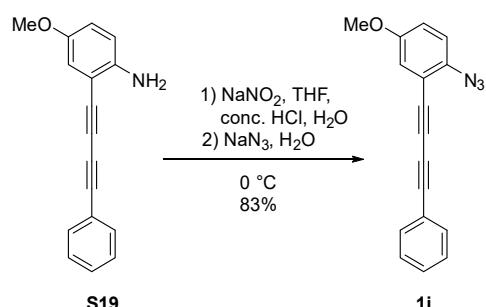
By a procedure identical with that described for the preparation of **1a**, **S17** (242 mg, 1.00 mmol) was converted to **1h** (56.4 mg, 21%): orange powder; mp 138–140 °C; IR (neat) 2226, 2122, 2084; ¹H NMR (500 MHz, CDCl₃) δ: 7.23 (d, *J* = 8.6 Hz, 1H), 7.35–7.43 (m, 3H), 7.54–7.56 (m, 2H), 7.63 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.77 (d, *J* = 1.7 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 73.0, 74.3, 81.6, 84.7, 108.5, 115.3, 117.4, 119.4, 121.1, 128.5 (2C), 129.8, 132.6 (2C), 133.2, 137.9, 146.9; HRMS (FAB) calcd for C₁₇H₉N₄ [M+H]⁺: 269.0827, found 269.0826.

4-Methoxy-2-(phenylbuta-1,3-diyn-1-yl)aniline (S19)



By a procedure identical with that described for the preparation of **S3**, **S18** (452 mg, 3.07 mmol) was converted to **S19** (680 mg, 90%) by the reaction with **S2** (723 mg, 3.99 mmol): yellow crystals; mp 85–87 °C; IR (neat) 3456, 3367, 2209, 2125; ¹H NMR (500 MHz, CDCl₃) δ: 3.73 (s, 3H), 4.05 (s, 2H), 6.65 (d, *J* = 8.7 Hz, 1H), 6.80 (dd, *J* = 8.7, 2.9 Hz, 1H), 6.87 (d, *J* = 2.9 Hz, 1H), 7.32–7.39 (m, 3H), 7.52 (dd, *J* = 7.9, 2.4 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 55.8, 73.8, 78.6, 79.0, 82.8, 106.6, 116.1, 118.8, 121.8, 128.4 (3C), 129.2, 132.4 (2C), 144.0, 151.7; HRMS (ESI) calcd for C₁₇H₁₄NO [M+H]⁺: 248.1075, found 248.1088.

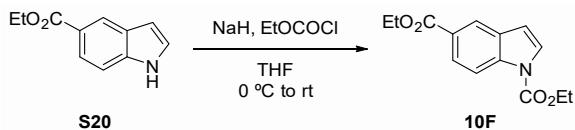
1-Azido-4-methoxy-2-(phenylbuta-1,3-diyn-1-yl)benzene (1i)



By a procedure identical with that described for the preparation of **1a**, **S19** (495 mg, 2.00 mmol) was converted to **1i** (454 mg, 83%): pale yellow solid; mp 75–77 °C; IR (neat) 2121, 2088; ¹H NMR (500 MHz, CDCl₃) δ: 3.79 (s, 3H), 6.95 (dd, *J* = 8.6, 2.9 Hz, 1H), 7.01 (d, *J* = 2.9 Hz, 1H), 7.05 (d, *J* = 8.6 Hz, 1H), 7.32–7.40 (m, 3H), 7.52–7.55

(m, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 55.7, 73.5, 76.9, 79.1, 83.4, 114.4, 117.6, 118.1, 119.7, 121.5, 128.4 (2C), 129.4, 132.5 (2C), 135.3, 156.3; HRMS (FAB) calcd for $\text{C}_{17}\text{H}_{12}\text{N}_3$ [$\text{M}+\text{H}]^+$: 274.0980, found 274.0981.

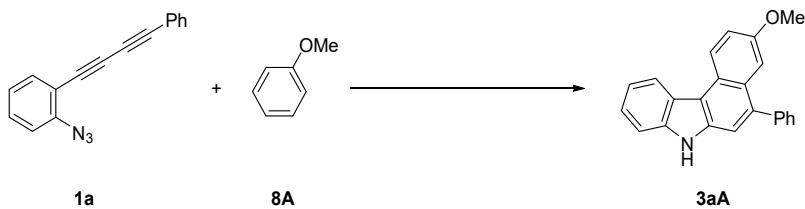
Diethyl 1*H*-Indole-1,5-dicarboxylate (10F)



To a suspension of NaH (60% dispersion in paraffin liquid; 160 mg, 4.00 mmol) in THF (2.0 mL) at 0 °C was added **S20** (378 mg, 2.00 mmol), and the mixture was stirred at 0 °C for 10 min under Ar. To the reaction mixture was added dropwise ClCO_2Et (0.286 mL, 3.00 mmol). After being stirred at room temperature for 2 h, the reaction mixture was diluted with H_2O and extracted with EtOAc . The combined organic layer was dried over Na_2SO_4 and filtered and concentrated in vacuo. The residue was purified by column chromatography (hexane/ EtOAc = 30/1) to give **10F** (453 mg, 87%) as a white solid: mp 73–74 °C; IR (neat) 1742, 1710; ^1H NMR (500 MHz, CDCl_3) δ : 1.42 (t, J = 7.2 Hz, 3H), 1.48 (t, J = 7.2 Hz, 3H), 4.40 (q, J = 7.2 Hz, 2H), 4.51 (q, J = 7.2 Hz, 2H), 6.67 (d, J = 3.5 Hz, 1H), 7.67 (d, J = 3.5 Hz, 1H), 8.04 (dd, J = 8.7, 1.7 Hz, 1H), 8.21 (d, J = 9.3 Hz, 1H), 8.30–8.31 (m, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 14.3 (2C), 60.8, 63.5, 108.3, 114.7, 123.1, 125.2, 125.7, 126.7, 130.2, 137.8, 150.7, 166.9; HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{16}\text{NO}_4$ [$\text{M}+\text{H}]^+$: 262.1079, found 262.1066.

3. Gold-Catalysed Cascade Cyclisation

General Procedure for Synthesis of Benzo[*c*]carbazole: Synthesis of 3-Methoxy-5-phenyl-7*H*-benzo[*c*]carbazole (**3aA**)



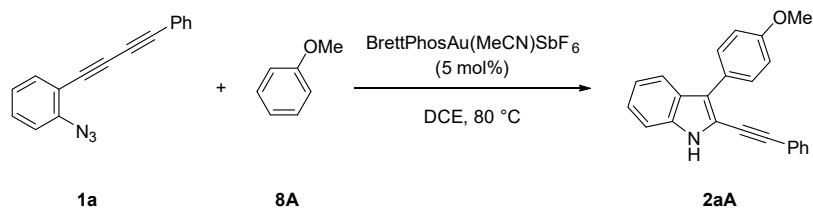
Condition A (Table 1, Entry 10): To a solution of **1a** (48.6 mg, 0.200 mmol) and **8A** (216 mg, 2.00 mmol) in DCE (1.00 mL) was added [JohnPhosAu(MeCN) SbF_6] (7.70 mg, 10.0 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 1 h. After disappearance of **1a** on TLC, the reaction mixture was allowed to warm to 140 °C and stirred at this temperature for 16 h. The mixture was concentrated in vacuo. The residue was purified by column chromatography on silica gel (hexane/ EtOAc = 20/1) to give **3aA** (48.3 mg, 75%).

Condition B (Table 1, Entry 12): To a solution of **1a** (48.6 mg, 0.200 mmol) in anisole (**8A**) (1.00 mL) was added [BrettPhosAu(MeCN) SbF_6] (10.1 mg, 10.0 μmol) at 140 °C. The mixture was stirred at 140 °C in pre-heated bath for 19.5 h and concentrated in vacuo. The residue was purified by column chromatography on silica gel (hexane/ EtOAc = 20/1) to give **3aA** (55.6 mg, 86%) as a white solid. Recrystallization from EtOAc –hexane gave pure **3aA**.

Compound **3aA**: colorless crystals; mp 168–172 °C; IR (neat): 3404; ^1H NMR (500 MHz, CDCl_3) δ : 3.80 (s, 3H), 7.37–7.42 (m, 3H), 7.44–7.47 (m, 2H), 7.51–7.60 (m, 6H), 8.39 (s, 1H), 8.55 (d, J = 8.0 Hz, 1H), 8.78 (d, J = 9.7 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 55.3, 107.6, 111.1, 114.0, 115.3, 117.8, 120.1, 121.9, 123.6, 124.3, 124.9,

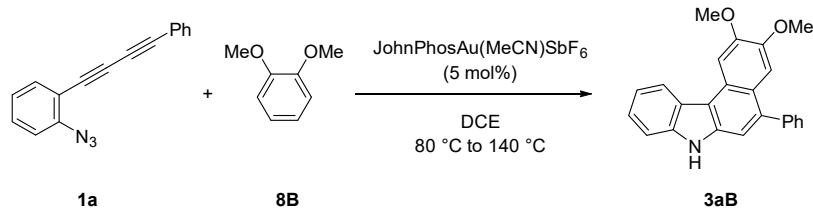
125.3, 127.3, 128.3 (2C), 128.9, 130.1 (2C), 135.5, 138.6, 138.7, 141.4, 155.4; HRMS (ESI) calcd for C₂₃H₁₈NO [M+H]⁺ 324.1383, found 324.1379.

3-(4-Methoxyphenyl)-2-(phenylethyynyl)-1*H*-indole (2aA) (Table 1, Entry 4).



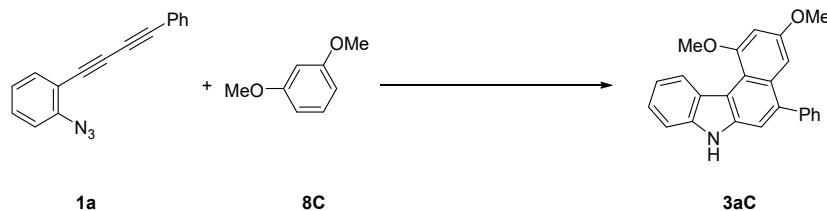
To a solution of **1a** (48.6 mg, 0.200 mmol) and anisole (**8A**) (216 mg, 2.00 mmol) in 1,2-DCE (1.00 mL) was added [BrettPhosAu(MeCN)SbF₆] (10.1 mg, 10.0 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 30 h and concentrated in vacuo. The residue was purified by column chromatography on silica gel (hexane/EtOAc = 20/1) to give **2aA** as brown solid (42.1 mg, 65%). Recrystallization from EtOAc–hexane gave pure **2aA**: colorless crystals; mp 97–107 °C; IR (neat): 3409, 2205; ¹H NMR (500 MHz, CDCl₃) δ: 3.89 (s, 3H), 7.06 (d, J = 8.6 Hz, 2H), 7.17 (dd, J = 7.4, 7.4 Hz, 1H), 7.28 (dd, J = 7.4, 7.4 Hz, 1H), 7.34–7.37 (m, 4H), 7.47–7.49 (m, 2H), 7.80 (d, J = 8.6 Hz, 2H), 7.82 (d, J = 8.0 Hz, 1H), 8.26 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 55.3, 82.3, 94.3, 110.9, 114.0 (2C), 115.5, 120.1, 120.6, 122.4, 122.7, 123.9, 126.1, 126.7, 128.4 (3C), 130.0 (2C), 131.3 (2C), 136.0, 158.4; HRMS (ESI) calcd for C₂₃H₁₈NO [M+H]⁺ 324.1383, found 324.1378.

2,3-Dimethoxy-5-phenyl-7*H*-benzo[c]carbazole (3aB).



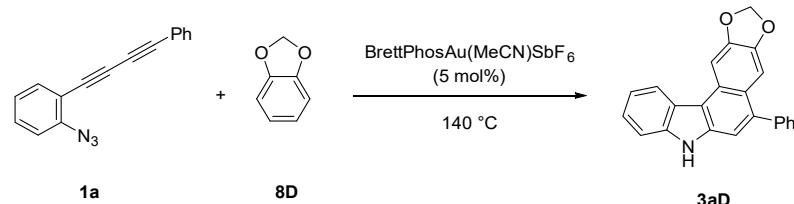
According to the general procedure described for the preparation of **3aA**, **1a** (48.6 mg, 0.200 mmol) was converted into **3aB** (49.9 mg, 70%) (condition A; 80 °C, 2 h then 140 °C, 15 h). The reaction of **1a** (48.6 mg, 0.200 mmol) and **2B** (1.00 mL) under condition B (140 °C, 27 h) gave **3aB** (70 mg, quant) as a white solid. Recrystallization from EtOAc–hexane gave pure **3aB**: colorless crystals; mp 245–257 °C; IR (neat): 3332; ¹H NMR (500 MHz, CDCl₃) δ: 3.82 (s, 3H), 4.20 (s, 3H), 7.38–7.40 (m, 3H), 7.44 (dd, J = 7.4 Hz, 2H), 7.50–7.57 (m, 5H), 8.13 (s, 1H), 8.34 (s, 1H), 8.46 (d, J = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 55.7, 55.9, 103.3, 107.3, 111.1, 111.5, 114.7, 120.0, 121.4, 122.6, 123.6, 124.2, 125.7, 127.2, 128.3 (2C), 130.1 (2C), 136.2, 138.4, 138.8, 141.7, 146.7, 149.9; HRMS (ESI) calcd for C₂₄H₂₀NO₂ [M+H]⁺ 354.1489, found 354.1489.

1,3-Dimethoxy-5-phenyl-7*H*-benzo[c]carbazole (3aC).



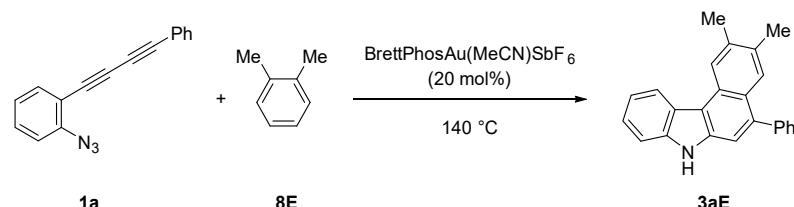
According to the general procedure (condition A; 80 °C, 2 h then 140 °C, 15 h) described for the preparation of **3aA**, **1a** (48.6 mg, 0.200 mmol) was converted into **3aC** (28.3 mg, 40%). The reaction of **1a** (48.6 mg, 0.200 mmol) and **8C** (1.00 mL) under the condition B (140 °C, 26 h) gave **3aC** (67.3 mg, 95%) as a brown solid. Recrystallization from EtOAc–hexane gave pure **3aC**: colorless crystals; mp 205–210 °C; IR (neat): 3396; ¹H NMR (400 MHz, CDCl₃) δ: 3.73 (s, 3H), 4.14 (s, 3H), 6.77 (d, *J* = 2.3 Hz, 1H), 6.94 (d, *J* = 2.3 Hz, 1H), 7.25–7.29 (dd, 1H), 7.36–7.51 (m, 8H), 8.29 (s, 1H), 8.87 (d, *J* = 8.1 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 55.1, 55.2, 98.4, 99.5, 110.5, 114.4, 114.7, 118.0, 119.3, 124.3, 124.3, 126.7, 127.2, 128.2 (2C), 130.0 (2C), 130.2, 136.0, 138.4, 139.1, 141.9, 155.6, 156.9; HRMS (ESI) calcd for C₂₄H₂₀NO₂ [M+H]⁺ 354.1489, found 354.1490.

5-Phenyl-7*H*-[1,3]dioxolo[4',5':4,5]benzo[1,2-*c*]carbazole (3aD**).**



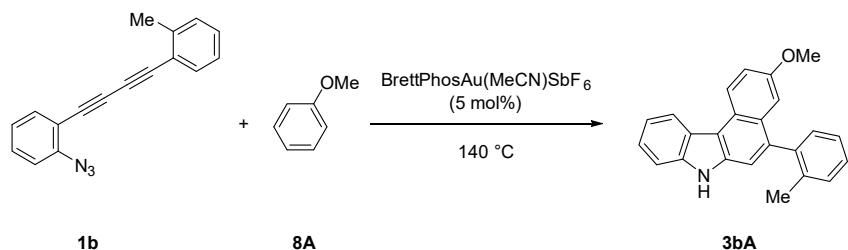
According to the general procedure (condition B; 140 °C, 18 h) described for the preparation of **3aA**, **1a** (48.6 mg, 0.200 mmol) was converted into **3aD** (51.1 mg, 76%) as a white solid. Recrystallization from EtOAc–hexane gave pure **3aD**: colorless crystals; mp 192–198 °C; IR (neat): 3411; ¹H NMR (500 MHz, CDCl₃) δ: 6.07 (s, 2H), 7.31 (s, 1H), 7.37 (dd, *J* = 7.7, 7.7 Hz, 1H), 7.41 (s, 1H), 7.43–7.46 (m, 2H), 7.48–7.55 (m, 5H), 8.18 (s, 1H), 8.30 (s, 1H), 8.47 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 100.9, 101.1, 104.6, 111.0, 111.5, 115.4, 120.1, 121.6, 123.7, 123.9, 124.3, 126.8, 127.2, 128.3 (2C), 130.1 (2C), 136.2, 138.8, 139.0, 141.6, 145.2, 148.1; HRMS (ESI) calcd for C₂₃H₁₆NO₂ [M+H]⁺ 338.1176, found 338.1173.

2,3-Dimethyl-5-phenyl-7*H*-benzo[*c*]carbazole (3aE**).**



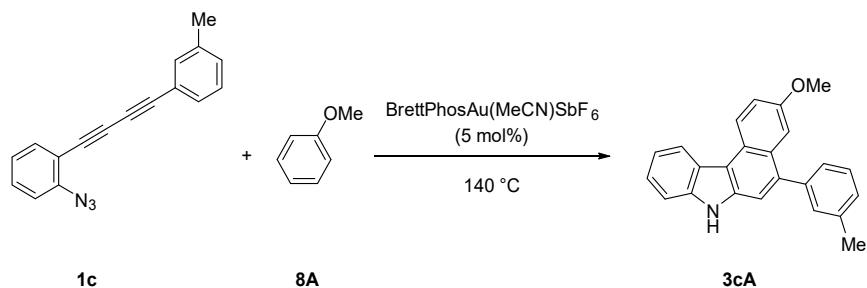
According to the general procedure (condition B; 140 °C, 1 h) described for the preparation of **3aA**, **1a** (48.6 mg, 0.200 mmol) was converted into **3aE** (26.7 mg, 42%) as a white solid. In the case, the reaction was conducted using 20 mol % BrettPhosAu(MeCN)SbF₆ (40.4 mg, 0.04 mmol). Recrystallization from EtOAc–hexane gave pure **3aE**: colorless crystals; mp 232–242 °C; IR (neat): 3422; ¹H NMR (500 MHz, CDCl₃) δ: 2.38 (s, 3H), 2.58 (s, 3H), 7.37–7.40 (m, 1H), 7.42–7.47 (m, 3H), 7.50–7.58 (m, 5H), 7.73 (s, 1H), 8.29 (s, 1H), 8.60 (d, *J* = 7.4 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 20.3, 20.7, 110.0, 112.7, 114.5, 120.0, 122.0, 123.5, 124.0, 124.1, 126.5, 127.1 (2C), 128.2 (2C), 128.9, 130.3 (2C), 132.3, 136.3, 136.4, 138.6, 139.0, 141.6; HRMS (ESI) calcd for C₂₄H₂₀N [M+H]⁺ 322.1590, found 322.1589.

3-Methoxy-5-(*o*-tolyl)-7*H*-benzo[*c*]carbazole (**3bA**)



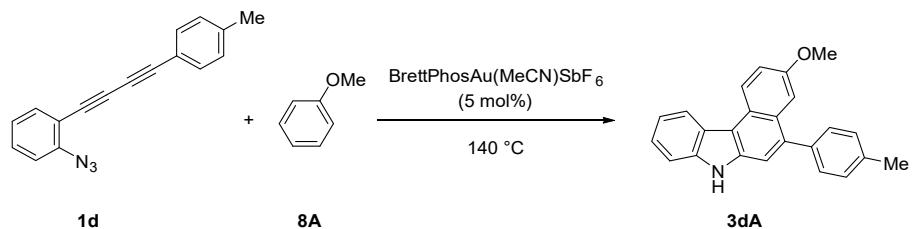
According to the general procedure (condition B; 140 °C, 20 h) described for the preparation of **3aA**, **1d** (51.4 mg, 0.200 mmol) was converted into **3bA** (60.3 mg, 89%) as a colorless oil: IR (neat): 3407; ¹H NMR (500 MHz, CDCl₃) δ: 2.05 (s, 3H), 3.73 (s, 3H), 6.93 (d, *J* = 2.9 Hz, 1H), 7.31-7.32 (m, 2H), 7.36-7.39 (m, 4H), 7.41 (s, 1H), 7.42-7.45 (m, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 8.28 (s, 1H), 8.54 (d, *J* = 7.4 Hz, 1H), 8.76 (d, *J* = 9.2 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 20.1, 55.2, 107.4, 111.1, 113.7, 115.2, 117.7, 120.1, 121.8, 123.7, 124.3, 124.8, 125.0, 125.7, 127.7, 129.2, 129.9, 130.4, 135.5, 137.0, 138.2, 138.6, 140.7, 155.5; HRMS (ESI) calcd for C₂₄H₂₀NO [M+H]⁺ 338.1539, found 338.1538.

3-Methoxy-5-(*m*-tolyl)-7*H*-benzo[*c*]carbazole (**3cA**)



According to the general procedure (condition B; 140 °C, 20 h) described for the preparation of **3aA**, **1c** (51.4 mg, 0.200 mmol) was converted into **3cA** (63.6 mg, 94%) as a colorless oil: IR (neat): 3411; ¹H NMR (500 MHz, CDCl₃) δ: 2.46 (s, 3H), 3.80 (s, 3H), 7.27 (d, *J* = 7.4 Hz, 1H), 7.36-7.39 (m, 4H), 7.41-7.42 (m, 2H), 7.44 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.53 (s, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 8.33 (s, 1H), 8.54 (d, *J* = 7.4 Hz, 1H), 8.77 (d, *J* = 9.2 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.5, 55.3, 107.7, 111.1, 113.9, 115.2, 117.8, 120.1, 121.9, 123.7, 124.3, 124.8, 125.3, 127.2, 128.0, 128.2, 128.9, 130.8, 135.5, 138.0, 138.7, 138.8, 141.4, 155.4; HRMS (ESI) calcd for C₂₄H₂₀NO [M+H]⁺ 338.1539, found 338.1538.

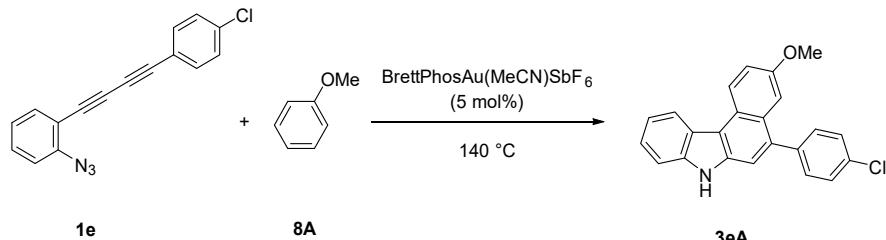
3-Methoxy-5-(*p*-tolyl)-7*H*-benzo[*c*]carbazole (**3dA**)



According to the general procedure (condition B; 140 °C, 20 h) described for the preparation of **3aA**, **1b** (51.4 mg, 0.200 mmol) was converted into **3dA** (47.0 mg, 70%). Recrystallization from EtOAc–hexane gave pure **3dA**: colorless crystals; mp 162–170 °C; IR (neat): 3407; ¹H NMR (500 MHz, CDCl₃) δ: 2.47 (s, 3H), 3.79 (s, 3H), 7.30

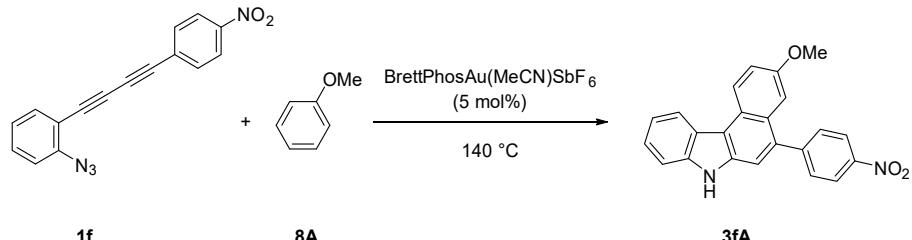
(d, $J = 8.0$ Hz, 2H), 7.34–7.40 (m, 2H), 7.41–7.46 (m, 5H), 7.48 (d, $J = 8.0$ Hz, 1H), 8.22 (s, 1H) 8.52 (d, $J = 8.0$ Hz, 1H), 8.75 (d $J = 9.2$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 21.3, 55.3, 107.6, 111.1, 114.0, 117.8, 120.0, 121.9, 123.6, 124.3, 124.8, 125.2, 128.9, 129.0 (2C), 130.0 (2C), 130.1, 135.5, 136.9, 138.4, 138.6, 138.7, 155.4; HRMS (ESI) calcd for $\text{C}_{24}\text{H}_{20}\text{NO} [\text{M}+\text{H}]^+$ 338.1539, found 338.1539.

5-(4-chlorophenyl)-3-methoxy-7*H*-benzo[*c*]carbazole (**3eA**)



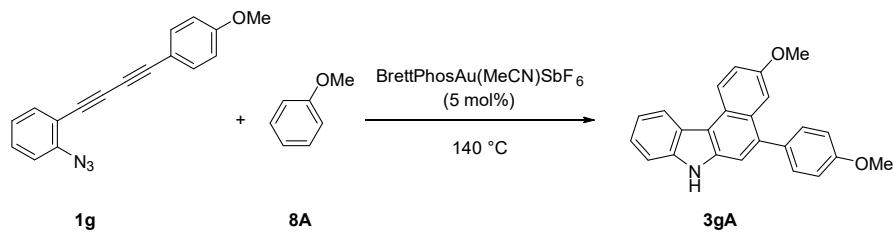
According to the general procedure (condition B; 140 °C, 22.5 h) described for the preparation of **3aA**, **1e** (55.5 mg, 0.200 mmol) was converted into **3eA** (53.5 mg, 74%). Recrystallization from EtOAc–hexane gave pure **3eA**: colorless crystals; mp 170–177 °C; IR (neat): 3406; ^1H NMR (500 MHz, CDCl_3) δ : 3.11 (s, 3H), 7.30 (d, $J = 2.9$ Hz, 1H), 7.36–7.40 (m, 3H), 7.43–7.51 (m, 6H), 8.23 (s, 1H), 8.52 (d, $J = 7.4$ Hz, 1H), 8.76 (d, $J = 9.2$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 55.3, 107.1, 111.1, 114.0, 115.5, 118.0, 120.2, 121.9, 123.5, 124.5, 124.9, 125.2, 128.5 (2C), 128.6, 131.4 (2C), 133.2, 135.3, 137.1, 138.7, 139.8, 155.5; HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{17}\text{ClNO} [\text{M}+\text{H}]^+$ 358.0993, found 358.0991.

3-Methoxy-5-(4-nitrophenyl)-7*H*-benzo[*c*]carbazole (**3fA**)



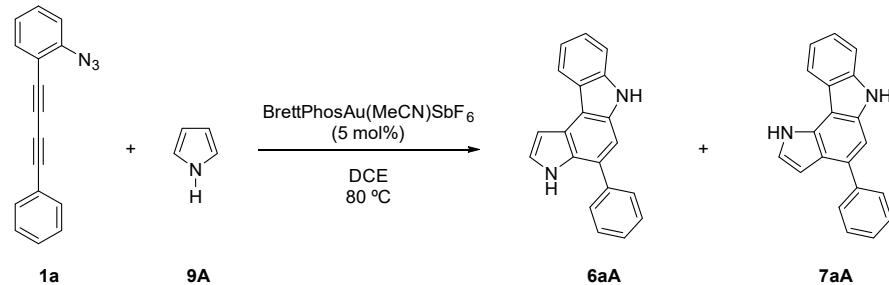
According to the general procedure (condition B; 140 °C, 23 h) described for the preparation of **3aA**, **1f** (57.6 mg, 0.200 mmol) was converted into **3fA** (32.0 mg, 43%). Recrystallization from EtOAc–hexane gave pure **3fA**: colorless crystals; mp 244–248 °C; IR (neat): 3343, 1498, 1344; ^1H NMR (600 MHz, CDCl_3) δ : 3.82 (s, 3H), 7.39–7.45 (m, 3H), 7.49 (dd, $J = 7.4$ Hz, 1H), 7.57 (s, 1H), 7.60 (d, $J = 7.7$ Hz, 1H), 7.77 (d, $J = 8.8$ Hz, 2H), 8.40 (d, $J = 8.8$ Hz, 2H), 8.43 (s, 1H), 8.56 (d, $J = 7.7$ Hz, 1H), 8.81 (d, $J = 8.8$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 55.4, 106.8, 111.3, 114.3, 116.5, 118.3, 120.4, 122.2, 123.4, 123.7 (2C), 125.0, 125.2, 125.4, 128.1, 131.0 (2C), 135.2, 135.8, 139.0, 147.2, 148.6, 155.9; HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{17}\text{N}_2\text{O}_3 [\text{M}-\text{H}]^-$ 367.1088, found 367.1088.

3-Methoxy-5-(4-methoxyphenyl)-7*H*-benzo[*c*]carbazole (**3gA**)



According to the general procedure (condition B; 140 °C, 12 h) described for the preparation of **3aA**, **1g** (54.6 mg, 0.200 mmol) was converted into **3gA** (42.8 mg, 61%) as a brown solid: mp 175–185 °C; IR (neat): 3406; ¹H NMR (500 MHz, CDCl₃) δ: 3.78 (s, 3H), 3.88 (s, 3H), 7.00 (d, *J* = 8.6 Hz, 2H), 7.33–7.45 (m, 8H), 8.19 (s, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.74 (d, *J* = 8.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 55.3, 55.3, 107.5, 111.1, 113.7 (2C), 114.0, 115.0, 117.8, 120.0, 121.8, 123.6, 124.2, 124.8, 125.2, 129.0, 131.1 (2C), 133.7, 135.5, 138.2, 138.7, 155.4, 158.8; HRMS (ESI) calcd for C₂₄H₂₀NO₂ [M+H]⁺ 354.1489, found 354.1489.

4-Phenyl-3,6-dihydropyrrolo[2,3-*c*]carbazole (6aA**) and Its [3,2-*c*]-Isomer (**7aA**) (Table 3, Entry 1)¹**

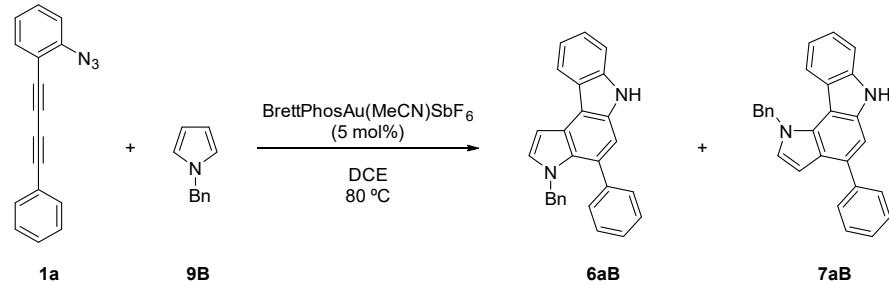


To a solution of **1a** (24.3 mg, 0.100 mmol) and **9A** (0.0347 mL, 0.5 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 8 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give a mixture of **6aA** and **7aA** containing a small amount of impurities (17.6 mg, <62%; **6aA**:**7aA** = 25:75). These isomers were separated by column chromatography on amine silica gel (hexane/CHCl₃ = 2/1) to give, in the order of elution, **6aA** and **7aA**.

Compound **6aA**: brown viscous oil; IR (neat) 3406; ¹H NMR (500 MHz, CDCl₃) δ: 7.15 (dd, *J* = 2.5, 1.5 Hz, 1H), 7.24 (s, 1H), 7.28–7.32 (m, 1H), 7.35 (dd, *J* = 3.0, 3.0 Hz, 1H), 7.38–7.45 (m, 3H), 7.52 (dd, *J* = 8.0, 8.0 Hz, 2H), 7.66–7.69 (m, 2H), 8.04 (s, 1H), 8.28 (d, *J* = 7.0 Hz, 1H), 8.54 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 101.4, 106.2, 110.5, 113.7, 119.2, 121.1, 121.3, 123.6, 124.18, 124.23, 125.0, 127.4, 128.5 (2C), 128.9, 129.2 (2C), 134.9, 139.1, 139.7; HRMS (ESI) calcd for C₂₀H₁₅N₂ [M+H]⁺: 283.1230, found 283.1231.

Compound **7aA**: white solid; mp 231–234 °C; IR (neat) 3395; ¹H NMR (500 MHz, CDCl₃) δ: 6.87 (dd, *J* = 3.5, 2.5 Hz, 1H), 7.31–7.34 (m, 3H), 7.39–7.44 (m, 2H), 7.51–7.54 (m, 3H), 7.78–7.79 (m, 2H), 8.08 (d, *J* = 6.5 Hz, 1H), 8.27 (s, 1H), 8.79 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 103.4, 104.6, 106.8, 110.7, 119.5, 120.2, 120.3, 121.7 (2C), 124.3, 127.0, 128.5 (2C), 129.1 (2C), 130.2, 133.7, 137.1, 138.7, 141.6; HRMS (ESI) calcd for C₂₀H₁₅N₂ [M+H]⁺: 283.1230, found 283.1231.

3-Benzyl-4-phenyl-3,6-dihydropyrrolo[2,3-*c*]carbazole (6aB**) and Its [3,2-*c*]-Isomer (**7aB**) (Table 3, Entry 2)¹**

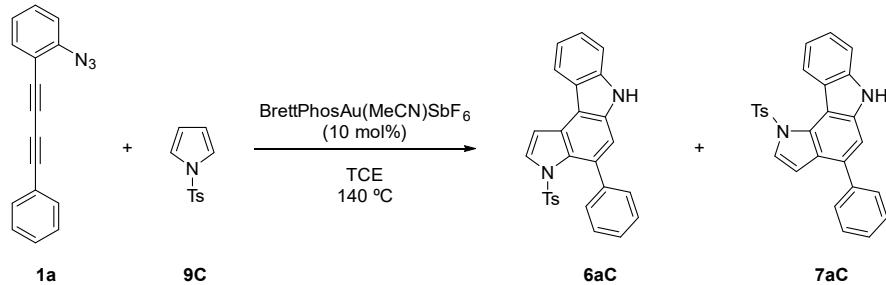


To a solution of **1a** (24.3 mg, 0.100 mmol) and **9B** (78.6 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 10 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 10/1) to give a **6aB** (4.20 mg, 11%) and **7aB** (19.0 mg, 51%) in the order of elution, **6aB** and **7aB**; (**6aB**:**7aB** = 18:82).

Compound **6aB**: yellow solid; mp 163–168 °C; IR (neat) 3728; ¹H NMR (500 MHz, CDCl₃) δ: 5.00 (s, 2H), 6.47 (m, 2H), 7.05 (s, 1H), 7.06-7.13 (m, 3H), 7.20 (d, *J* = 3.0 Hz, 1H), 7.22-7.28 (m, 5H), 7.30-7.35 (m, 2H), 7.41 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 8.06 (s, 1H), 8.30 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 52.2, 100.2, 108.7, 110.5, 113.8, 119.2, 121.2, 123.4, 123.7, 124.2, 125.8 (2C), 126.6, 126.9, 127.1, 127.6 (2C), 128.2 (2C), 128.7, 129.9 (2C), 130.8, 134.0, 138.8, 139.2, 140.6; HRMS (ESI) calcd for C₂₇H₂₁N₂ [M+H]⁺: 373.1699, found 373.1700.

Compound **7aB**: white solid; mp 173–178 °C; IR (neat) 3728; ¹H NMR (500 MHz, CDCl₃) δ: 6.02 (s, 2H), 6.84 (d, *J* = 3.0 Hz, 1H), 7.00-7.04 (m, 2H), 7.24-7.33 (m, 7H), 7.39-7.42 (m, 2H), 7.50 (t, *J* = 8.0 Hz, 2H), 7.75 (d, *J* = 7.5 Hz, 2H), 8.01 (d, *J* = 8.5 Hz, 1H), 8.23 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 53.2, 103.6, 104.8, 106.9, 110.6, 119.3, 121.0, 121.7, 122.6, 123.9, 126.5 (2C), 126.6, 127.0, 127.5, 128.4 (2C), 128.9 (2C), 129.2 (2C), 132.6, 134.1, 138.0, 138.7 (2C), 141.3; HRMS (ESI) calcd for C₂₇H₂₁N₂ [M+H]⁺: 373.1699, found 373.1702.

4-Phenyl-3-tosyl-3,6-dihydropyrrolo[2,3-*c*]carbazole (**6aC**) and Its [3,2-*c*]-Isomer (**7aC**) (Table 3, Entry 3)¹

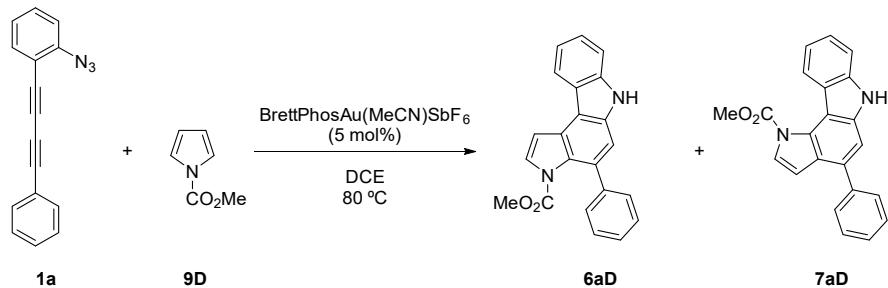


To a solution of **1a** (24.3 mg, 0.100 mmol) and **9C** (111 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (10.1 mg, 10.0 μmol) at 140 °C. The mixture was stirred at 140 °C in pre-heated bath for 30 min and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give a mixture of **6aC** and **7aC** (14.7 mg, 34%; **6aC**:**7aC** = 58:42). These isomers were separated by column chromatography on silica gel (toluene) to give, in the order of elution, **6aC** and **7aC**.

Compound **6aC**: brown viscous oil; IR (neat) 3736; ¹H NMR (500 MHz, CDCl₃) δ: 2.24 (s, 3H), 6.98 (d, *J* = 8.5 Hz, 2H), 7.18-7.21 (m, 3H), 7.27-7.31 (m, 2H), 7.37-7.47 (m, 7H), 7.80 (d, *J* = 4.0 Hz, 1H), 8.13-8.14 (m, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.5, 110.0, 110.8, 111.6, 114.1, 119.8, 121.1, 122.8, 125.4, 126.5 (2C), 127.0, 127.4, 127.7 (2C), 128.9, 129.1 (2C), 129.3 (2C), 130.2, 132.0, 134.4, 136.6, 139.6, 141.8, 144.0; HRMS (ESI) calcd for C₂₇H₂₁N₂O₂S [M+H]⁺: 437.1318, found 437.1318.

Compound **7aC**: white viscous oil; IR (neat) 3390; ¹H NMR (500 MHz, CDCl₃) δ: 2.24 (s, 3H), 6.98 (d, *J* = 8.6 Hz, 2H), 7.19 (t, *J* = 7.2 Hz, 3H), 7.29 (dt, *J* = 12.4, 3.3 Hz, 2H), 7.37-7.39 (m, 3H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.45-7.47 (m, 3H), 7.80 (d, *J* = 4.0 Hz, 1H), 8.14 (d, *J* = 7.4 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.5, 109.3, 110.0, 112.6, 114.2, 119.2, 121.7, 125.5, 125.7, 125.9, 126.8 (2C), 127.3, 128.3, 128.6 (2C), 128.9 (2C), 129.0 (2C), 132.4, 133.1, 133.6, 139.6, 139.7, 140.1, 144.3; HRMS (ESI) calcd for C₂₇H₂₁N₂O₂S [M+H]⁺: 437.1318, found 437.1317.

Methyl 4-Phenylpyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6aD**) and Its [3,2-*c*]-Isomer (**7aD**) (Table 3, Entry 4)**

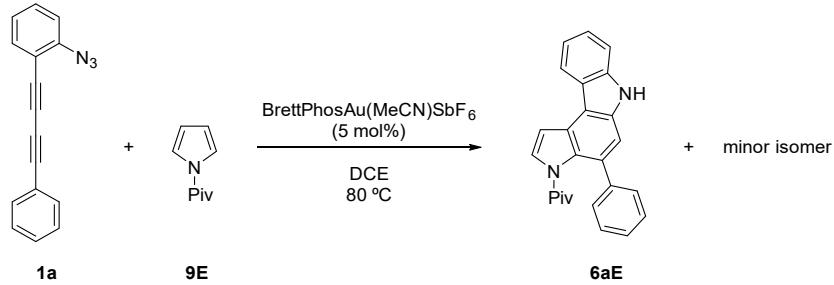


To a solution of **1a** (24.3 mg, 0.100 mmol) and **9D** (62.3 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 1.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give a mixture of **6aD** and **7aD** (21.2 mg, 62%; **6aD**:**7aD** = 81:19). These isomers were separated by column chromatography on amine silica gel (hexane/CHCl₃ = 1/2) to give, in the order of elution, **6aD** and **7aD**.

Compound **6aD**: white powder; mp 173–175 °C; IR (neat) 3380, 1751; ¹H NMR (500 MHz, CDCl₃) δ: 3.25 (s, 3H), 7.28 (d, *J* = 3.5 Hz, 1H), 7.31–7.37 (m, 3H), 7.42–7.53 (m, 6H), 7.80 (d, *J* = 3.4 Hz, 1H), 8.21 (s, 1H), 8.25 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 53.2, 106.2, 110.2, 110.7, 114.1, 119.6, 121.3, 123.1, 125.1, 125.8, 126.6, 127.2, 127.4, 128.5 (2C), 128.7 (2C), 136.4, 139.6, 139.6, 143.1, 152.0; HRMS (ESI) calcd for C₂₂H₁₇N₂O₂ [M+H]⁺: 341.1290, found 341.1284.

Compound **7aD**: orange viscous oil; IR (neat) 3398, 1734; ¹H NMR (500 MHz, CDCl₃) δ: 4.12 (s, 3H), 6.88 (d, *J* = 4.6 Hz, 1H), 7.27–7.30 (m, 1H), 7.41–7.47 (m, 3H), 7.49–7.53 (m, 3H), 7.63 (d, *J* = 3.5 Hz, 1H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.94 (d, *J* = 8.0 Hz, 1H), 8.36 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 54.2, 107.8, 109.0, 110.2, 110.4, 119.2, 122.0, 123.3, 124.9, 125.3, 125.5, 127.2, 128.6 (2C), 129.2 (2C), 129.9, 133.4, 139.3, 139.5, 140.6, 152.2; HRMS (ESI) calcd for C₂₂H₁₇N₂O₂ [M+H]⁺: 341.1290, found 341.1290

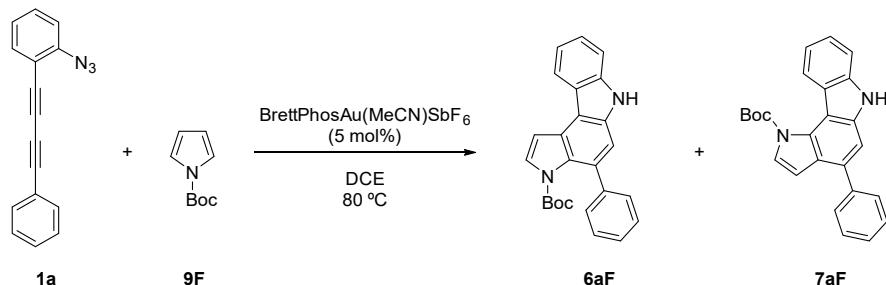
2,2-Dimethyl-1-(4-phenylpyrrolo[2,3-*c*]carbazol-3(6*H*)-yl)propan-1-one (6aE**) (Table 3, Entry 5)**



To a solution of **1a** (24.3 mg, 0.100 mmol) and **9E** (75.6 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 1.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give an inseparable mixture of **6aE** and minor isomer (21.9 mg, 60%; **6aE**: minor isomer = 82:18): orange viscous oil; IR (neat) 3393, 1695; ¹H NMR (500 MHz, CDCl₃) δ: 1.37 (s, 9H), 7.23 (d, *J* = 3.4 Hz, 1H), 7.25 (s, 1H), 7.32 (dd, *J* = 7.2, 5.4 Hz, 2H), 7.42–7.49 (m, 6H), 7.74 (d, *J* = 4.0 Hz, 1H), 8.23 (d, *J* = 9.7 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃; including amide rotamers) δ: 28.8 and 29.3 (totally 3C), 41.6, 105.6, 107.9, 108.3, 109.9, 110.1, 119.1, 122.0, 122.5,

123.5, 124.6, 124.9, 125.7, 126.6, 127.1, 127.4, 128.5 (2C), 129.3 (2C), 131.3, 139.2, 140.8, 177.9; HRMS (ESI) calcd for C₂₅H₂₃N₂O [M+H]⁺: 367.1810, found 367.1801.

tert-Butyl 4-Phenylpyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6aF**) and Its [3,2-*c*]-Isomer (**7aF**) (Table 3, Entry 6)¹**

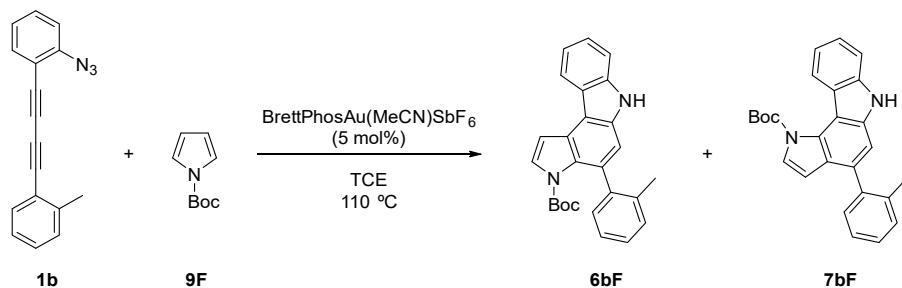


To a solution of **1a** (24.3 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 µmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 1.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 10/1) to give a mixture of **6aF** and **7aF** (23.1 mg, 60%; **6aF**:**7aF** = 92:8). These isomers were separated by reverse-column chromatography on silica gel (MeCN/0.1% TFA aq.) to give, in the order of elution, **6aF** and **7aF**.

Compound **6aF**: pale yellow solid; mp 173–178 °C; IR (neat) 3397, 1742; ¹H NMR (500 MHz, CDCl₃) δ: 1.30 (s, 9H), 7.21 (d, *J* = 3.5 Hz, 1H), 7.29 (s, 1H), 7.32 (t, *J* = 8.0 Hz, 2H), 7.40–7.46 (m, 4H), 7.57 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 4.0 Hz, 1H), 8.16 (s, 1H), 8.24 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.6 (3C), 83.5, 105.5, 110.1, 110.7, 113.8, 119.5, 121.2, 123.2, 124.9, 125.9, 126.6, 127.4 (2C), 127.5, 128.5 (2C), 128.8, 129.1, 136.4, 139.5, 142.7, 149.6; HRMS (ESI) calcd for C₂₅H₂₃N₂O₂ [M+H]⁺: 383.1754, found 383.1755.

Compound **7aF**: white viscous oil; IR (neat) 3592, 1747; ¹H NMR (500 MHz, CDCl₃) δ: 1.72 (s, 9H), 6.84 (d, *J* = 3.5 Hz, 1H), 7.28 (ddd, *J* = 8.0, 6.5, 1.5 Hz, 1H), 7.38–7.44 (m, 4H), 7.50 (t, *J* = 7.0 Hz, 2H), 7.56 (d, *J* = 4.0 Hz, 1H), 7.65 (d, *J* = 7.0 Hz, 2H), 7.98 (d, *J* = 8.5 Hz, 1H), 8.27 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 28.2 (3C), 83.6, 107.6, 108.1, 110.0, 110.4, 119.0, 122.1, 123.2, 124.7, 125.4, 125.9, 127.1, 128.5 (2C), 129.2 (2C), 129.9, 133.3, 139.2, 139.5, 140.8, 150.0; HRMS (ESI) calcd for C₂₅H₂₃N₂O₂ [M+H]⁺: 383.1754, found 383.1754.

tert-Butyl 4-(*o*-Tolyl)pyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6bF**) and Its [3,2-*c*]-Isomer (**7bF**) (Table 5)**

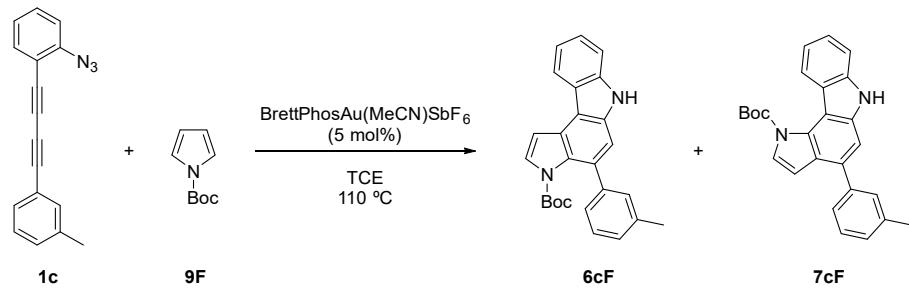


To a solution of **1b** (25.7 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 µmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 0.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 10/1) to give a mixture of **6bF** and **7bF** (22.8 mg, 58%; **6bF**:**7bF** = 95:5). These isomers were separated by column chromatography on amine silica gel (toluene) to give, in the order of elution, **6bF** and **7bF**.

Compound 6bF: pale yellow solid; mp 154–156 °C; IR (neat) 3734, 1730; ¹H NMR (500 MHz, CDCl₃) δ: 1.32 (s, 9H), 2.17 (s, 3H), 7.20 (d, *J* = 3.4 Hz, 1H), 7.21 (s, 1H), 7.23–7.26 (m, 3H), 7.30 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.34–7.37 (m, 1H), 7.42 (dd, *J* = 7.5, 7.5 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 3.4 Hz, 1H), 8.13 (br s, 1H), 8.25 (d, *J* = 7.4 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 20.2, 27.6 (3C), 83.1, 104.9, 110.5, 110.7, 113.8, 119.5, 121.2, 123.3, 124.9, 125.2, 125.6, 126.7, 128.0, 128.1, 128.4, 129.0, 129.9, 135.5, 135.9, 139.5, 142.1, 149.1; HRMS (FAB) calcd for C₂₆H₂₅N₂O₂ [M+H]⁺: 397.1911, found 397.1921.

Compound 7bF: white solid; mp 96–98 °C; IR (neat) 3707, 1732; ¹H NMR (500 MHz, CDCl₃) δ: 1.73 (s, 9H), 2.17 (s, 3H), 6.36 (d, *J* = 4.0 Hz, 1H), 7.23 (s, 1H), 7.28 (m, 2H), 7.32–7.34 (m, 3H), 7.40 (ddd, *J* = 7.4, 7.4, 1.1 Hz, 1H), 7.44 (d, *J* = 7.4 Hz, 1H), 7.49 (d, *J* = 4.0 Hz, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 8.26 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 20.3, 28.2 (3C), 83.5, 108.1, 108.3, 110.0, 110.2, 119.0, 122.1, 124.1, 124.6, 125.1, 125.5, 126.0, 127.5, 129.4, 130.1, 130.3, 133.1, 136.4, 138.9, 139.4, 140.2, 150.1; HRMS (FAB) calcd for C₂₆H₂₅N₂O₂ [M+H]⁺: 397.1911, found 397.1912.

tert-Butyl 4-(*m*-Tolyl)pyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6cF**) and Its [3,2-*c*]-Isomer (**7cF**) (Table 5)**

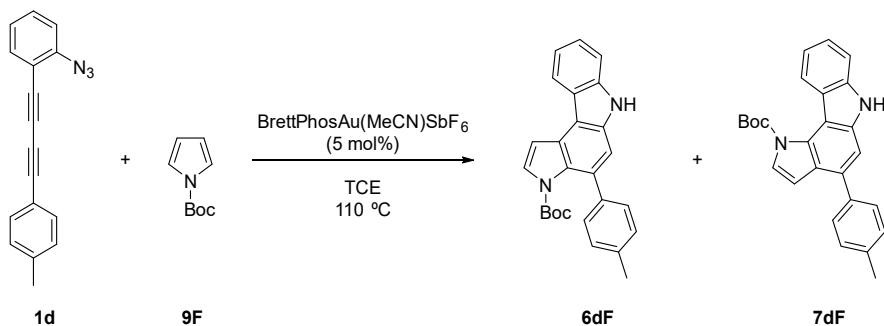


To a solution of **1c** (25.7 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 0.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 10/1) to give a mixture of **6cF** and **7cF** (21.7 mg, 55%; **6cF**:**7cF** = 95:5). These isomers were separated by column chromatography on amine silica gel (hexane/toluene = 1/5) to give, in the order of elution, **6cF** and **7cF**.

Compound 6cF: pale yellow solid; mp 185–186 °C; IR (neat) 3413, 1732; ¹H NMR (500 MHz, CDCl₃) δ: 1.30 (s, 9H), 2.37 (s, 3H), 7.13 (d, *J* = 7.4 Hz, 1H), 7.20 (d, *J* = 3.4 Hz, 1H), 7.26–7.32 (m, 3H), 7.36–7.42 (m, 4H), 7.73 (d, *J* = 3.4 Hz, 1H), 8.13 (br s, 1H), 8.23 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.5, 27.5 (3C), 83.5, 105.4, 110.1, 110.7, 113.7, 119.5, 121.2, 123.2, 124.5, 124.9, 125.8, 127.4, 127.5, 128.1, 128.6, 128.9, 129.1, 136.4, 137.9, 139.5, 142.6, 149.7; HRMS (FAB) calcd for C₂₆H₂₅N₂O₂ [M+H]⁺: 397.1911, found 397.1920.

Compound 7cF: pale yellow viscous oil; IR (neat) 3394, 1743; ¹H NMR (500 MHz, CDCl₃) δ: 1.72 (s, 9H), 2.46 (s, 3H), 6.84 (d, *J* = 4.0 Hz, 1H), 7.23 (d, *J* = 7.4 Hz, 1H), 7.25–7.29 (m, 1H), 7.37 (s, 1H), 7.38–7.47 (m, 5H), 7.55 (d, *J* = 3.4 Hz, 1H), 7.98 (d, *J* = 8.0 Hz, 1H), 8.25 (br s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.6, 28.2 (3C), 83.6, 107.5, 108.2, 110.0, 110.3, 119.0, 122.1, 123.2, 124.7, 125.3, 125.9, 126.3, 127.9, 128.4, 129.9, 129.9, 133.4, 138.1, 139.2, 139.5, 140.7, 150.0; HRMS (FAB) calcd for C₂₆H₂₅N₂O₂ [M+H]⁺: 397.1911, found 397.1909.

tert-Butyl 4-(*p*-Tolyl)pyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6dF**) and Its [3,2-*c*]-Isomer (**7dF**) (Table 5)**

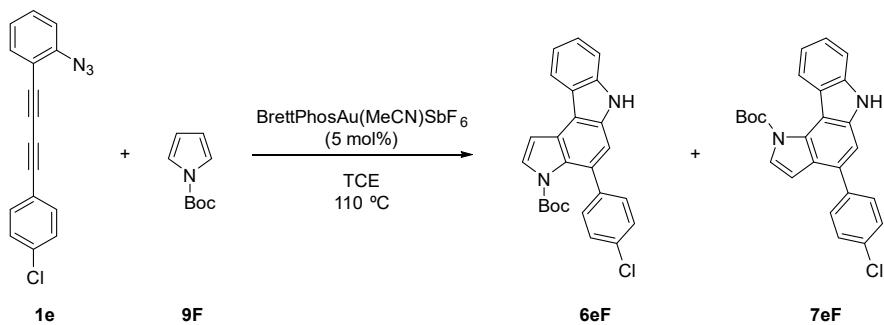


To a solution of **1d** (24.3 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 0.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 10/1) to give a mixture of **6dF** and **7dF** (22.6 mg, 57%; **6dF**:**7dF** = 95:5). These isomers were separated by column chromatography on amine silica gel (toluene) to give, in the order of elution, **6dF** and **7dF**.

Compound **6dF**: pale yellow solid; mp 170–172 °C; IR (neat) 3402, 1736; ¹H NMR (500 MHz, CDCl₃) δ: 1.31 (s, 9H), 2.41 (s, 3H), 7.21 (d, *J* = 3.4 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.29–7.32 (m, 2H), 7.40–7.43 (m, 1H), 7.46–7.49 (m, 3H), 7.72 (d, *J* = 4.0 Hz, 1H), 8.16 (s, 1H), 8.23 (d, *J* = 7.4 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.2, 27.6 (3C), 83.4, 105.4, 109.4, 110.6, 113.7, 119.5, 121.2, 123.3, 124.8, 125.8, 127.3 (2C), 127.7, 128.8, 129.1, 129.2 (2C), 136.2, 136.5, 139.5, 139.8, 149.6; HRMS (FAB) calcd for C₂₆H₂₅N₂O₂ [M+H]⁺: 397.1911, found 397.1912.

Compound **7dF**: white solid; mp 164–166 °C; IR (neat) 3415, 1742; ¹H NMR (500 MHz, CDCl₃) δ: 1.72 (s, 9H), 2.45 (s, 3H), 6.84 (d, *J* = 3.4 Hz, 1H), 7.27 (ddd, *J* = 7.4, 7.4, 1.7 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.36 (s, 1H), 7.38–7.43 (m, 2H), 7.55 (t, *J* = 3.7 Hz, 3H), 7.98 (d, *J* = 8.0 Hz, 1H), 8.25 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 21.2, 28.2 (3C), 83.5, 107.4, 108.2, 110.0, 110.2, 119.0, 122.1, 123.2, 124.6, 125.3, 125.9, 129.1 (2C), 129.3 (2C), 129.9, 133.3, 136.9, 137.9, 139.3, 139.5, 150.0; HRMS (FAB) calcd for C₂₆H₂₅N₂O₂ [M+H]⁺: 397.1911, found 397.1923.

tert-Butyl 4-(4-Chlorophenyl)pyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6eF**) and Its [3,2-*c*]-Isomer (**7eF**) (Table 5)**

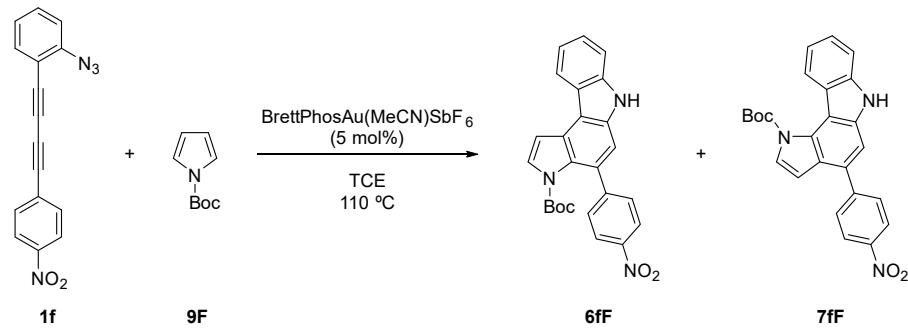


To a solution of **1e** (27.8 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 1.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 10/1) to give a mixture of **6eF** and **7eF** (27.2 mg, 65%; **6eF**:**7eF** = 95:5). These isomers were separated by column chromatography on amine silica gel (toluene) to give, in the order of elution, **6eF** and **7eF**.

Compound **6eF**: pale yellow solid; mp 185–187 °C; IR (neat) 3395, 1740; ¹H NMR (500 MHz, CDCl₃) δ: 1.37 (s, 9H), 7.21 (d, *J* = 3.4 Hz, 1H), 7.26 (s, 1H), 7.32 (dd, *J* = 7.3, 7.3, 1.0 Hz, 1H), 7.39 (dd, *J* = 6.3, 1.7 Hz, 2H), 7.43 (ddd, *J* = 6.5, 6.5, 1.0 Hz, 1H), 7.48 (m, 3H), 7.72 (d, *J* = 4.0 Hz, 1H), 8.18 (s, 1H), 8.24 (d, *J* = 7.4 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.7 (3C), 83.6, 105.6, 110.0, 110.8, 114.1, 119.6, 121.3, 123.1, 125.1, 125.9, 127.4, 128.5 (2C), 128.7 (2C), 129.1, 132.3, 136.4, 139.6, 141.3, 149.3; HRMS (FAB) calcd for C₂₅H₂₂ClN₂O₂ [M+H]⁺: 417.1364, found 417.1373.

Compound **7eF**: white solid; mp 103–105 °C; IR (neat) 3394, 1747; ¹H NMR (500 MHz, CDCl₃) δ: 1.72 (s, 9H), 6.77 (d, *J* = 3.4 Hz, 1H), 7.25–7.31 (m, 1H), 7.31 (s, 1H), 7.41 (d, *J* = 3.4 Hz, 2H), 7.45–7.47 (m, 2H), 7.55–7.57 (m, 3H), 7.98 (d, *J* = 8.0 Hz, 1H), 8.25 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 28.2 (3C), 83.7, 107.4, 107.7, 110.1, 110.6, 119.1, 122.0, 123.0, 124.9, 125.6, 126.0, 128.7 (2C), 123.0, 130.4 (2C), 131.9, 133.1, 139.1, 139.3, 139.5, 150.0; HRMS (FAB) calcd for C₂₅H₂₂ClN₂O₂ [M+H]⁺: 417.1364, found 417.1367.

tert-Butyl 4-(4-Nitrophenyl)pyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6fF**) and Its [3,2-*c*]-Isomer (**7fF**) (Table 5)**

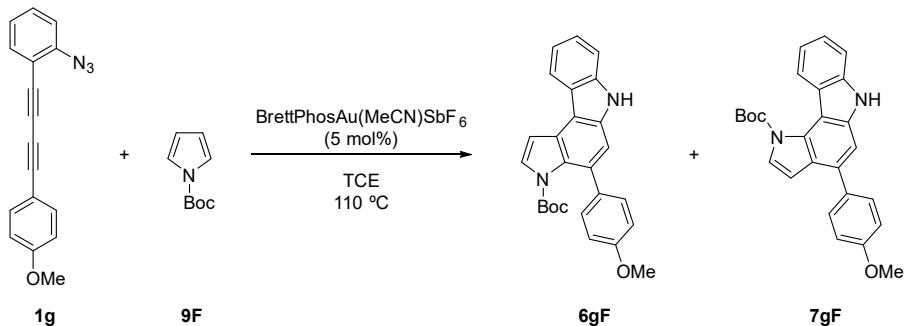


To a solution of **1f** (28.8 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 1.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give a mixture of **6fF** and **7fF** (27.4 mg, 64%; **6fF**:**7fF** = 91:9). These isomers were separated by column chromatography on amine silica gel (hexane/toluene = 1/5) to give, in the order of elution, **6fF** and **7fF**.

Compound **6fF**: yellow solid; mp 186–188 °C; IR (neat) 3396, 1738, 1514; ¹H NMR (500 MHz, CDCl₃) δ: 1.40 (s, 9H), 7.24 (d, *J* = 3.4 Hz, 1H), 7.31 (s, 1H), 7.34 (dd, *J* = 7.3, 1.5 Hz, 1H), 7.46–7.51 (m, 2H), 7.66–7.68 (m, 2H), 7.73 (d, *J* = 3.4 Hz, 1H), 8.26–8.29 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.8 (3C), 83.8, 105.9, 110.2, 110.9, 115.0, 119.9, 121.5, 122.9, 123.6 (2C), 125.6, 126.1, 126.1, 127.1, 128.1 (2C), 129.0, 136.2, 139.8, 146.2, 149.1, 149.8; HRMS (FAB) calcd for C₂₅H₂₂N₃O₄ [M+H]⁺: 428.1605, found 428.1614.

Compound **7fF**: orange solid; mp 189–191 °C; IR (neat) 3415, 1744, 1508; ¹H NMR (500 MHz, CDCl₃) δ: 1.74 (s, 9H), 6.77 (d, *J* = 4.0 Hz, 1H), 7.29–7.32 (m, 1H), 7.40 (s, 1H), 7.44–7.46 (m, 2H), 7.61 (d, *J* = 4.0 Hz, 1H), 7.80 (dd, *J* = 6.6, 2.0 Hz, 2H), 7.99 (d, *J* = 8.0 Hz, 1H), 8.35–8.37 (m, 3H); ¹³C NMR (125 MHz, CDCl₃) δ: 28.1 (3C), 84.0, 107.2, 107.8, 110.2, 111.4, 119.3, 121.8, 122.8, 123.9 (2C), 125.3, 126.1, 126.2, 129.8 (2C), 130.1, 130.4, 139.0, 139.7, 146.8, 147.6, 149.8; HRMS (FAB) calcd for C₂₅H₂₂N₃O₄ [M+H]⁺: 428.1605, found 428.1606.

tert-Butyl 4-(4-Methoxyphenyl)pyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6gF**) and Its [3,2-*c*]-Isomer (**7gF**) (Table 5)**

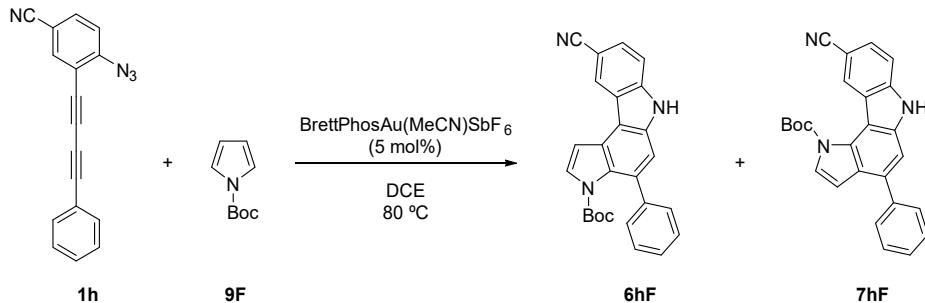


To a solution of **1g** (27.3 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 0.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give a mixture of **6gF** and **7gF** (27.8 mg, 67%; **6gF**:**7gF** = 95:5). These isomers were separated by column chromatography on amine silica gel (toluene) to give, in the order of elution, **6gF** and **7gF**.

Compound **6gF**: white solid; mp 118–120 °C; IR (neat) 3397, 1743; ¹H NMR (500 MHz, CDCl₃) δ: 1.34 (s, 9H), 3.82 (s, 3H), 6.93 (dd, *J* = 6.6, 2.0 Hz, 2H), 7.20 (d, *J* = 4.0 Hz, 1H), 7.24 (s, 1H), 7.30 (ddd, *J* = 7.3, 7.3, 1.3 Hz, 1H), 7.39–7.44 (m, 2H), 7.47 (dd, *J* = 6.3, 2.3 Hz, 2H), 7.71 (d, *J* = 3.4 Hz, 1H), 8.17 (s, 1H), 8.23 (d, *J* = 7.4 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.7 (3C), 55.4, 83.4, 105.5, 109.8, 110.7, 113.5, 114.0 (2C), 119.4, 121.1, 123.2, 124.8, 125.9, 127.7, 128.4 (2C), 128.5, 129.1, 135.3, 136.6, 139.5, 149.6, 158.5; HRMS (FAB) calcd for C₂₆H₂₅N₂O₃ [M+H]⁺: 413.1860, found 413.1861.

Compound **7gF**: white solid; mp 102–105 °C; IR (neat) 3398, 1728; ¹H NMR (500 MHz, CDCl₃) δ: 1.72 (s, 9H), 3.89 (s, 3H), 6.83 (d, *J* = 3.4 Hz, 1H), 7.04 (dd, *J* = 6.3, 2.3 Hz, 2H), 7.26–7.29 (m, 1H), 7.35 (s, 1H), 7.40 (ddd, *J* = 7.4, 7.4, 1.1 Hz, 1H), 7.44 (d, *J* = 7.4 Hz, 1H), 7.55 (d, *J* = 4.0 Hz, 1H), 7.58 (dd, *J* = 6.6, 2.0 Hz, 2H), 7.97 (d, *J* = 7.4 Hz, 1H), 8.26 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 28.2 (3C), 55.4, 83.6, 107.3, 108.1, 110.0, 110.1, 114.0 (2C), 118.9, 122.1, 123.2, 124.6, 125.2, 125.8, 129.9, 130.2 (2C), 133.0, 133.3, 139.3, 139.5, 150.0, 158.9; HRMS (FAB) calcd for C₂₆H₂₅N₂O₃ [M+H]⁺: 413.1860, found 413.1860.

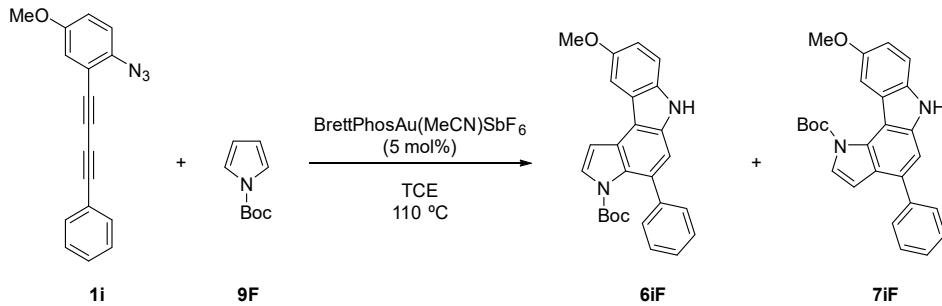
tert-Butyl 9-Cyano-4-phenylpyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6hF**) and Its [3,2-*c*]-Isomer (**7hF**) (Table 5)**



To a solution of **1h** (26.8 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 1 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 3/1) to give an inseparable mixture of **6hF** and **7hF** (24.9 mg, 61%; **6hF**:**7hF** = 81:19): brown solid; IR (neat) 3332, 2218, 1730;

¹H NMR (500 MHz, CDCl₃; mixture of isomers) δ: 1.33 (s, 9H), 6.84 (d, *J* = 4.0 Hz, 0.2H), 7.15 (d, *J* = 4.0 Hz, 0.8H), 7.27 (s, 0.8H), 7.32-7.36 (m, 1.2H), 7.39-7.53 (m, 4.2H), 7.59-7.62 (m, 1H), 7.65 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.76 (d, *J* = 3.4 Hz, 0.8H), 8.32 (s, 0.2H), 8.51 (d, *J* = 1.1 Hz, 0.8H), 8.57 (s, 0.8H), 8.69 (s, 0.2H); ¹³C NMR (125 MHz, CDCl₃; mixture of isomers) δ: 27.6, 28.4, 84.5, 105.0, 107.7, 108.2, 110.7, 111.3, 120.7, 121.0, 125.8, 126.1, 127.0, 127.4, 127.5, 127.6, 128.7, 129.2, 129.7, 131.5, 135.0, 139.5, 140.2, 141.2, 150.1; HRMS (ESI) calcd for C₂₆H₂₂N₃O₂ [M+H]⁺: 408.1712, found 408.1709.

tert-Butyl 9-Methoxy-4-phenylpyrrolo[2,3-*c*]carbazole-3(6*H*)-carboxylate (6iF) and Its [3,2-*c*]-Isomer (7iF) (Table 5)

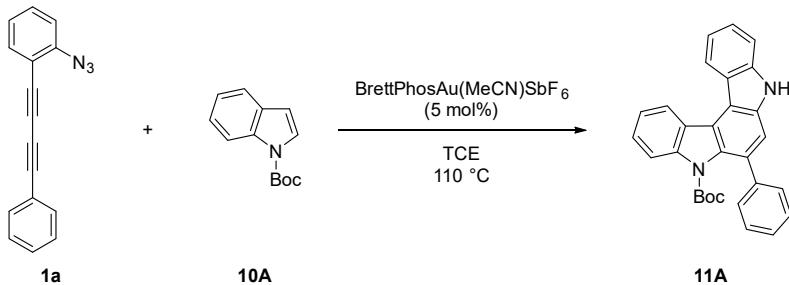


To a solution of **1i** (27.3 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 0.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give a mixture of **6iF** and **7iF** (21.5 mg, 52%; **6iF**:**7iF** = 91:9). These isomers were separated by column chromatography on amine silica gel (toluene/Et₂O = 6/1) to give, in the order of elution, **6iF** and **7iF**.

Compound **6iF**: light brown solid; mp 189–190 °C; IR (neat) 3413, 1747; ¹H NMR (500 MHz, CDCl₃) δ: 1.30 (s, 9H), 3.98 (s, 3H), 7.07 (dd, *J* = 8.9, 2.6 Hz, 1H), 7.18 (d, *J* = 4.0 Hz, 1H), 7.28 (s, 1H), 7.32 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.36 (d, *J* = 9.2 Hz, 1H), 7.42 (dd, *J* = 7.7, 7.7 Hz, 2H), 7.57 (d, *J* = 7.4 Hz, 2H), 7.70 (d, *J* = 2.3 Hz, 1H), 7.72 (d, *J* = 4.0 Hz, 1H), 8.03 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.6 (3C), 56.2, 83.5, 104.4, 105.3, 110.3, 111.3, 113.8, 113.9, 123.7, 125.8, 126.6, 127.3, 127.4 (2C), 128.5 (2C), 128.8, 129.0, 134.6, 137.3, 142.7, 149.6, 153.9; HRMS (FAB) calcd for C₂₆H₂₅N₂O₃ [M+H]⁺: 413.1860, found 413.1869.

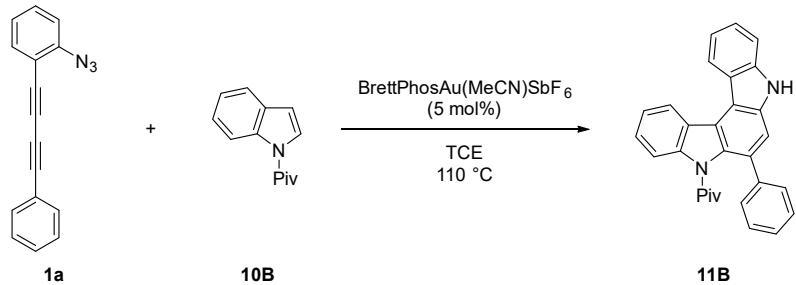
Compound **7iF**: white solid; mp 145–146 °C; IR (neat) 3589, 1734; ¹H NMR (500 MHz, CDCl₃) δ: 1.74 (s, 9H), 3.95 (s, 3H), 6.83 (d, *J* = 4.0 Hz, 1H), 7.08 (dd, *J* = 8.6, 2.9 Hz, 1H), 7.36 (d, *J* = 8.6 Hz, 1H), 7.40 (s, 1H), 7.41–7.43 (m, 1H), 7.49–7.52 (m, 2H), 7.55–7.56 (m, 2H), 7.64–7.67 (m, 2H), 8.19 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 28.2 (3C), 55.9, 83.4, 107.8, 108.3, 108.9, 110.5, 110.6, 114.5, 122.6, 123.0, 125.2, 127.1, 128.5 (2C), 129.2 (2C), 129.9, 133.2, 134.6, 140.1, 140.8, 150.1, 153.3; HRMS (FAB) calcd for C₂₆H₂₅N₂O₃ [M+H]⁺: 413.1860, found 413.1867.

tert-Butyl 6-Phenylindolo[2,3-*c*]carbazole-5(8*H*)-carboxylate (11A) (Table 6)



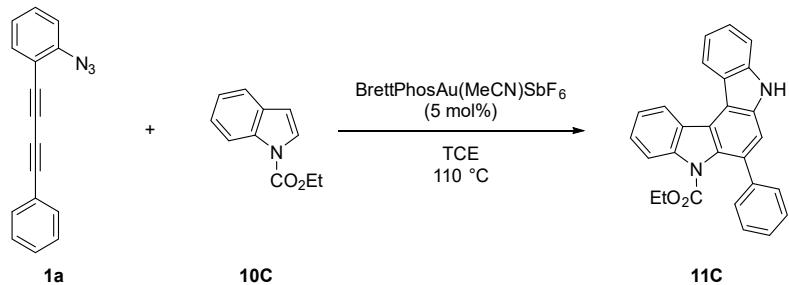
To a solution of **1a** (24.3 mg, 0.100 mmol) and **10A** (109 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 3 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/toluene = 1/1) to give **11A** (15.1 mg, 35%): white powder; mp 194–195 °C; IR (neat) 3412, 1750; ¹H NMR (500 MHz, CDCl₃) δ: 1.23 (s, 9H), 7.33 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.36–7.39 (m, 1H), 7.44–7.46 (m, 5H), 7.51–7.57 (m, 2H), 7.67–7.68 (m, 2H), 8.25 (dd, *J* = 7.2, 2.0 Hz, 1H), 8.35 (s, 1H), 8.78 (d, *J* = 8.0 Hz, 1H), 8.82–8.83 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.5 (3C), 83.6, 110.9, 112.1, 114.6, 115.6, 119.4, 122.4, 122.6, 122.7, 122.9, 123.4, 125.4, 126.2, 126.5, 126.9, 127.2 (2C), 129.1 (2C), 129.4, 131.6, 137.4, 140.0, 140.5, 142.2, 151.1; HRMS (ESI) calcd for C₂₉H₂₅N₂O₂ [M+H]⁺: 433.1916, found 433.1912.

2,2-Dimethyl-1-[6-phenylindolo[2,3-*c*]carbazol-5(8*H*)-yl]propan-1-one (**11B**) (Table 6)



To a solution of **1a** (24.3 mg, 0.100 mmol) and **10B** (101 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 6.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/toluene = 1/1) to give **11B** (4.90 mg, 12%): yellow solid; mp 227–230 °C; IR (neat) 3412, 1692; ¹H NMR (500 MHz, CDCl₃) δ: 0.63 (s, 9H), 7.40–7.43 (m, 2H), 7.49–7.51 (m, 6H), 7.58 (dd, *J* = 8.0, 8.0 Hz, 2H), 7.74–7.75 (m, 2H), 8.38 (s, 1H), 8.86 (d, *J* = 8.6 Hz, 1H), 8.89–8.90 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.7 (3C), 46.4, 110.9, 111.4, 111.6, 116.1, 119.52, 119.55, 121.1, 122.9, 123.1, 123.6 (2C), 125.5, 126.1, 127.4, 128.0, 129.1 (2C), 130.5 (2C), 132.7, 136.4, 138.8, 139.5, 140.0, 186.7; HRMS (ESI) calcd for C₂₉H₂₅N₂O [M+H]⁺: 417.1967, found 417.1977

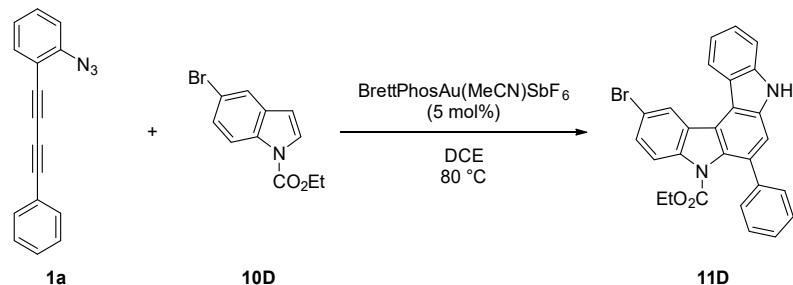
Ethyl 6-Phenylindolo[2,3-*c*]carbazole-5(8*H*)-carboxylate (**11C**) (Table 6)



To a solution of **1a** (24.3 mg, 0.100 mmol) and **10C** (94.6 mg, 0.500 mmol) in TCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 110 °C. The mixture was stirred at 110 °C in pre-heated bath for 6.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/toluene = 1/2) to give **11C** (17.9 mg, 44%): white powder; mp 226–228 °C; IR (neat) 3410, 1725; ¹H NMR (500 MHz, CDCl₃) δ: 0.94 (t, *J* = 7.2 Hz, 3H), 3.59 (q, *J* = 7.3 Hz, 2H), 7.32–7.35 (m, 1H), 7.36–7.40 (m, 1H), 7.45–7.47 (m, 5H), 7.52–7.59 (m, 2H), 7.63 (dd, *J* = 8.0, 1.1 Hz, 2H), 8.32–8.34 (m, 2H), 8.78 (d, *J* = 7.4 Hz, 1H), 8.82–8.83 (m, 1H); ¹³C NMR (125 MHz, CDCl₃) δ:

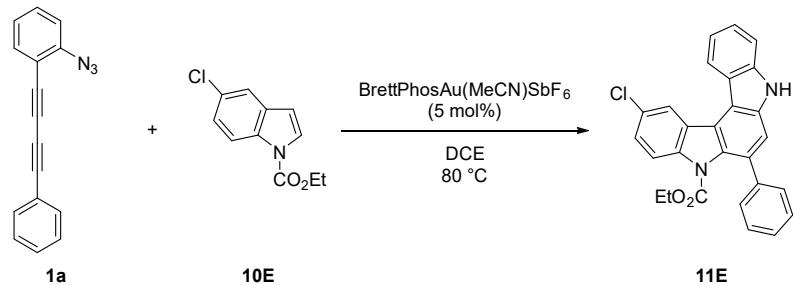
CDCl_3) δ : 13.9, 62.9, 110.9, 112.0, 114.6, 115.7, 119.5, 122.3, 122.6, 122.9 (2C), 123.5, 125.6, 125.9, 126.7, 126.9 (3C), 129.0 (3C), 131.1, 137.4, 140.05, 140.11, 142.3, 152.5; HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{21}\text{N}_2\text{O}_2$ [$\text{M}+\text{H}]^+$: 405.1603, found 405.1594.

Ethyl 2-Bromo-6-phenylindolo[2,3-*c*]carbazole-5(8*H*)-carboxylate (**11D**) (Table 6)



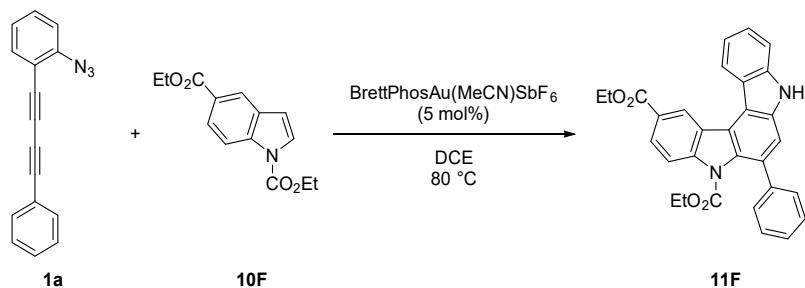
To a solution of **1a** (24.3 mg, 0.100 mmol) and **10D** (134 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 4.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give **11D** (24.0 mg, 50%): white powder; mp 246–248 °C; IR (neat) 3420, 1729; ¹H NMR (500 MHz, CDCl_3) δ : 0.95 (t, *J* = 7.2 Hz, 3H), 3.59 (q, *J* = 7.3 Hz, 2H), 7.37 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.42–7.45 (m, 1H), 7.49–7.54 (m, 4H), 7.55 (s, 1H), 7.64–7.66 (m, 3H), 8.19 (d, *J* = 8.6 Hz, 1H), 8.39 (s, 1H), 8.66 (d, *J* = 8.0 Hz, 1H), 8.90 (d, *J* = 1.7 Hz, 1H); ¹³C NMR (125 MHz, CDCl_3) δ : 13.9, 63.1, 111.0, 112.8, 115.7, 115.8, 116.0, 119.9, 121.2, 122.4, 123.3, 125.4, 125.9, 126.9 (2C), 127.1, 127.7, 129.0, 129.1 (2C), 129.4, 131.6, 137.3, 138.8, 140.0, 142.0, 152.1; HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{20}\text{BrN}_2\text{O}_2$ [$\text{M}+\text{H}]^+$: 483.0708, found 483.0695.

Ethyl 2-Chloro-6-phenylindolo[2,3-*c*]carbazole-5(8*H*)-carboxylate (**11E**) (Table 6)



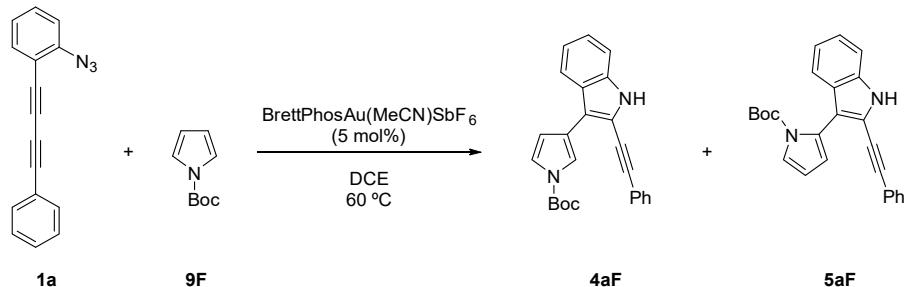
To a solution of **1a** (24.3 mg, 0.100 mmol) and **10E** (112 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 4.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give **11E** (23.0 mg, 52%): white powder; mp 241–243 °C; IR (neat) 3420, 1728; ¹H NMR (500 MHz, CDCl_3) δ : 0.95 (t, *J* = 7.2 Hz, 3H), 3.59 (q, *J* = 7.1 Hz, 2H), 7.36–7.39 (m, 1H), 7.42–7.45 (m, 1H), 7.51–7.53 (m, 5H), 7.56 (s, 1H), 7.65 (dd, *J* = 6.6, 1.4 Hz, 2H), 8.24 (d, *J* = 9.2 Hz, 1H), 8.40 (s, 1H), 8.69 (d, *J* = 8.0 Hz, 1H), 8.76 (d, *J* = 2.3 Hz, 1H); ¹³C NMR (125 MHz, CDCl_3) δ : 13.9, 63.1, 111.0, 112.8, 115.6, 115.7, 115.8, 119.9, 121.7, 122.4, 122.5, 123.4, 125.9, 126.6, 126.9 (2C), 127.1, 128.3, 129.0, 129.1 (3C), 131.7, 137.3, 138.5, 140.1, 142.1, 152.2; HRMS (ESI) calcd for $\text{C}_{27}\text{H}_{20}\text{ClN}_2\text{O}_2$ [$\text{M}+\text{H}]^+$: 439.1213, found 439.1195.

Diethyl 6-Phenylindolo[2,3-*c*]carbazole-2,5(8*H*)-dicarboxylate (11F**) (Table 6)**



To a solution of **1a** (24.3 mg, 0.100 mmol) and **10F** (131 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 80 °C. The mixture was stirred at 80 °C in pre-heated bath for 7.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give **11F** (31.8 mg, <67%): white powder; mp 292–294 °C; IR (neat) 3342, 1698; ¹H NMR (500 MHz, CDCl₃) δ: 0.97 (t, *J* = 7.2 Hz, 3H), 1.54 (t, *J* = 7.2 Hz, 3H), 3.60 (q, *J* = 7.3 Hz, 2H), 4.53 (q, *J* = 7.1 Hz, 2H), 7.39 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.46 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.51–7.60 (m, 4H), 7.61 (s, 1H), 7.68 (d, *J* = 7.4 Hz, 2H), 8.27 (dd, *J* = 8.6, 1.1 Hz, 1H), 8.31 (d, *J* = 8.6 Hz, 1H), 8.44 (s, 1H), 8.89 (d, *J* = 8.0 Hz, 1H), 9.61 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 13.9, 14.5, 61.1, 63.3, 111.0, 112.6, 114.0, 115.9, 120.0, 121.9, 122.5, 123.6, 125.0 (2C), 125.7, 126.0, 127.0 (3C), 127.2, 128.0, 128.9, 129.1 (2C), 131.6, 137.5, 140.2, 142.0, 152.1, 167.0; HRMS (ESI) calcd for C₃₀H₂₅N₂O₄ [M+H]⁺: 477.1814, found 477.1824

tert-Butyl 3-[2-(Phenylethynyl)-1*H*-indol-3-yl]-1*H*-pyrrole-1-carboxylate (4aF**) and Its 2-Indolyl Isomer (**5aF**) (Scheme 5)**



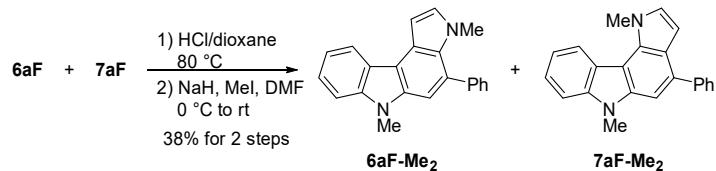
To a solution of **1a** (24.3 mg, 0.100 mmol) and **9F** (83.6 mg, 0.500 mmol) in DCE (0.5 mL) was added [BrettPhosAu(MeCN)SbF₆] (5.05 mg, 5.00 μmol) at 60 °C. The mixture was stirred at 60 °C in pre-heated bath for 1.5 h and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 10/1) to give a mixture of **4aF** and **5aF** (9.40 mg, 25%; **4aF:5aF** = 69:31). These isomers were separated by column chromatography on silica gel (hexane/toluene = 1/2) to give, in the order of elution, **4aF** and **5aF**.

Compound **4aF**: brown viscous oil; IR (neat) 3404, 2978, 1738; ¹H NMR (500 MHz, CDCl₃) δ: 1.63 (s, 9H), 6.96 (dd, *J* = 3.2, 1.4 Hz, 1H), 7.20 (ddd, *J* = 4.0, 4.0, 1.5 Hz, 1H), 7.29 (ddd, *J* = 3.5, 3.5, 1.0 Hz, 1H), 7.34–7.40 (m, 5H), 7.55–7.57 (m, 2H), 7.80 (s, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 8.21 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 28.0 (3C), 82.4, 83.6, 95.7, 110.9, 112.1, 115.4, 116.2, 116.9, 120.2, 120.3, 120.4, 120.6, 122.8, 123.9, 125.9, 128.5, 128.5 (2C), 131.3 (2C), 136.2, 148.9; HRMS (FAB) calcd for C₂₅H₂₂N₂O₂ [M⁺]: 382.1681, found 382.1677.

Compound **5aF**: brown viscous oil; IR (neat) 3370, 2979, 1734; ¹H NMR (500 MHz, CDCl₃) δ: 1.19 (s, 9H), 6.34 (dd, *J* = 3.2, 3.2 Hz, 1H), 6.44 (dd, *J* = 3.4, 1.7 Hz, 1H), 7.13 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 1H), 7.31–7.34 (m, 4H), 7.44–7.46 (m, 2H), 7.48–7.50 (m, 2H), 8.22 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 27.3 (3C),

81.7, 83.2, 94.8, 110.7, 110.8, 115.5, 116.3, 117.3, 120.4, 120.5, 122.6, 122.6, 123.7, 126.2, 127.8, 128.3 (2C), 128.4, 131.4 (2C), 135.4, 149.5; HRMS (FAB) calcd for C₂₅H₂₂N₂O₂ [M⁺]: 382.1681, found 382.1678.

3,6-Dimethyl-4-phenyl-3,6-dihydropyrrolo[2,3-*c*]carbazole (**6aF-Me₂**) and Its [3,2-*c*]-Isomer (**7aF-Me₂**)

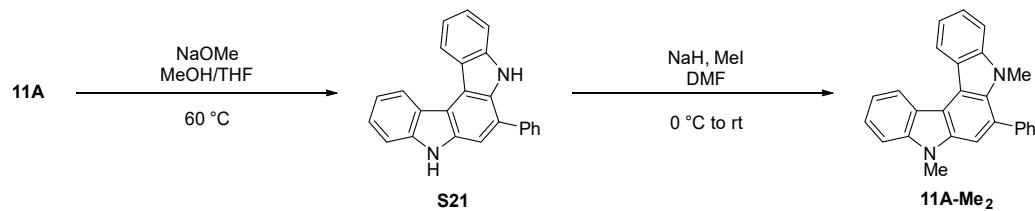


To a solution of **6aF** and **7aF** (50.0 mg, 0.130 mmol; **6aF**:**7aF** = 16:84) in 4 M HCl/dioxane (5.7 mL) was stirred at 80 °C for 16 min. The solvent was removed under vacuum. The crude was then poured into aqueous NaHCO₃, extracted with EtOAc, dried over Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by amine silica gel (hexane/CHCl₃ = 2/3) to give an isomeric mixture of **6aA** and **7aA** (22.0 mg, 60%). To a solution of this mixture (obtained by repeated reactions; 75.1 mg, 0.266 mmol) in DMF (0.912 mL) was added NaH (60% dispersion in paraffin liquid; 84.8 mg, 2.12 mmol) at 0 °C under Ar. The reaction mixture was stirred at room temperature for 30 min. MeI (0.132 ml, 2.12 mmol) was added to the reaction mixture. After being stirred for 14 h, the mixture was quenched with H₂O, extracted with CHCl₃, washed with brine, dried over Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 30/1) to give **6aF-Me₂** and **7aF-Me₂** (52.8 mg, 64%; **6aF-Me₂**: **7aF-Me₂** = 12:88). These isomers were separated by careful column chromatography on silica gel (hexane/EtOAc = 30/1) to give, in the order of elution, **6aF-Me₂** and **7aF-Me₂**.

Compound **6aF-Me₂**: white powder; mp 140–142 °C; ¹H NMR (500 MHz, CDCl₃) δ: 3.39 (s, 3H), 3.91 (s, 3H), 7.09–7.15 (m, 3H), 7.26–7.31 (m, 1H), 7.44–7.46 (m, 5H), 7.56 (d, *J* = 6.9 Hz, 2H), 8.30 (d, *J* = 7.4 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 29.4, 37.2, 99.1, 106.3, 112.9, 118.4, 121.2, 122.9, 123.0, 123.9, 126.0, 127.2, 127.3, 127.6 (2C), 129.3, 130.3 (2C), 131.1, 135.5, 140.4, 141.2; HRMS (ESI) calcd for C₂₂H₁₉N₂ [M+H]⁺: 311.1548, found 311.1556.

Compound **7aF-Me₂**: white powder; mp 111–113 °C; ¹H NMR (500 MHz, CDCl₃) δ: 3.96 (s, 3H), 4.55 (s, 3H), 6.74 (d, *J* = 2.9 Hz, 1H), 7.03 (d, *J* = 3.4 Hz, 1H), 7.23 (ddd, *J* = 7.6, 7.6, 1.3 Hz, 1H), 7.27 (s, 1H), 7.40–7.53 (m, 5H), 7.77–7.78 (m, 2H), 8.56 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃) δ: 29.5, 39.0, 102.0, 102.7, 106.7, 108.6, 118.6, 120.7, 121.8, 122.3, 123.7, 126.9, 127.0, 127.5, 128.4 (2C), 129.1 (2C), 132.3, 133.8, 139.2, 141.7; HRMS (ESI) calcd for C₂₂H₁₉N₂ [M+H]⁺: 311.1548, found 311.1545.

5,8-Dimethyl-6-phenyl-5,8-dihydroindolo[2,3-*c*]carbazole (**11A-Me₂**)

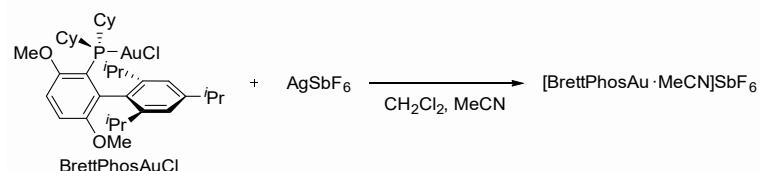


To a solution of **11A** (121 mg, 0.280 mmol) in THF (2.8 mL) was added NaOMe (5 M in MeOH) (1.68 mL, 8.39 mmol) at 60 °C. The mixture was stirred at 60 °C for 60 h. The mixture was quenched with sat. NaHCO₃ and the whole was extracted with Et₂O, dried over Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 5/1) to give **S21** (52.2 mg, 56%) as a solid. To a solution of this solid (52.2 mg, 0.157 mmol) in DMF (0.502 mL) was added NaH (60% dispersion in paraffin liquid; 50.2 mg, 1.26 mmol)

at 0 °C under Ar. After being stirred at room temperature for 10 min, MeI (0.0391 ml, 0.628 mmol) was added to the mixture. After being stirred for 40 h, the mixture was quenched with H₂O, extracted with CHCl₃, washed with brine, dried over Na₂SO₄, filtered, and concentrated in vacuo. The residue was purified by column chromatography (hexane/EtOAc = 30/1) to give **11A-Me₂** (36.1 mg, 64%): yellow viscous oil; ¹H NMR (500 MHz, CD₂Cl₂) δ: 3.46 (s, 3H), 3.97 (s, 3H), 7.40-7.44 (m, 3H), 7.47-7.57 (m, 7H), 7.62 (dd, *J* = 8.3, 1.4 Hz, 2H), 8.93 (d, *J* = 8.0 Hz, 1H), 8.96 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (125 MHz, CD₂Cl₂) δ: 29.4, 33.7, 108.5, 109.0, 110.1, 115.5, 117.9, 118.4, 118.6, 122.3, 122.5, 123.4, 123.5, 124.8, 125.0, 125.1, 127.3, 127.9 (2C), 130.1 (2C), 134.1, 135.9, 141.1, 141.4, 142.3; HRMS (ESI) calcd for C₂₆H₂₁N₂ [M+H]⁺: 361.1705, found 361.1690.

4. Preparation of the Catalyst

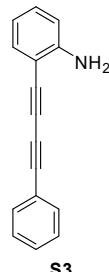
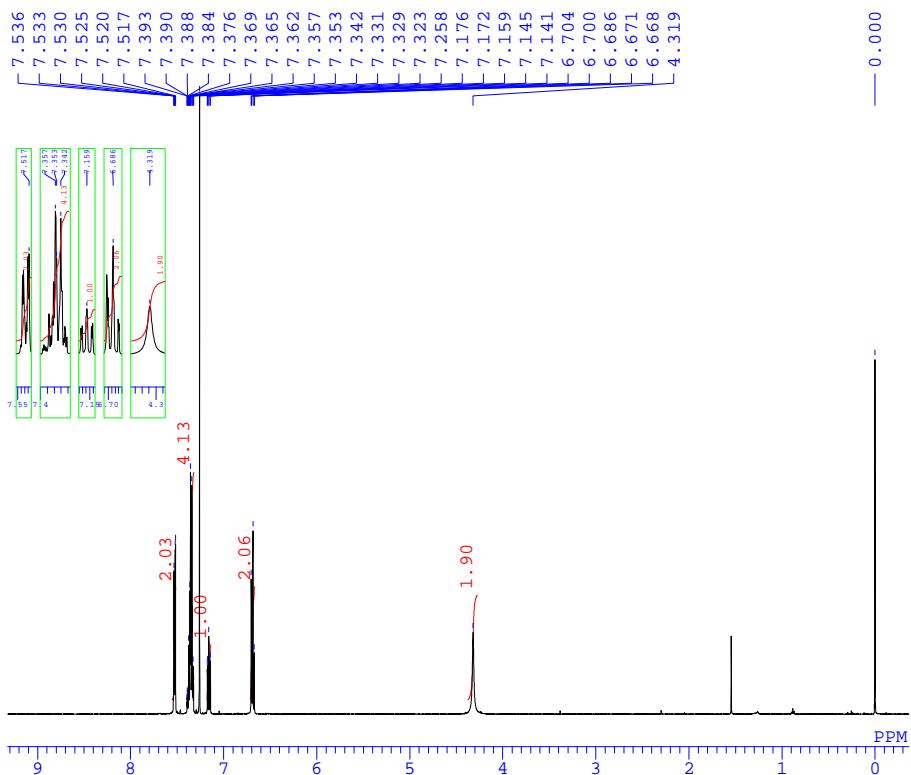
[BrettPhosAu·MeCN]SbF₆⁶



This catalyst was prepared according to the literature procedure for the synthesis of $[\text{LAu}\cdot\text{MeCN}]\text{SbF}_6$ ($\text{L} = \{2\text{-}[2,4,6-(i\text{-Pr})_3\text{C}_6\text{H}_2]\text{C}_6\text{H}_4\}\text{P}(t\text{-Bu})_2$). AgSbF_6 (0.6 M solution in CH_2Cl_2 ; 1.98 mL, 1.19 mmol) was added to a stirred solution of chloro[2-(dicyclohexylphosphino)-3,6-dimethoxy-2',4',6'-triisopropyl-1,1'-biphenyl]gold(I) (Brett-PhosAuCl) (897 mg, 1.17 mmol) in MeCN (7.5 mL) and CH_2Cl_2 (7.5 mL), and the mixture was stirred at room temperature in the dark (using aluminium foil) for 8 h. The mixture was filtered through a pad of Celite and the solvent was removed in vacuo to afford a white powder (1.29 g, quant): ^1H NMR (500 MHz, CDCl_3) δ : 0.90 (d, $J = 6.3$ Hz, 6H), 1.07-1.09 (m, 2H), 1.17-1.24 (m, 4H), 1.27 (d, $J = 6.9$ Hz, 6H), 1.33 (d, $J = 6.9$ Hz, 6H), 1.37-1.40 (m, 4H), 1.49-1.50 (m, 2H), 1.67-1.98 (m, 8H), 2.25-2.30 (m, 2H), 2.37 (s, 3H), 2.55-2.59 (m, 2H), 2.92-2.98 (m, 1H), 3.56 (s, 3H), 3.94 (s, 3H), 6.95-7.12 (m, 4H); ^{13}C NMR (125 MHz, CDCl_3) δ : 2.52, 24.1 (d, $J = 19.2$ Hz, 4C), 24.8 (2C), 25.6 (2C), 26.5 (d, $J = 16.8$ Hz, 2C), 27.0 (d, $J = 13.2$ Hz, 2C), 30.0 (2C), 30.6 (2C), 33.7, 34.6, 34.7, 38.1 (d, $J = 36.0$ Hz, 2C), 54.9, 56.0, 110.8 (d, $J = 7.2$ Hz), 114.2, 114.7, 115.1, 118.9, 121.6 (2C), 131.5 (d, $J = 8.4$ Hz), 136.7 (d, $J = 13.2$ Hz), 147.3, 149.2, 153.1 (d, $J = 10.8$ Hz), 154.8; HRMS (FAB) calcd for $\text{C}_{35}\text{H}_{53}\text{AuO}_2\text{P}^+ [\text{M}-\text{MeCN}-\text{SbF}_6]^+$: 733.3443, found 733.3444.

5. References

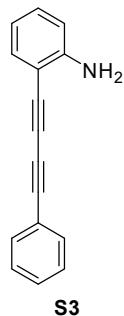
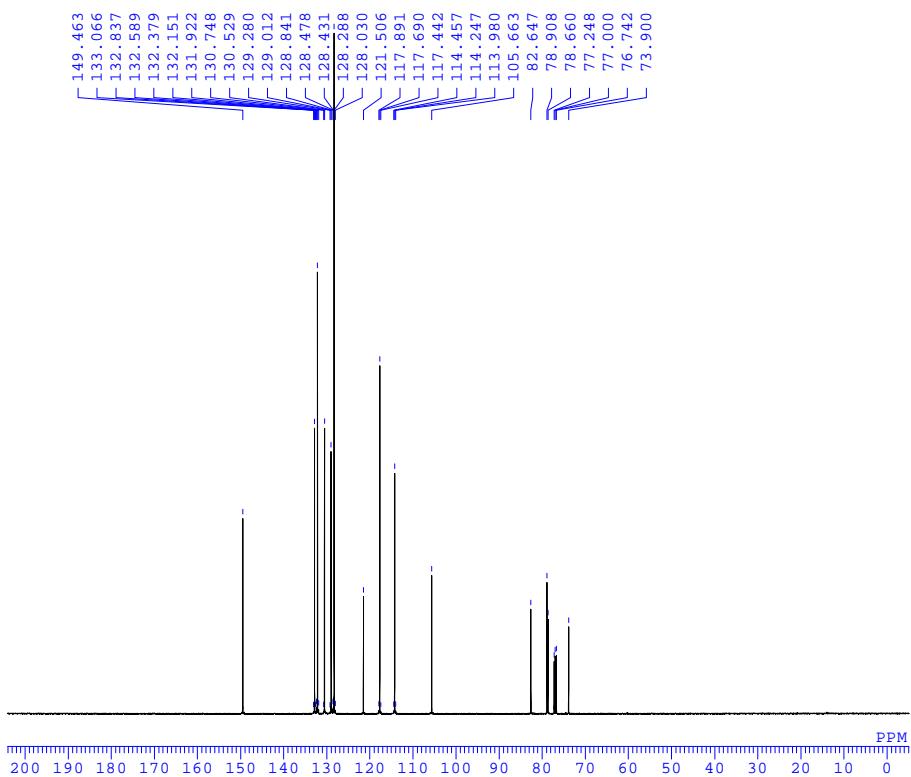
- (1) Matsuoka, J.; Matsuda, Y.; Kawada, Y.; Oishi, S.; Ohno, H. *Angew. Chem. Int. Ed.* **2017**, *56*, 7444–7448.
 - (2) Sakai, N.; Annaka, K.; Konakahara, T. *J. Org. Chem.* **2006**, *71*, 3653–3655.
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 - (6) Obradors, C.; Leboeuf, D.; Aydin, J.; Echavarren, A. M. *Org. Lett.* **2013**, *15*, 1576.



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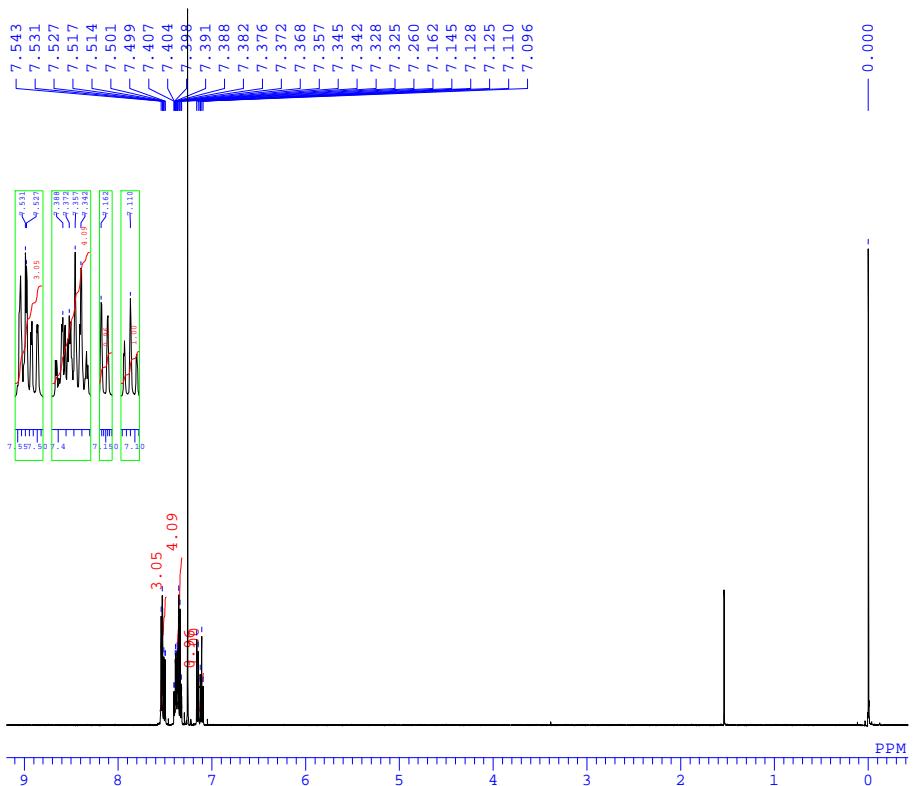
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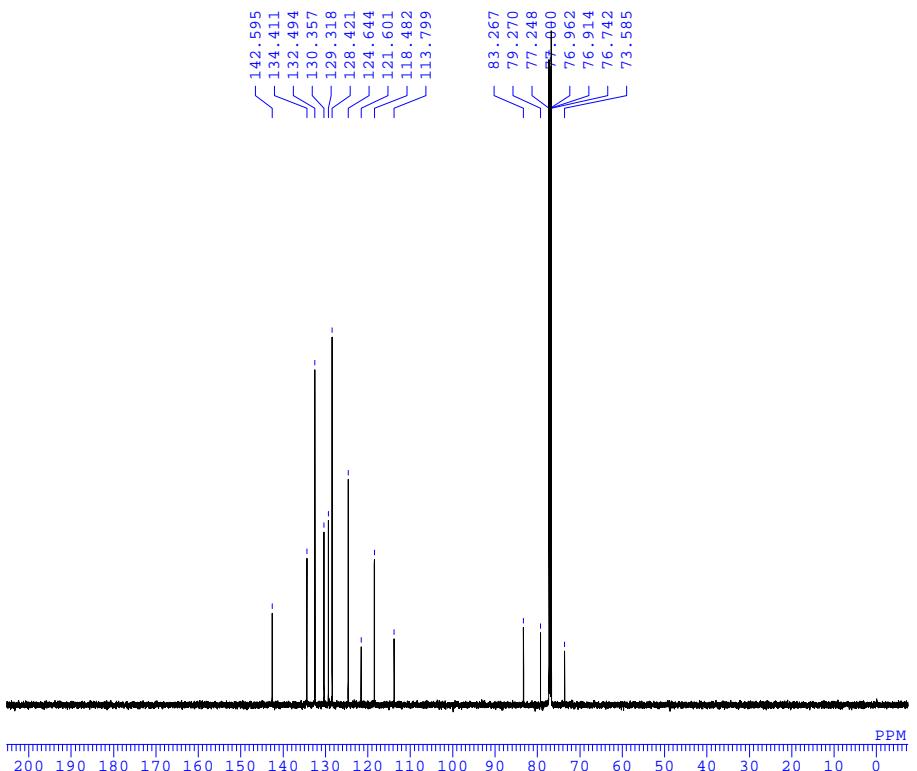
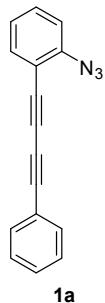
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EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54

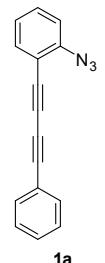
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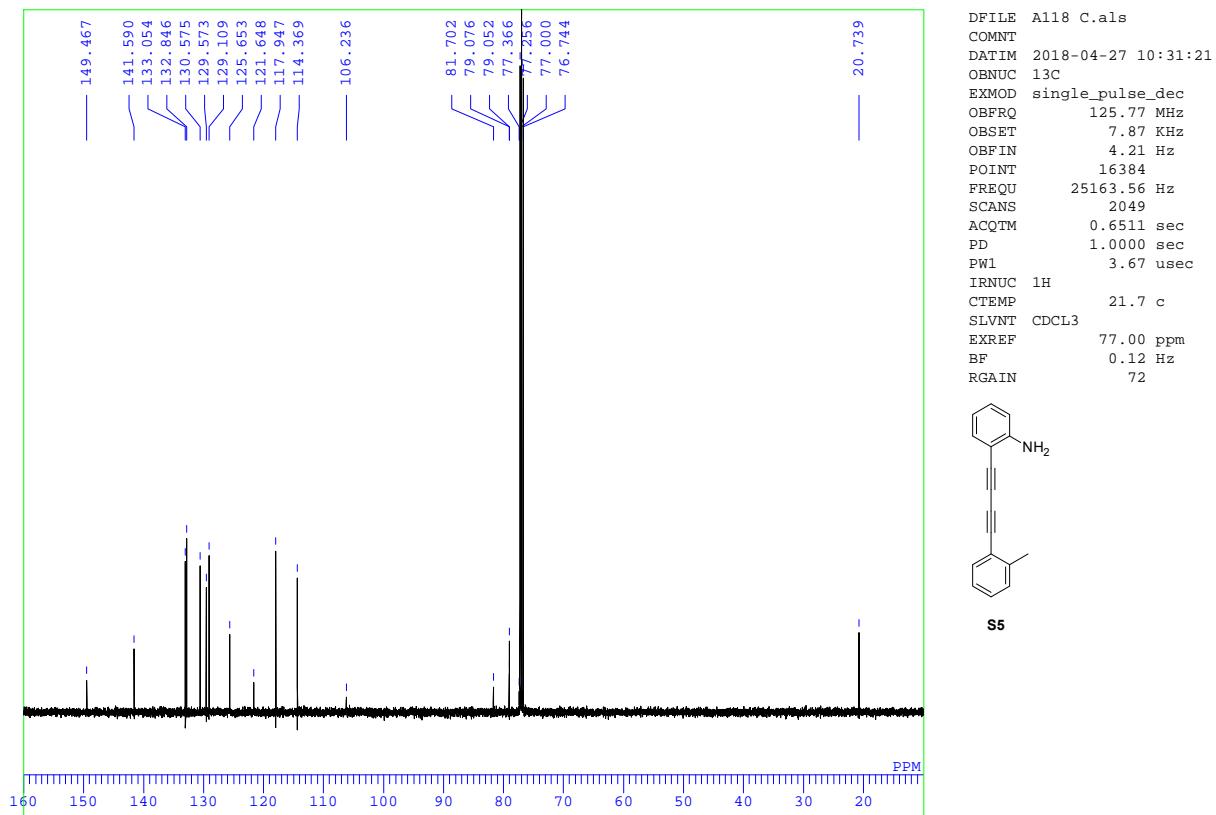
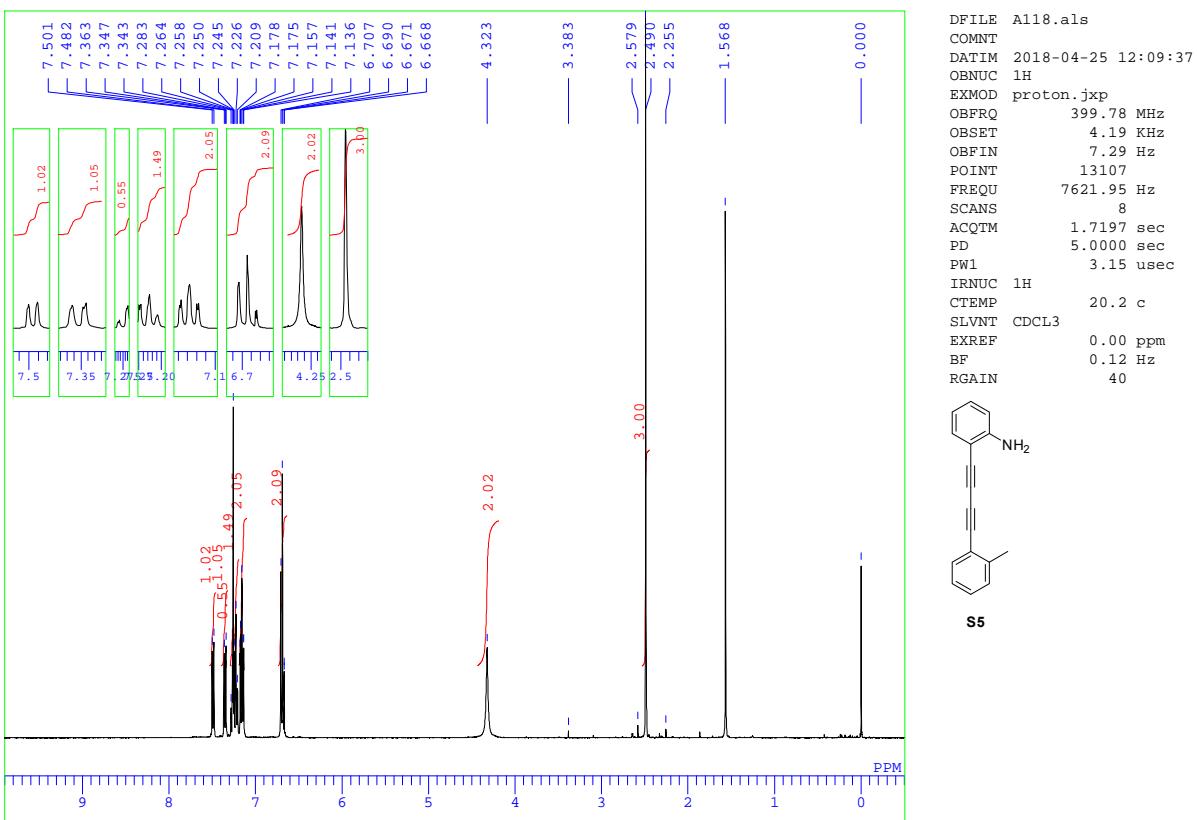


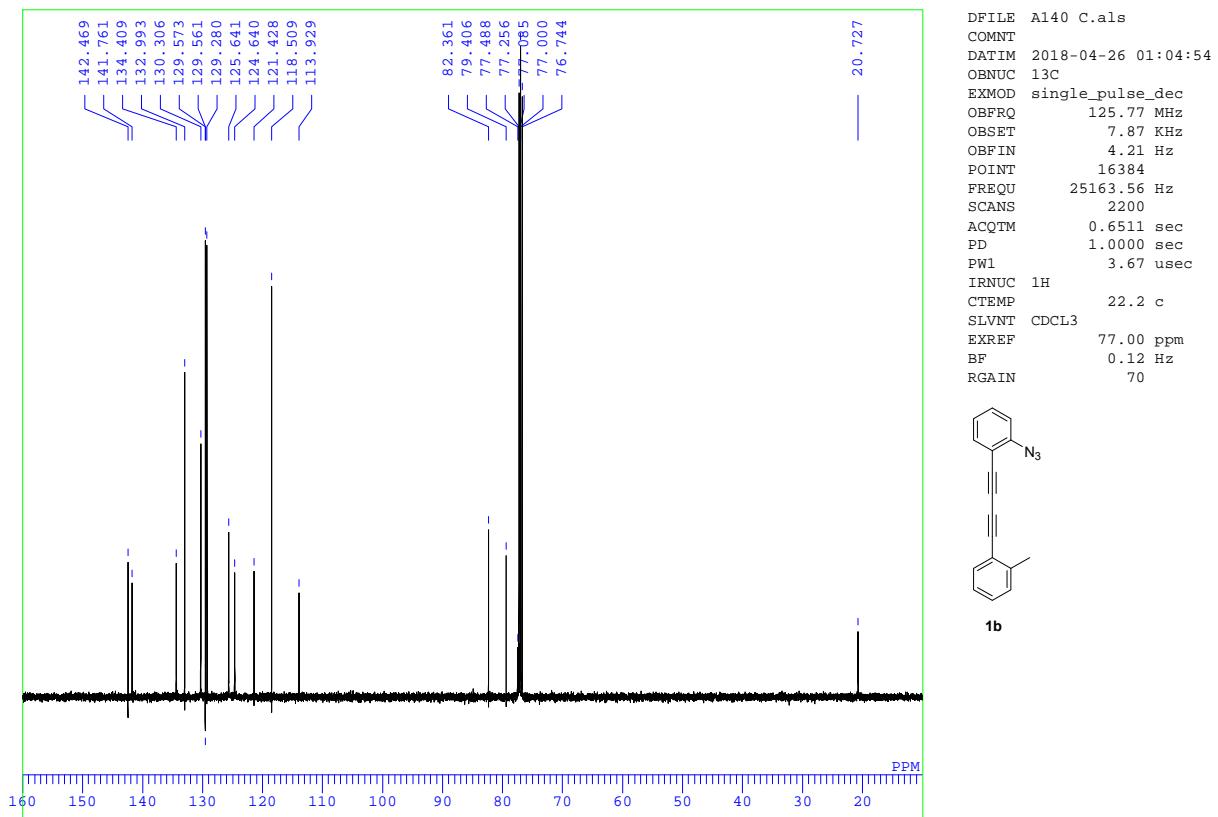
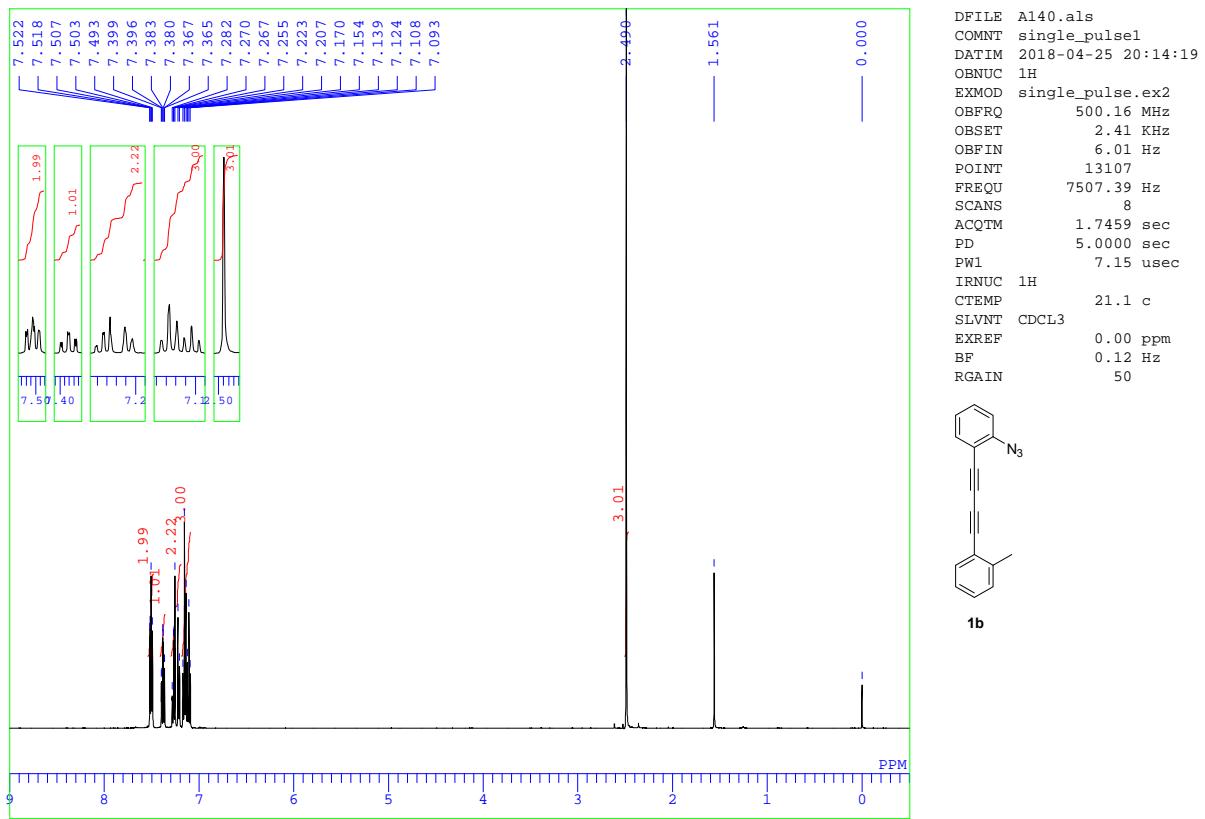
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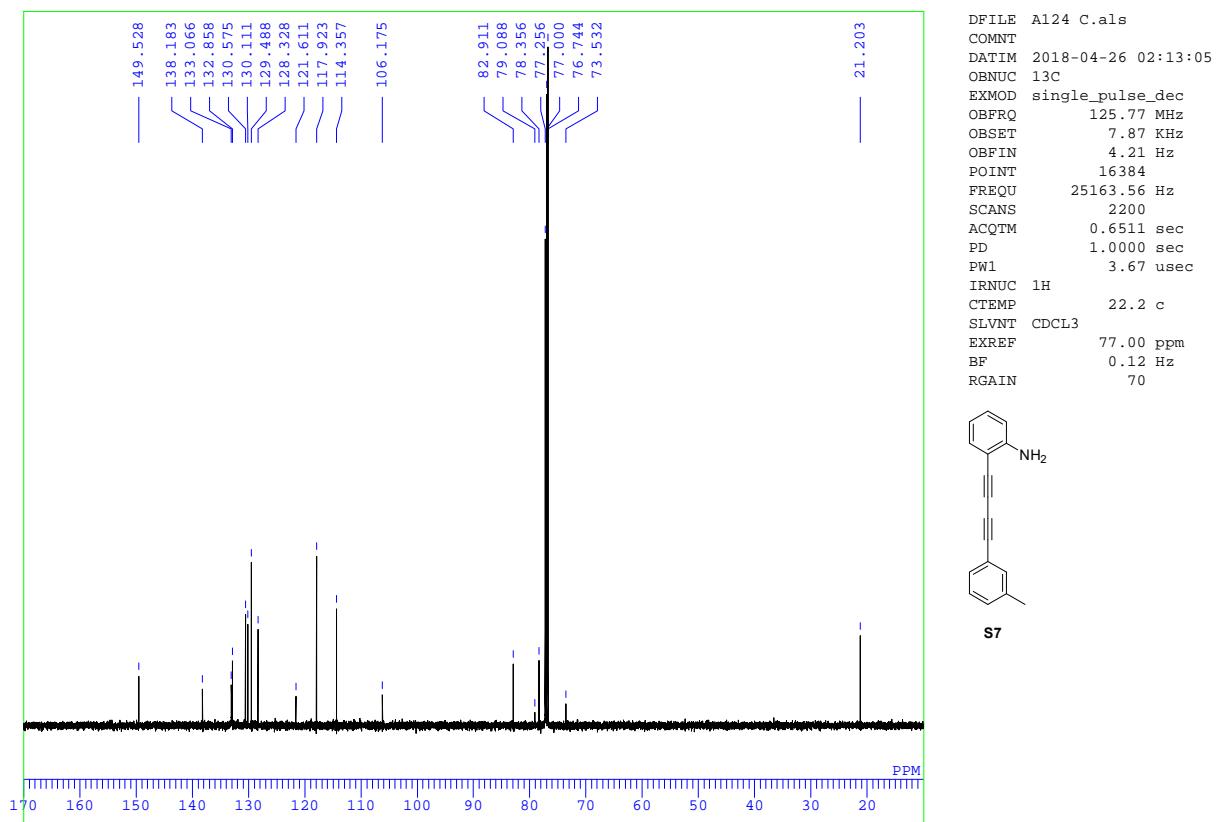
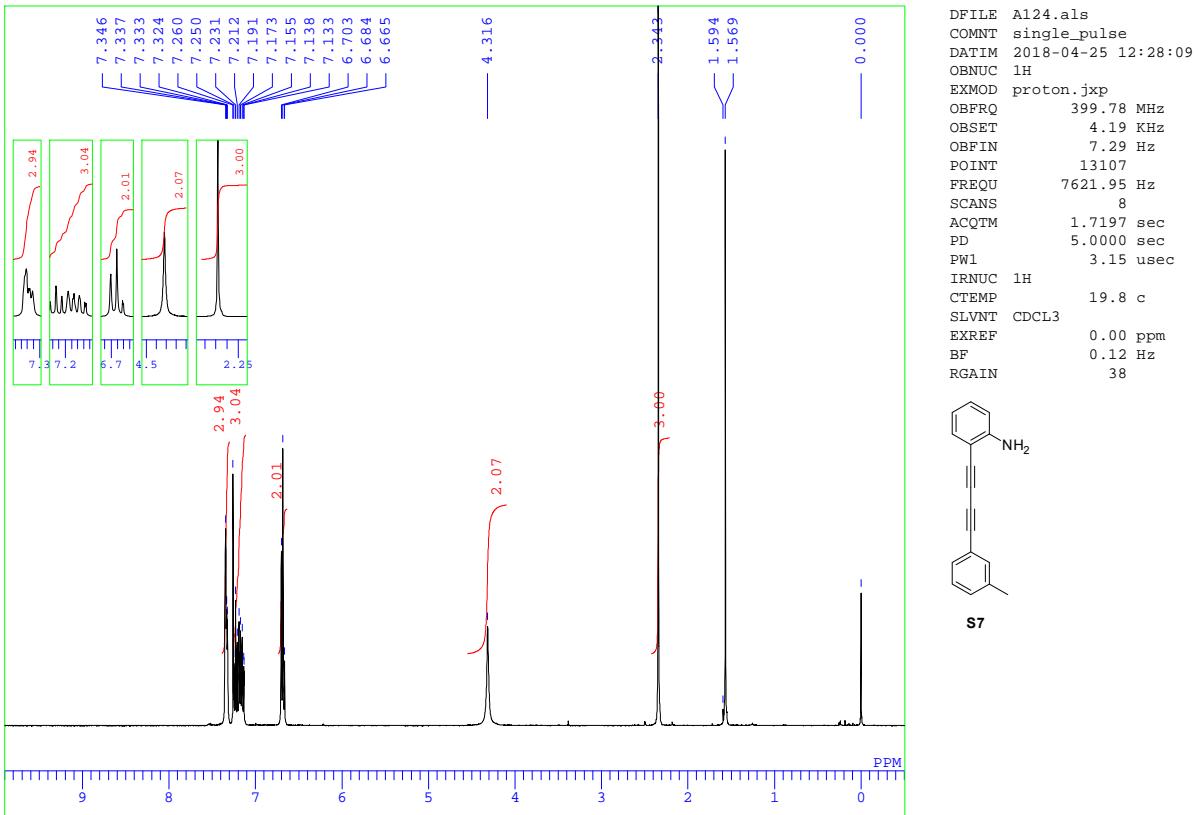
DFILE 10a-C.als
COMNT
DATIM 2017-03-15 12:29:01
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1058
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58

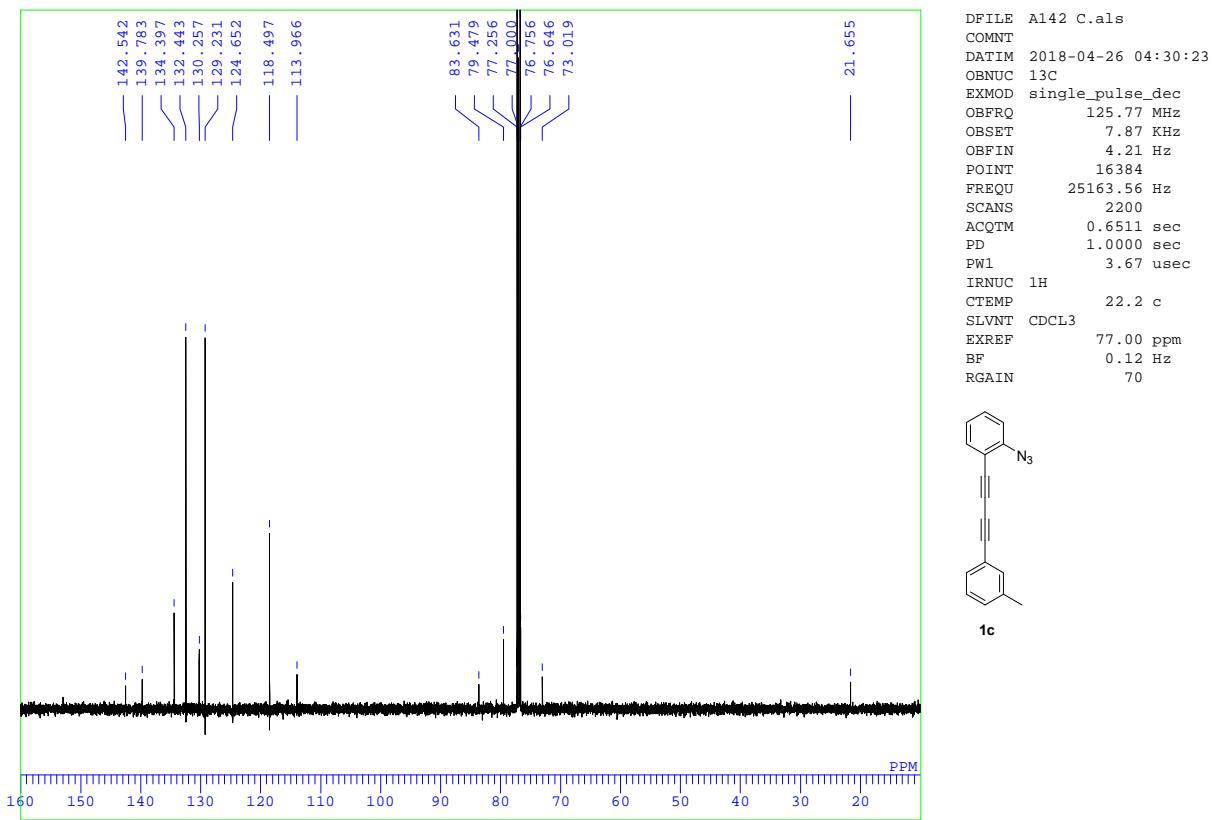
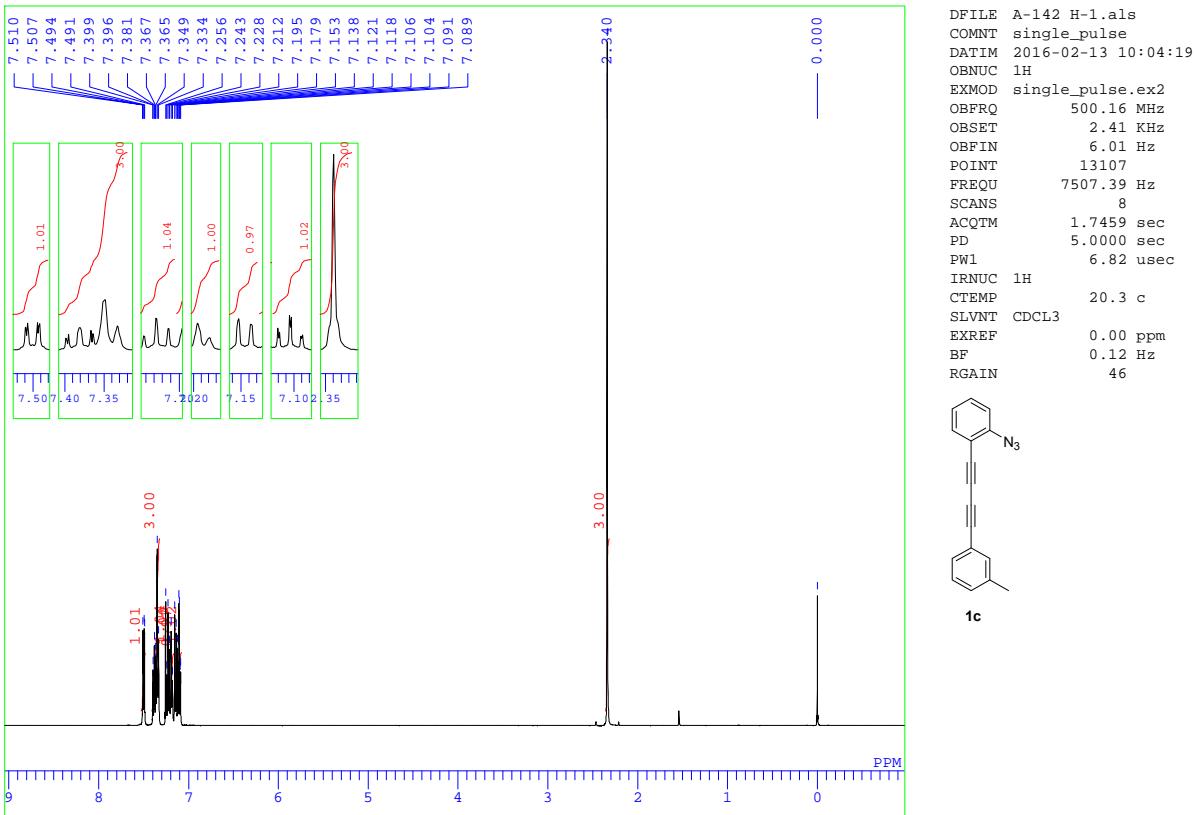
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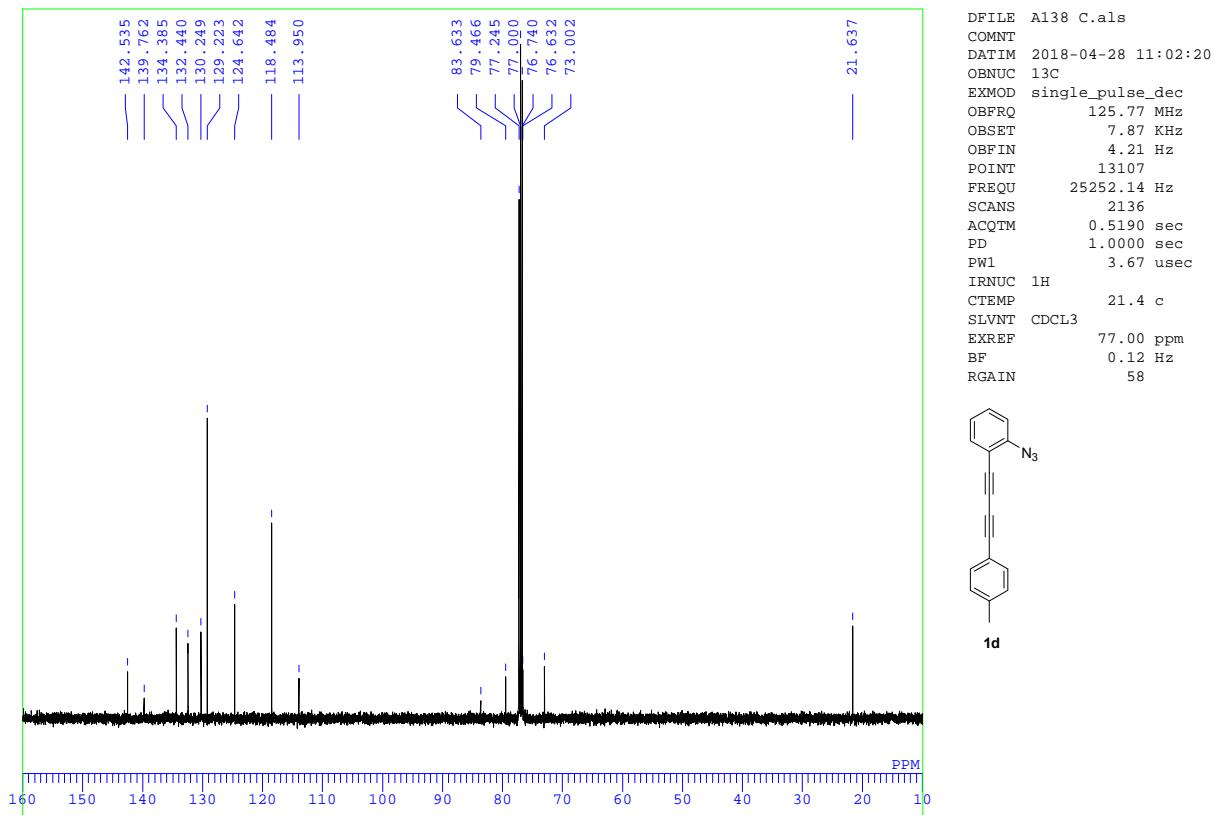
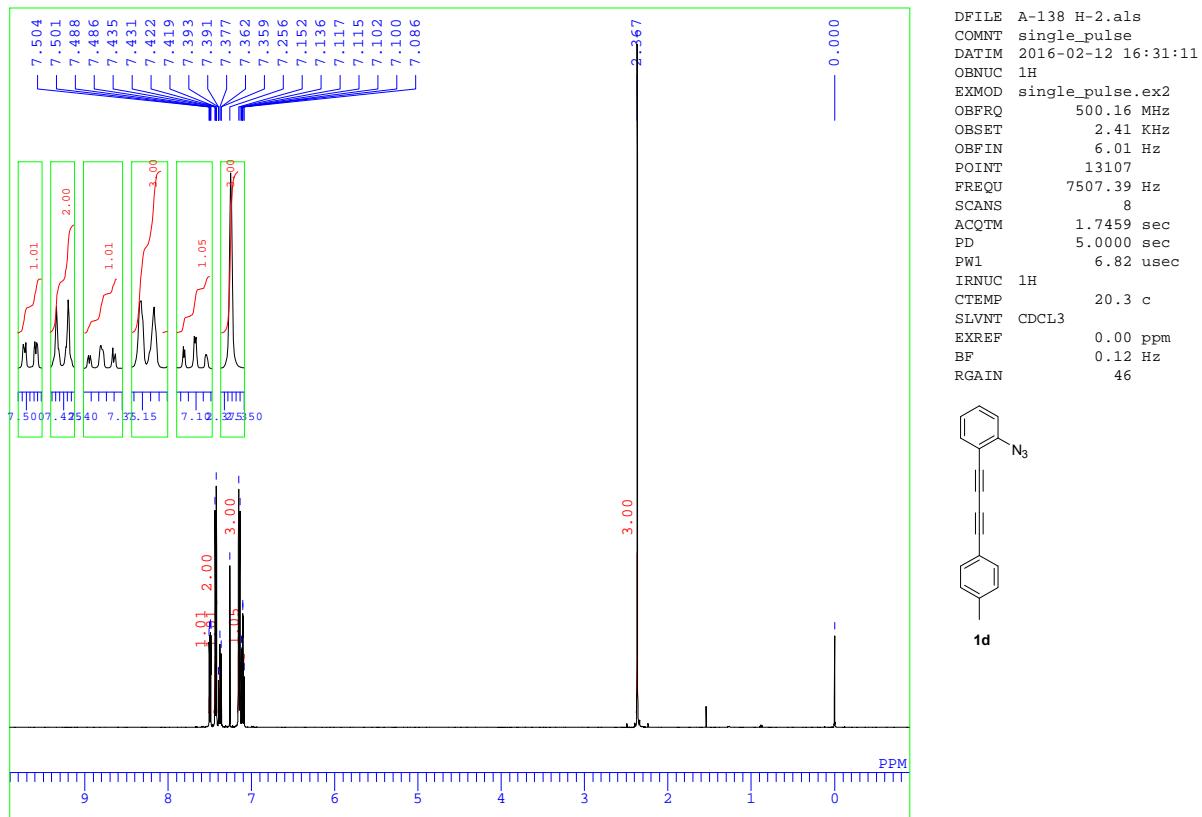


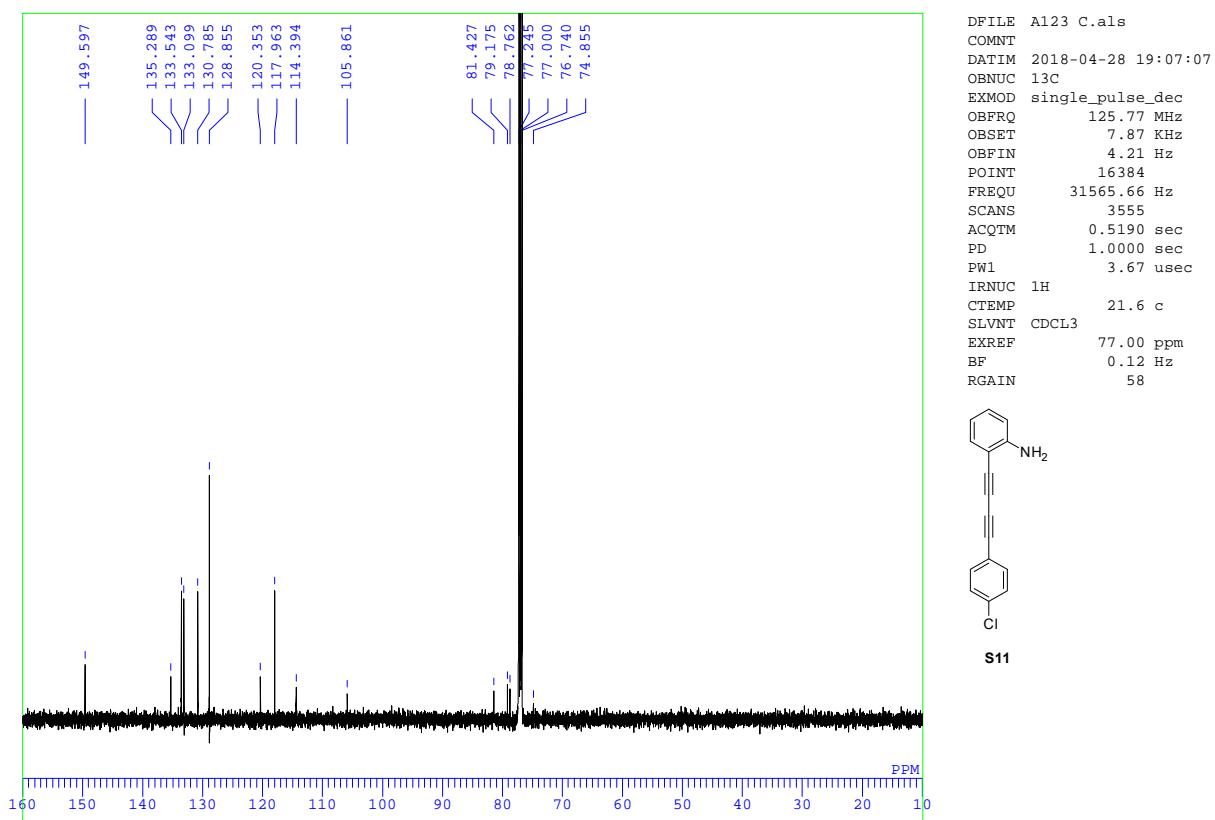
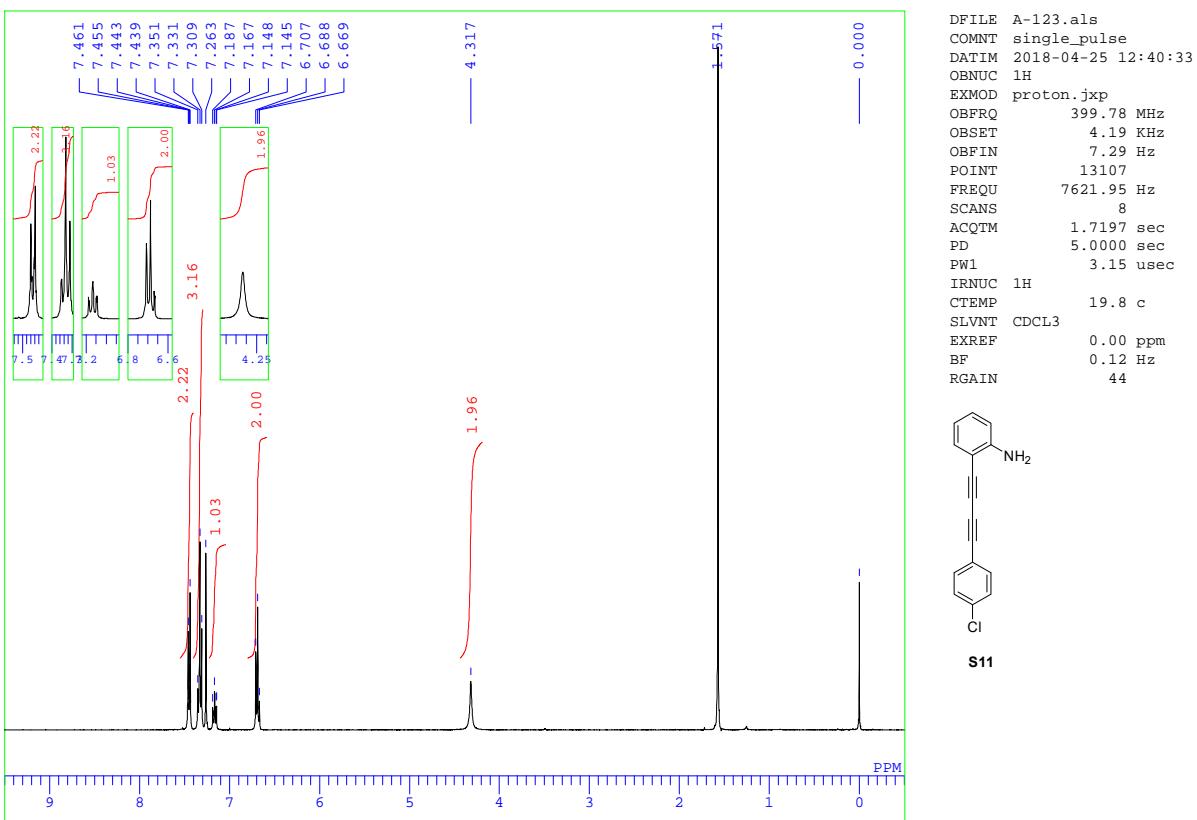


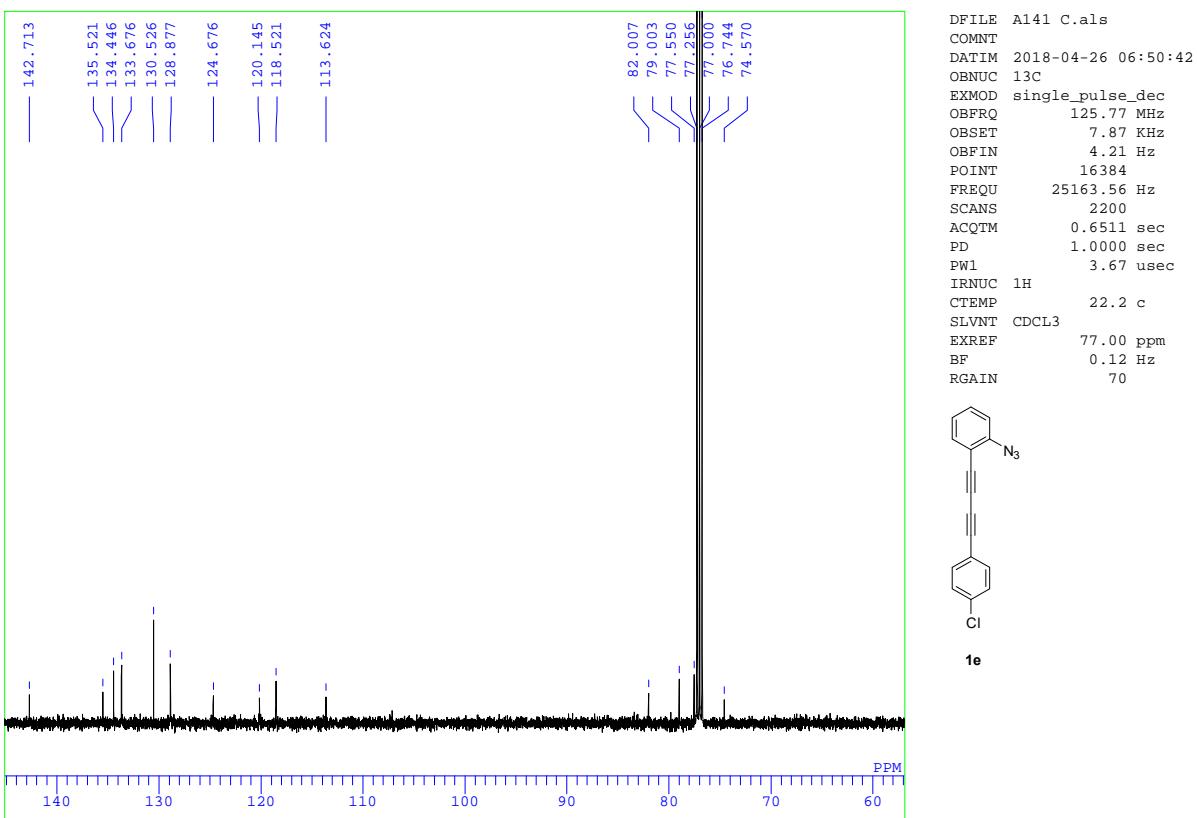
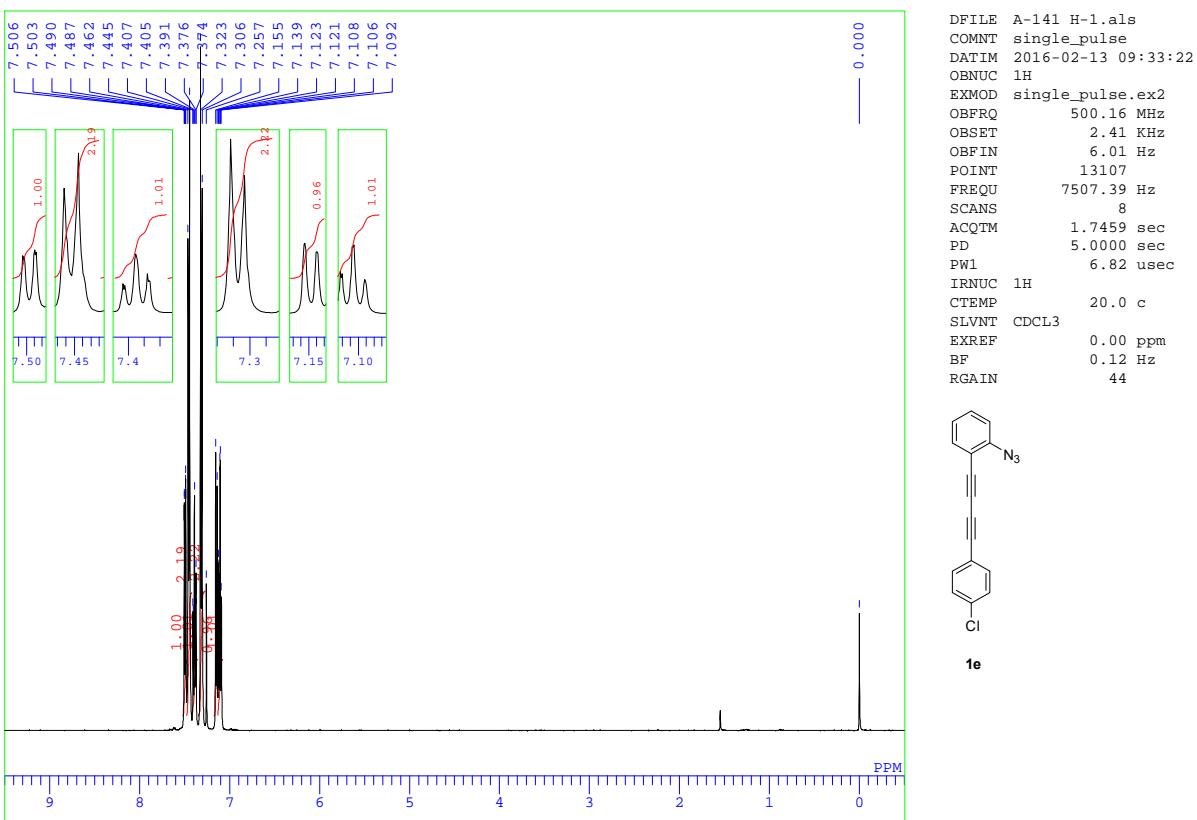


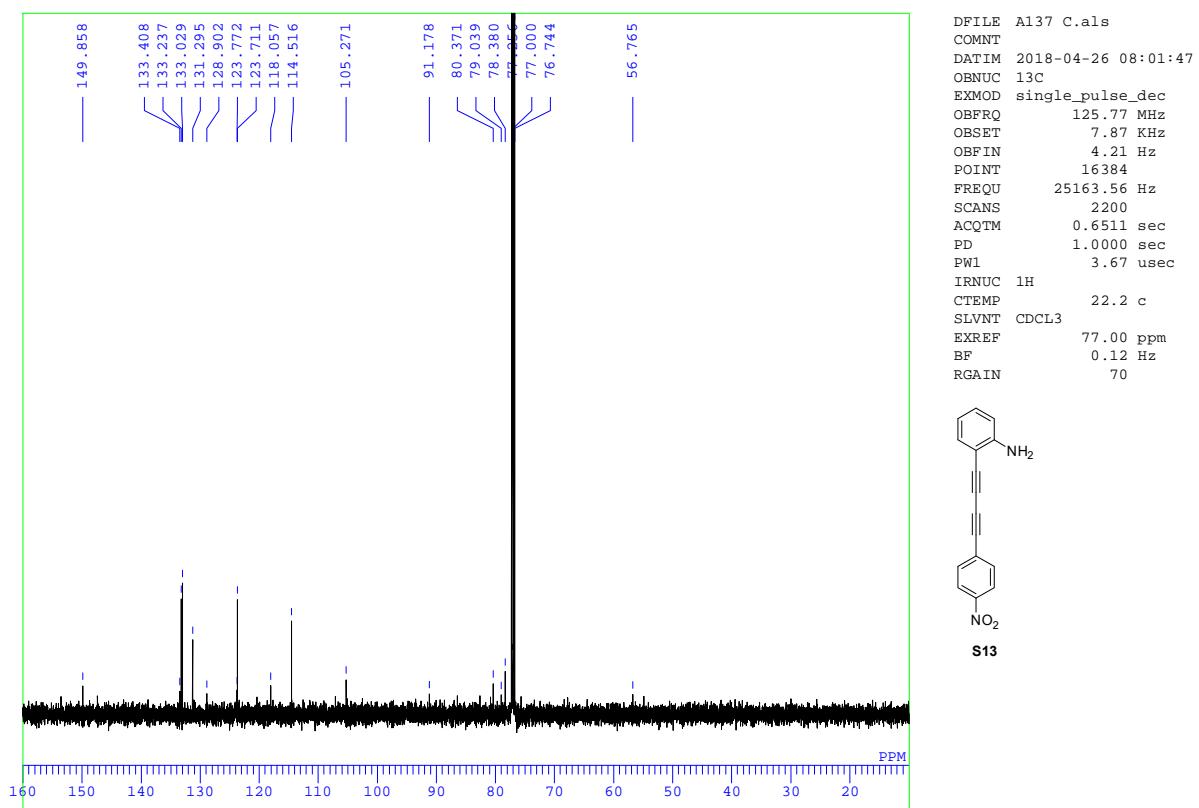
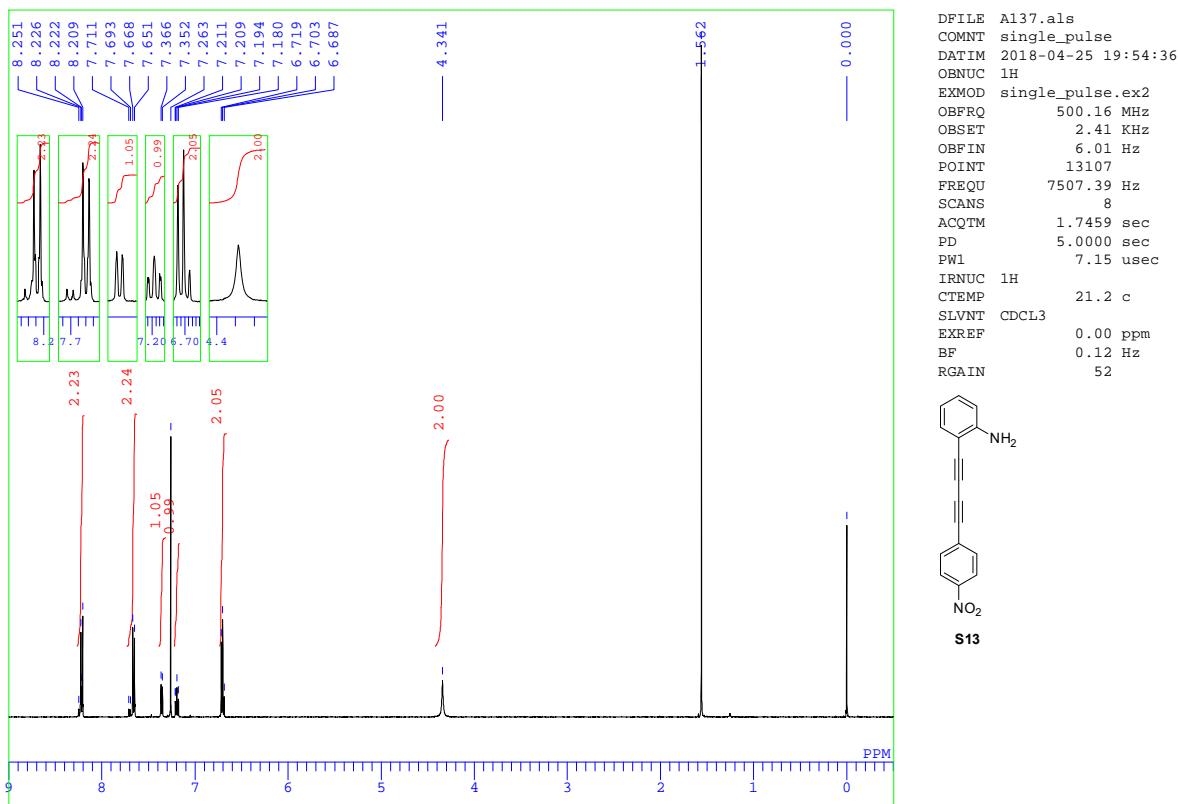


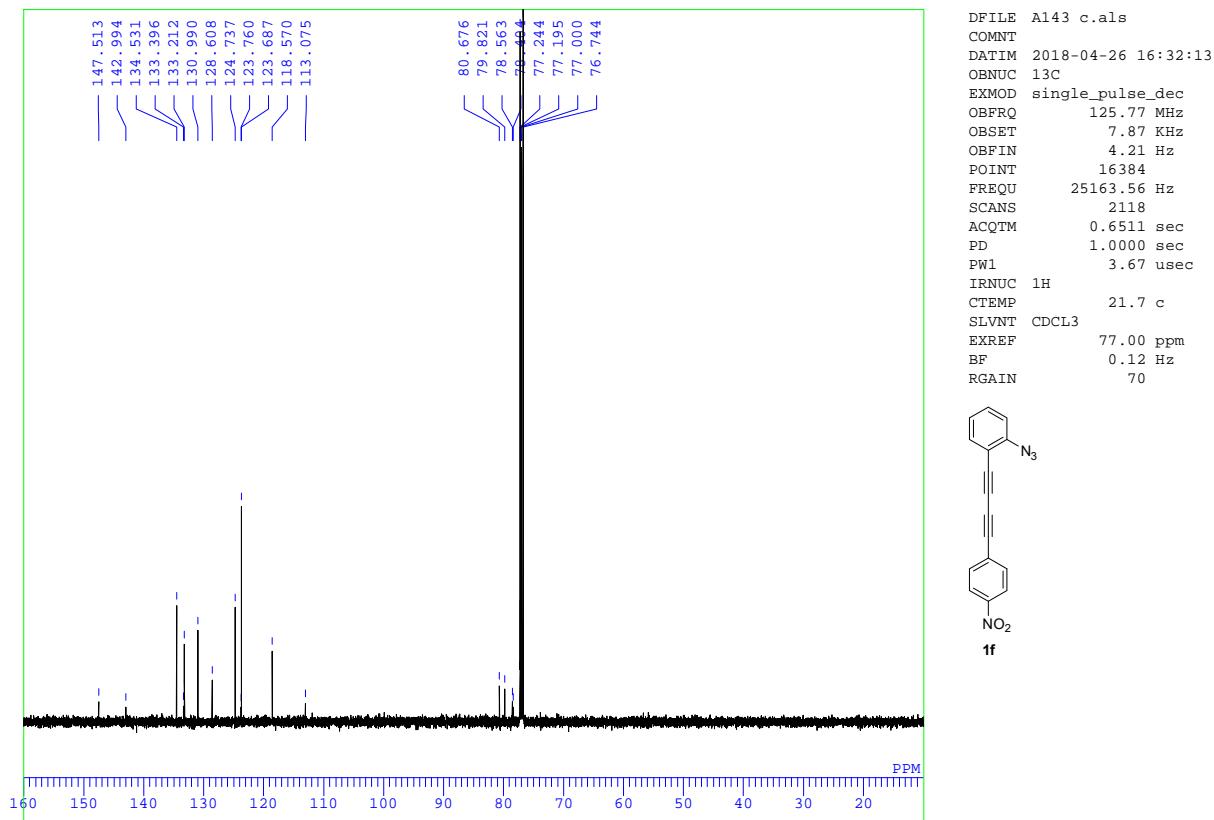
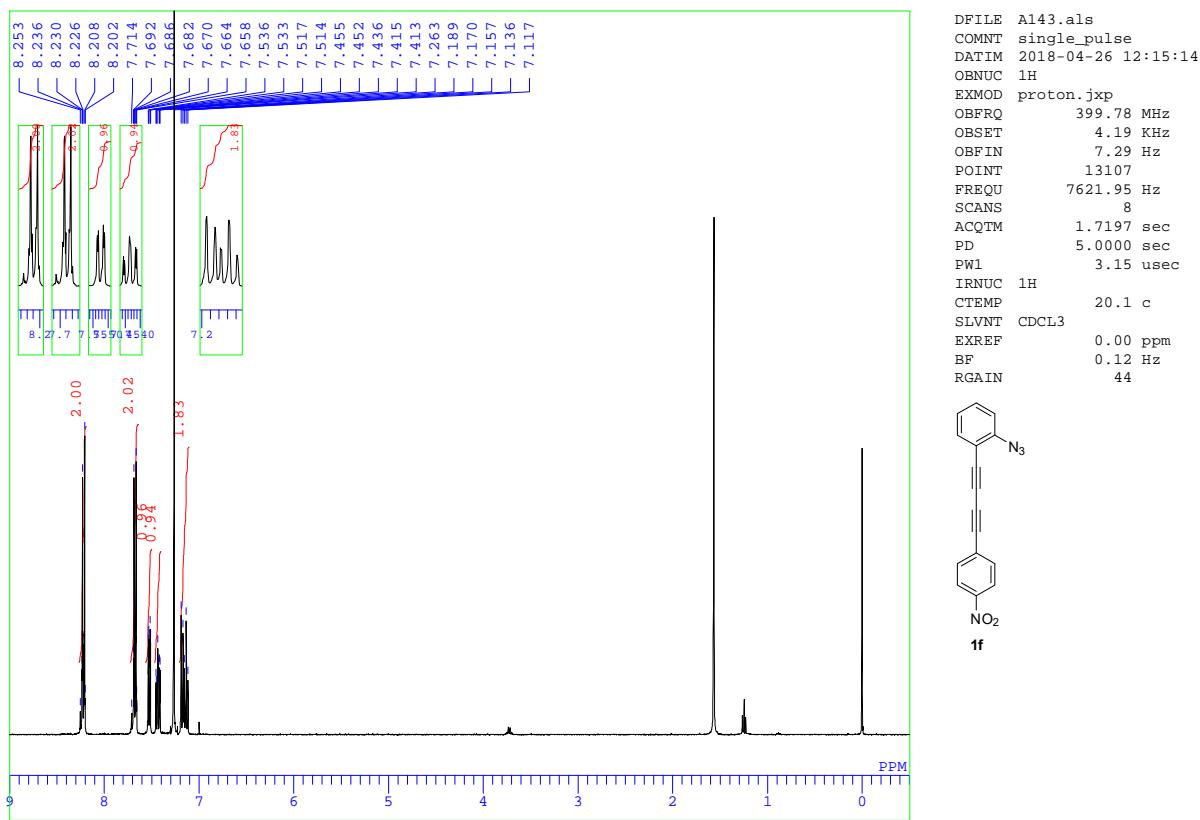


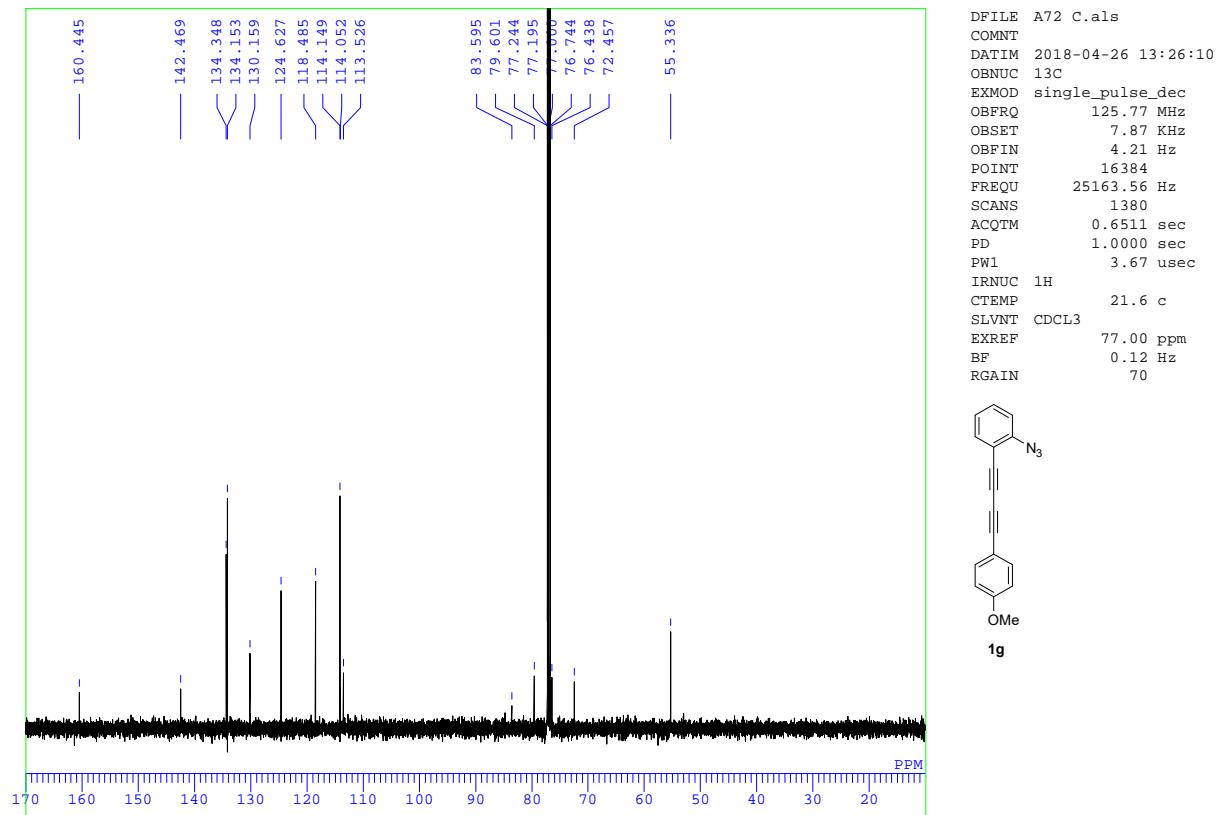
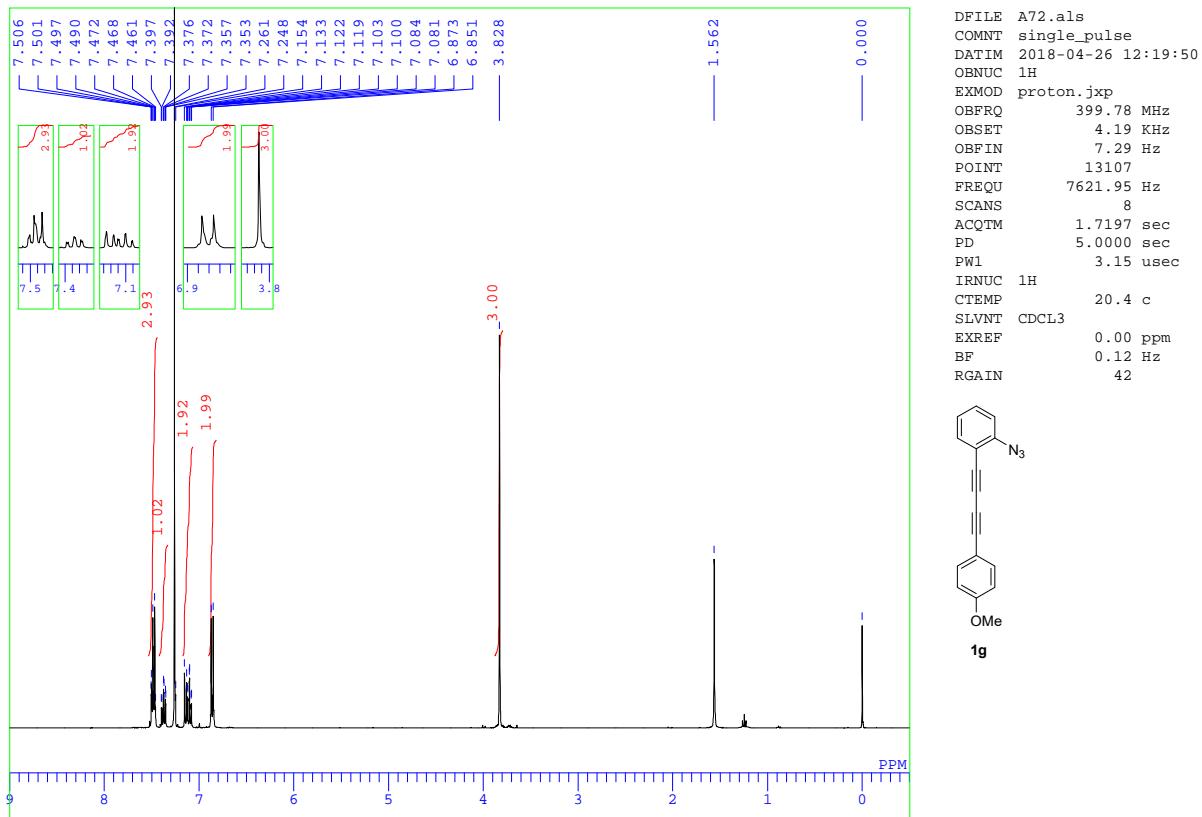


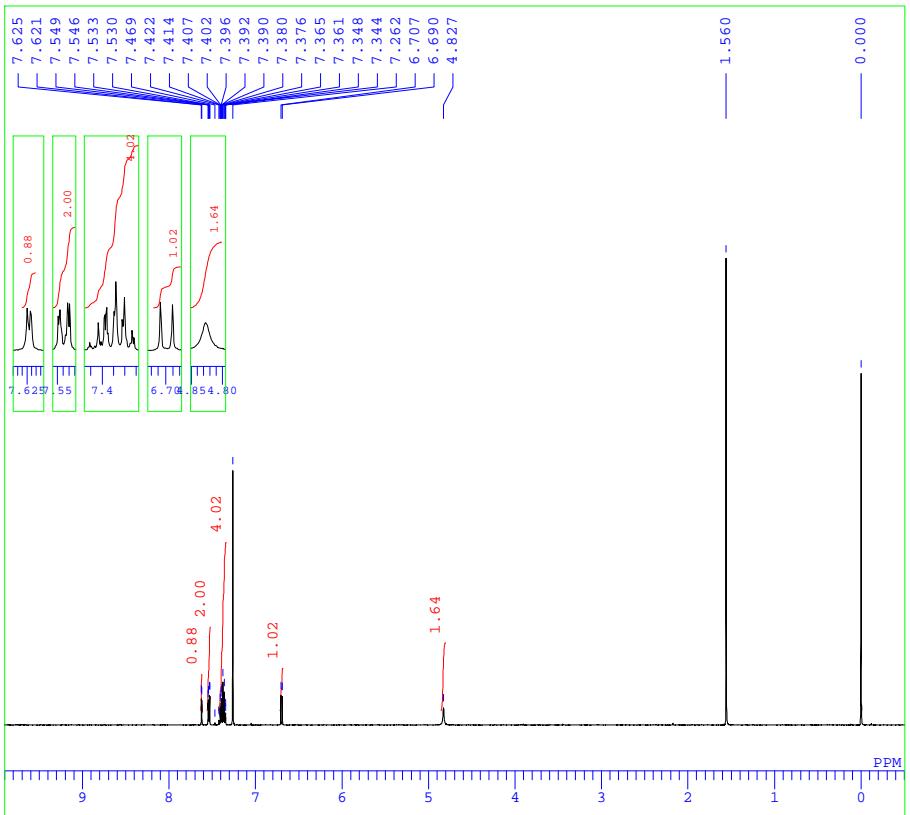








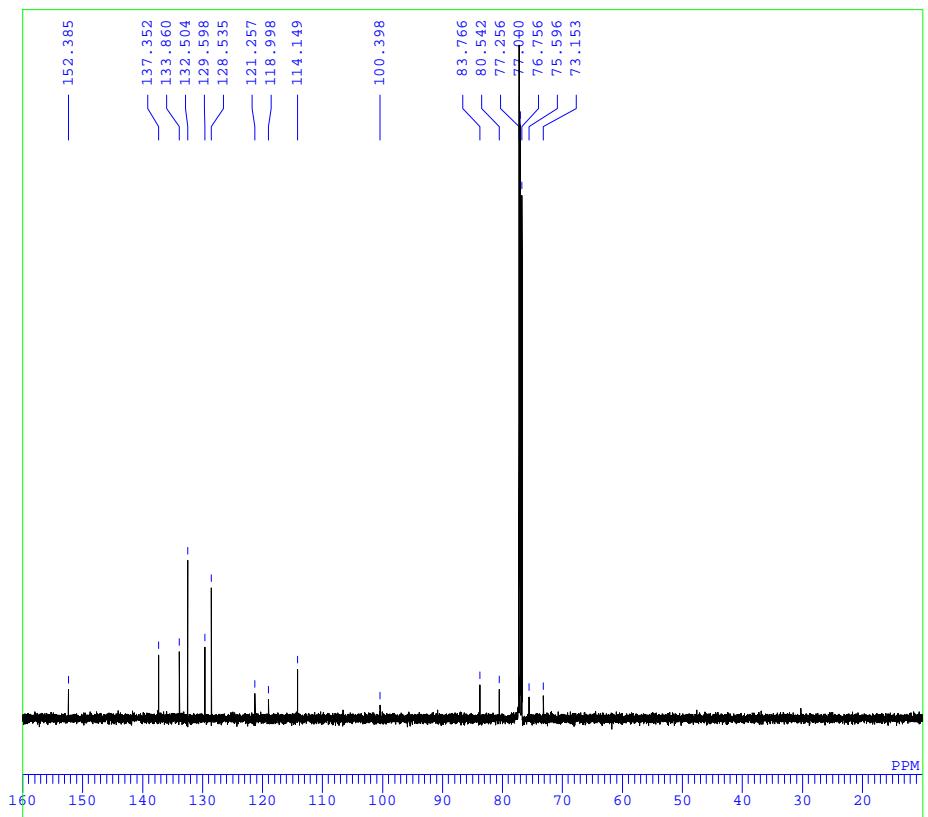
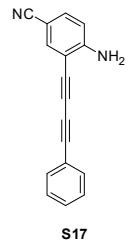




```

DFILE C-39 H-1.als
COMNT single_pulse
DATIM 2017-07-11 11:19:47
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54

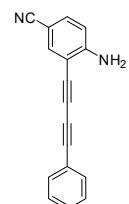
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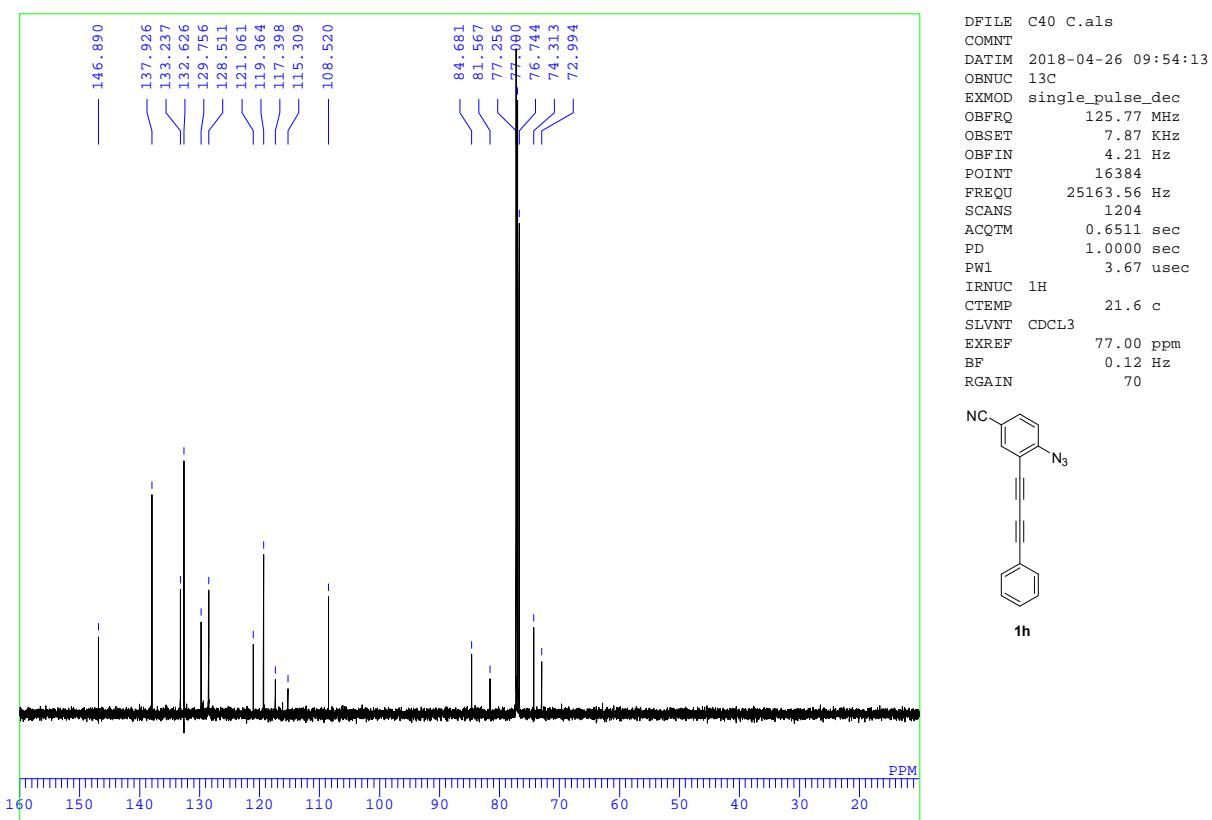
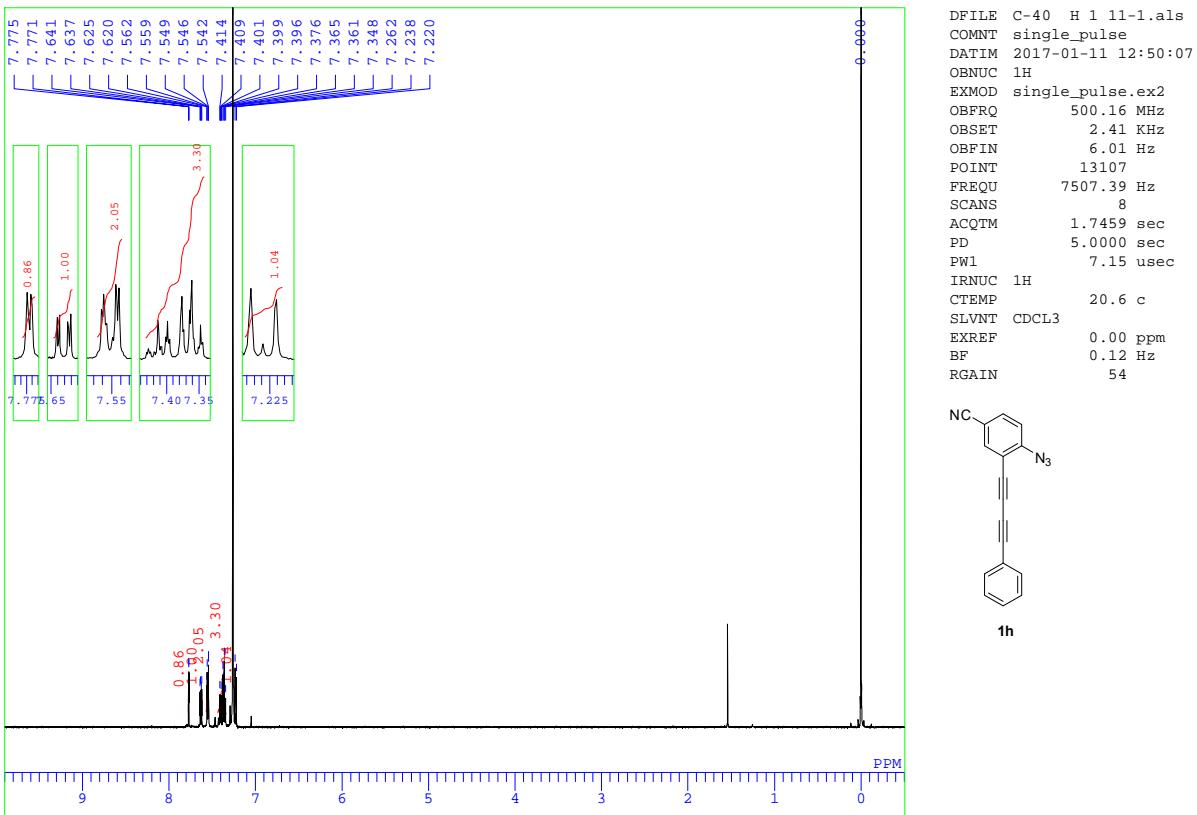


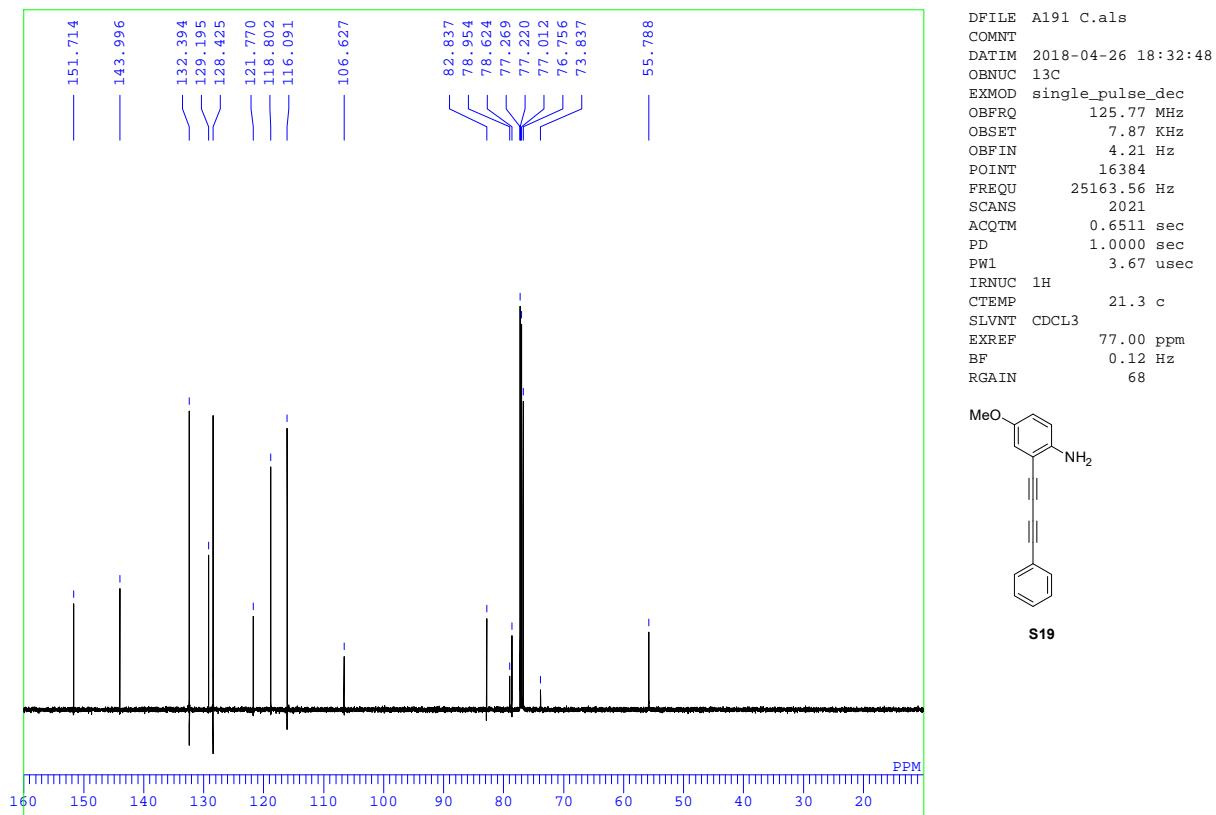
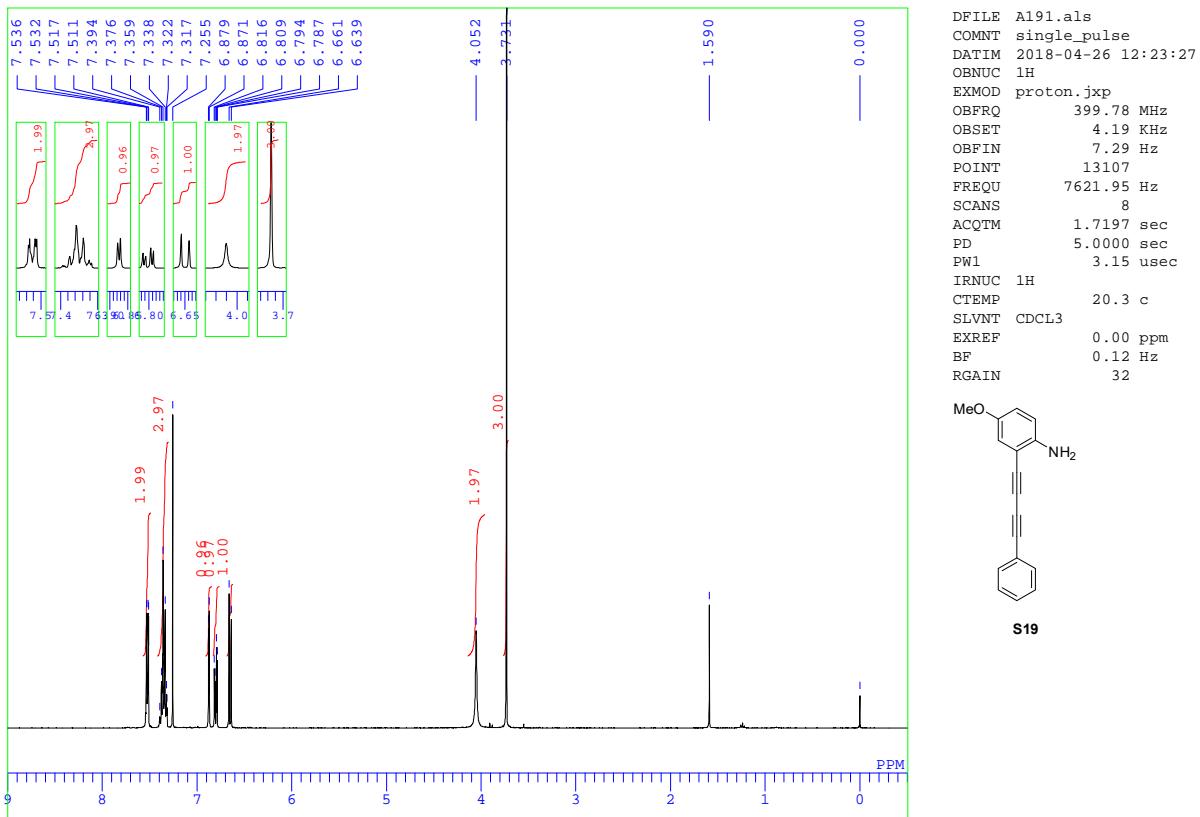
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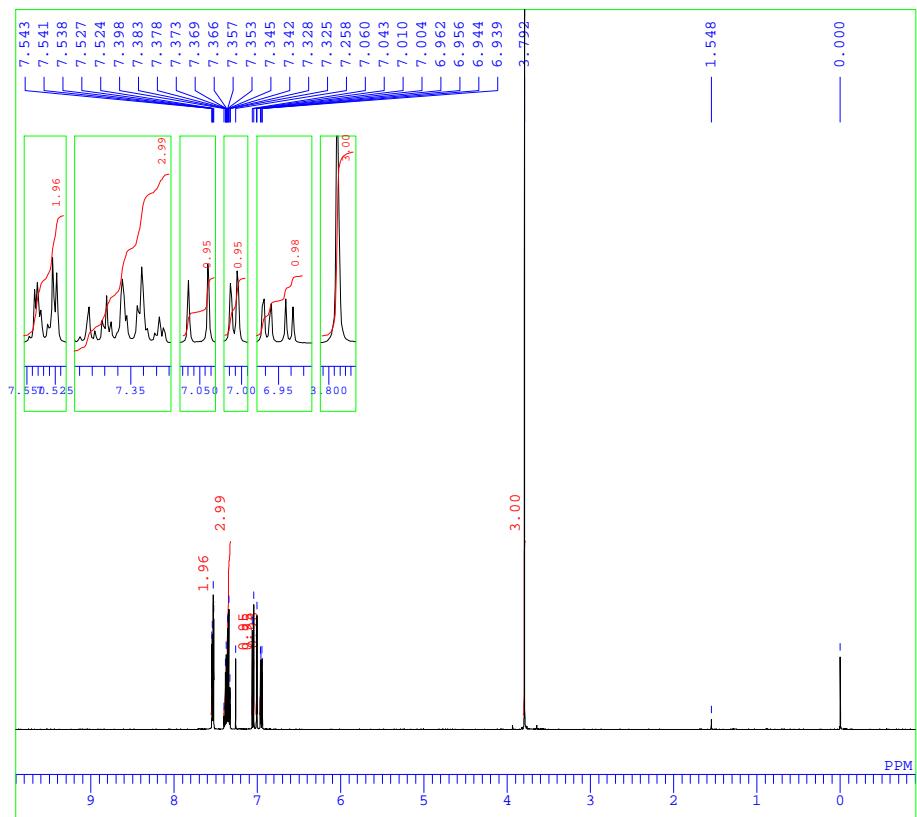
DFILE C39 C.als
COMNT
DATIM 2018-04-26 09:11:22
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 16384
FREQU 25163.56 Hz
SCANS 2200
ACQTM 0.6511 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 70

```





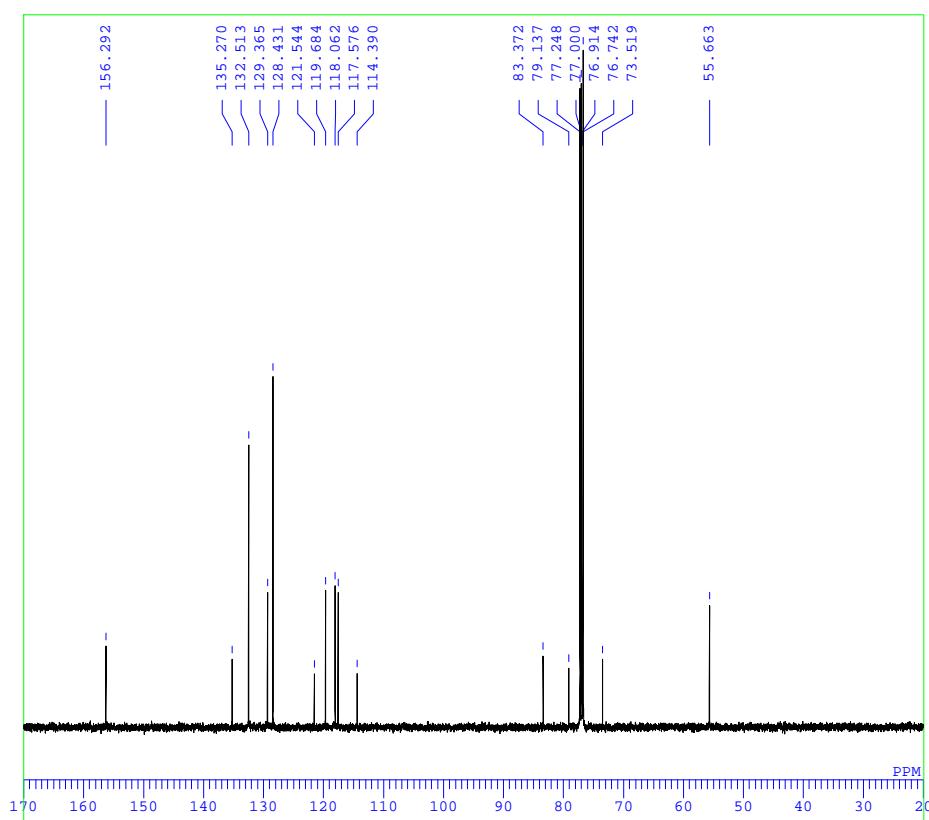
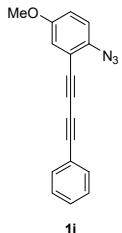




```

DFILE A-192 H.als
COMNT single_pulse
DATIM 2016-02-12 15:55:24
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46

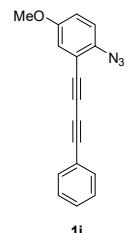
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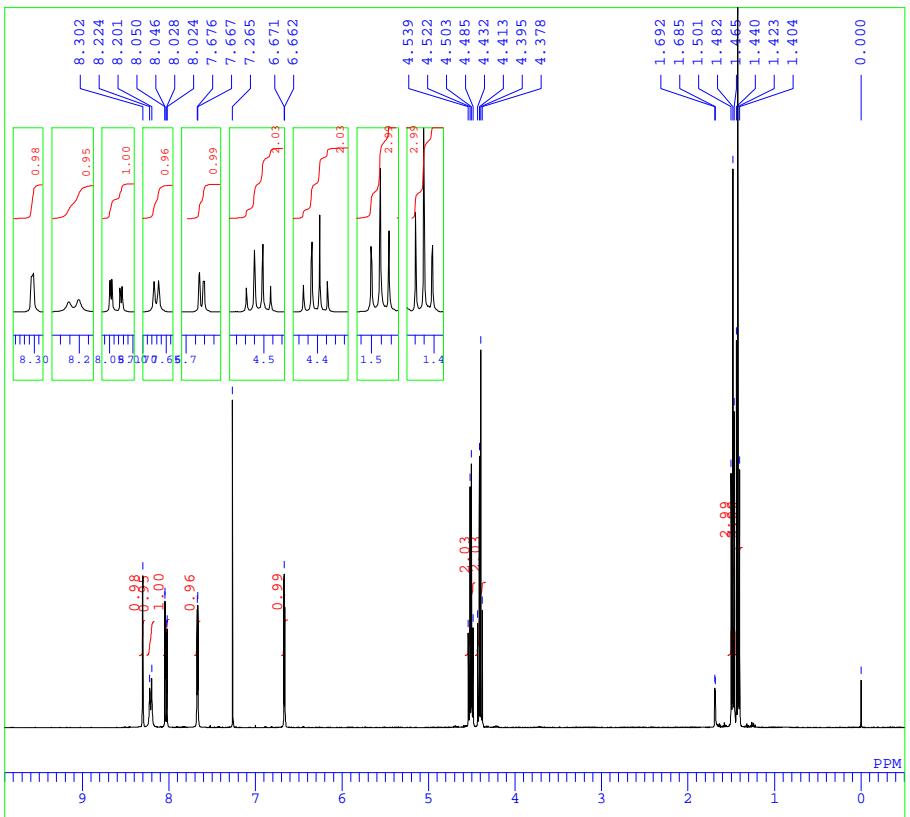


```

DFILE A-192 C-1.als
COMNT single pulse decoupled gat
DATIM 2016-02-12 16:25:56
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 632
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54

```

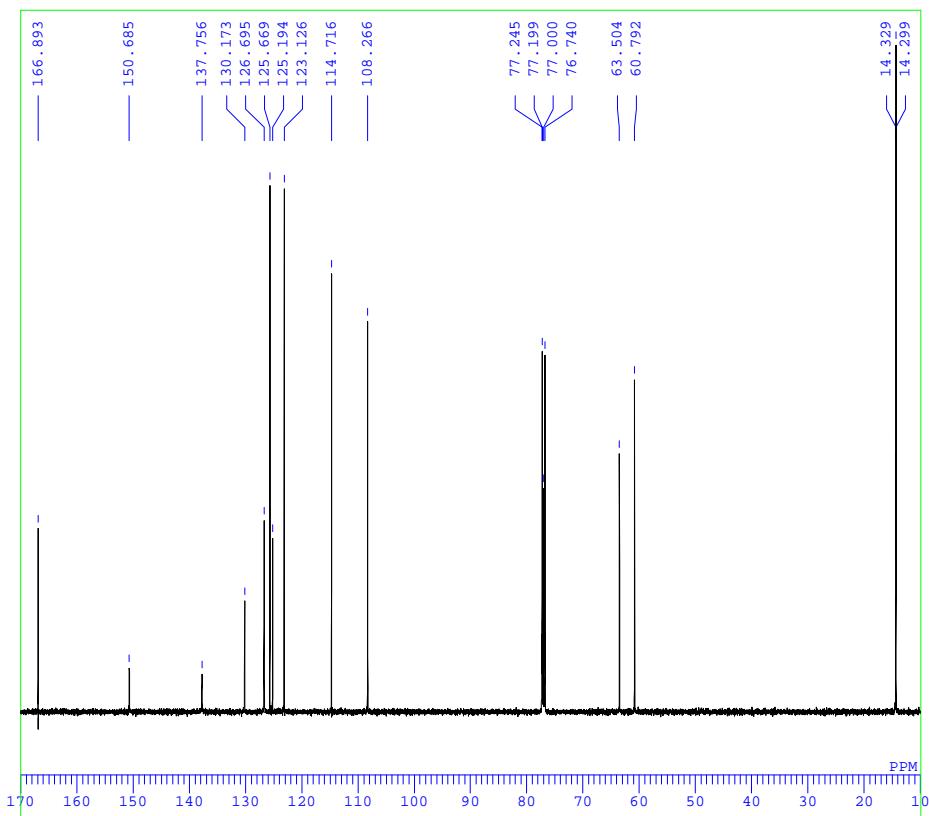
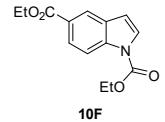




```

DFILE B176.als
COMNT single_pulse
DATIM 2018-04-28 11:36:26
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 kHz
OBFIN 7.29 Hz
POINT 13107
FREQU 7598.78 Hz
SCANS 8
ACQTM 1.7249 sec
PD 5.0000 sec
PW1 3.15 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34

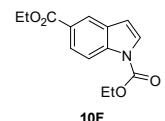
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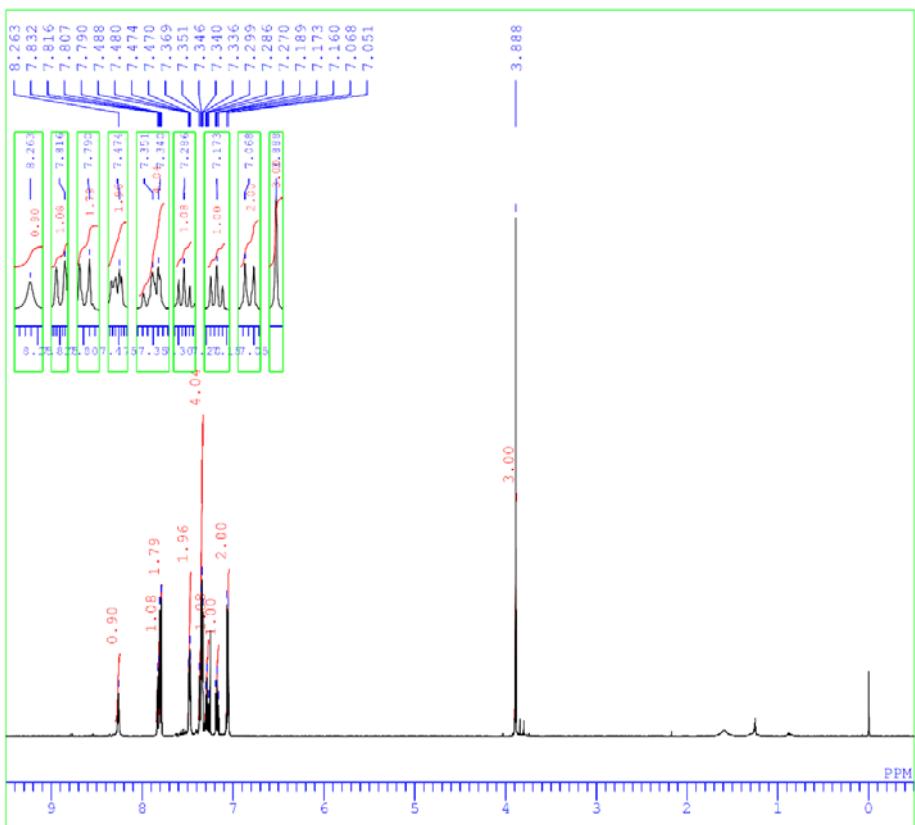


```

DFILE B176 C.als
COMNT
DATIM 2018-04-28 06:11:27
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 13107
FREQU 25252.14 Hz
SCANS 2500
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

```

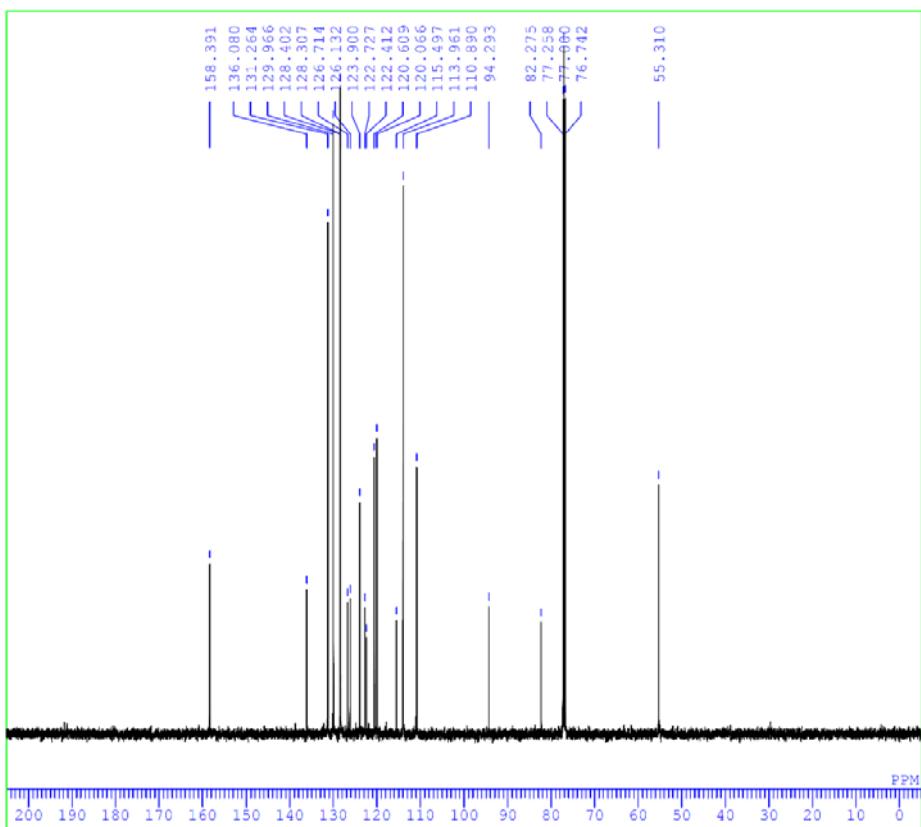
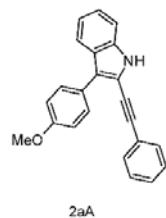




```

DFILE anisole intermediate h-1.als
COMNT single_pulse
DATIM 2018-01-22 19:27:10
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46

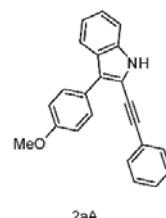
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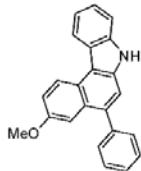
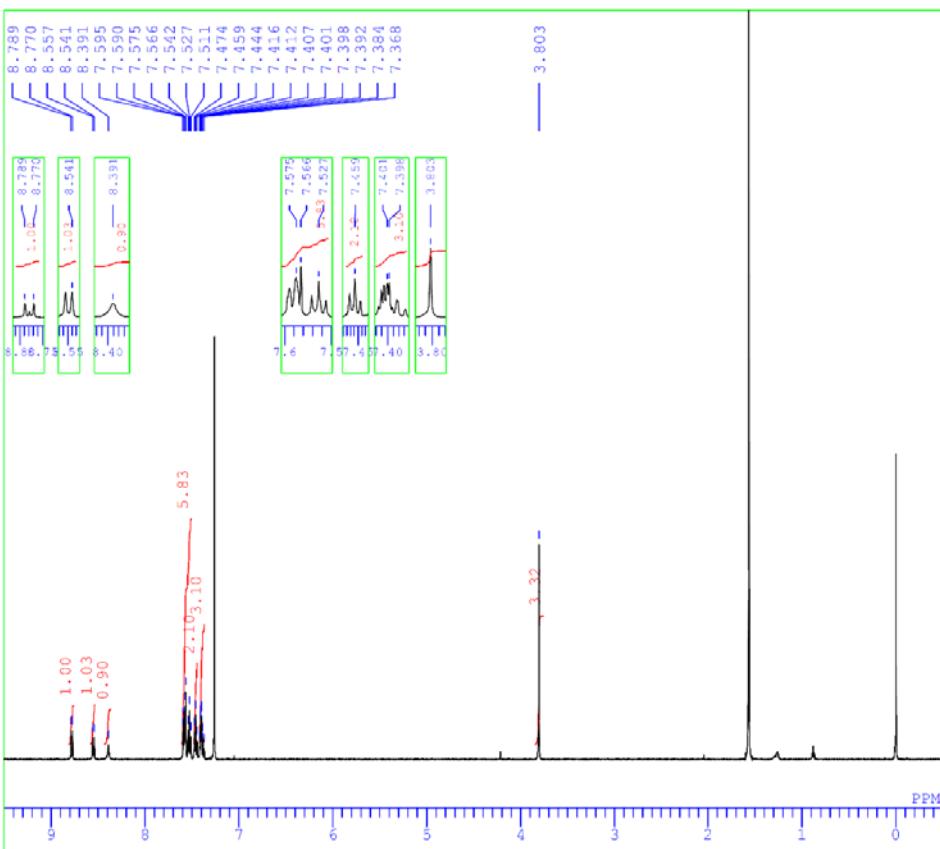


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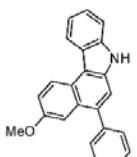
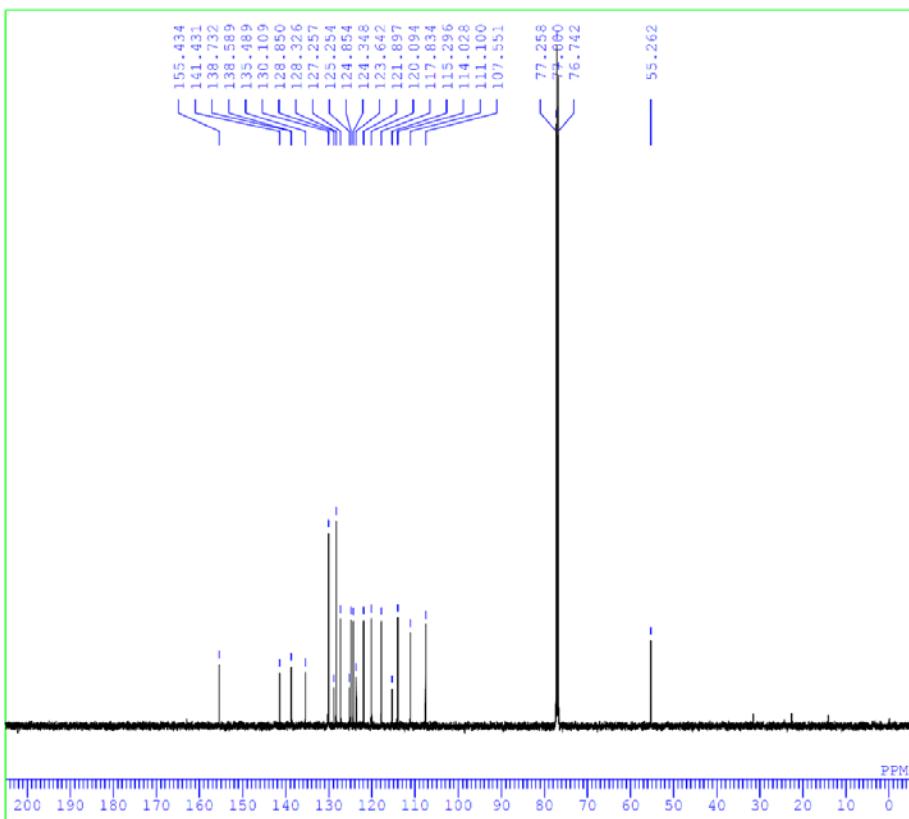
DFILE anisole intermediate c-1.als
COMNT
DATIM 2016-11-04 17:45:14
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 464
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

```





3aA

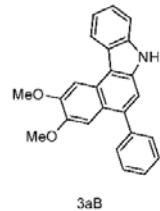
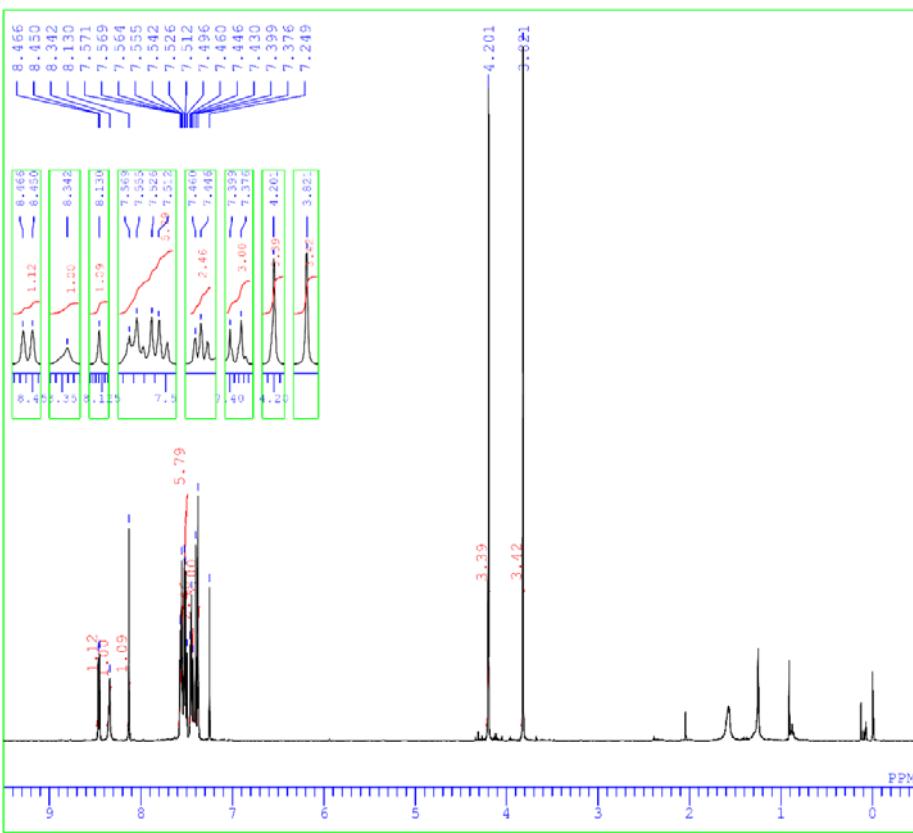


32A

```

DFILE anisole product h-1.als
COMNT single_pulse
DATIM 2016-05-23 15:36:49
QBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.50 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 52

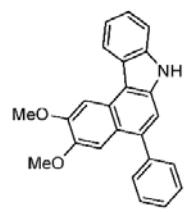
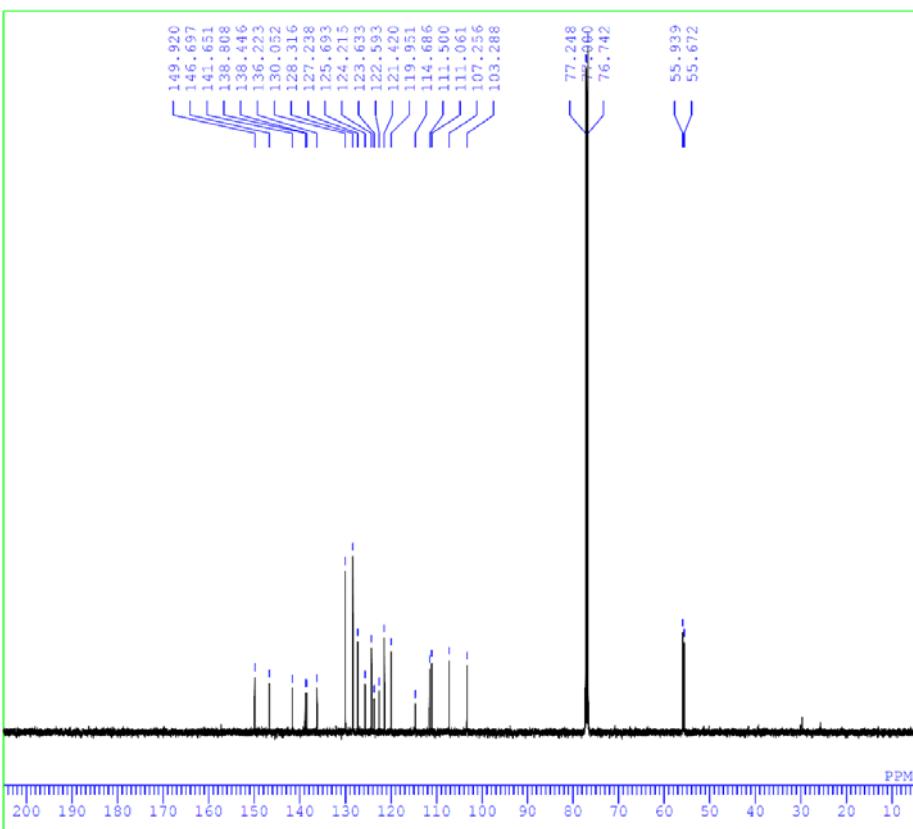
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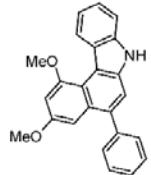
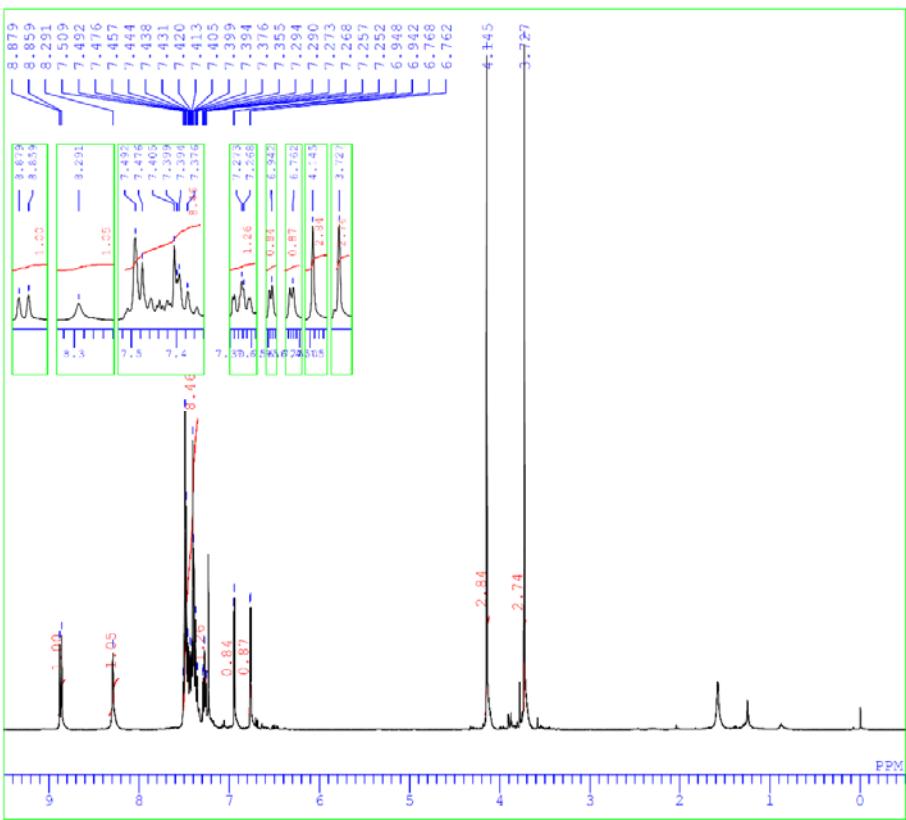


```

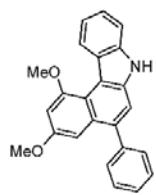
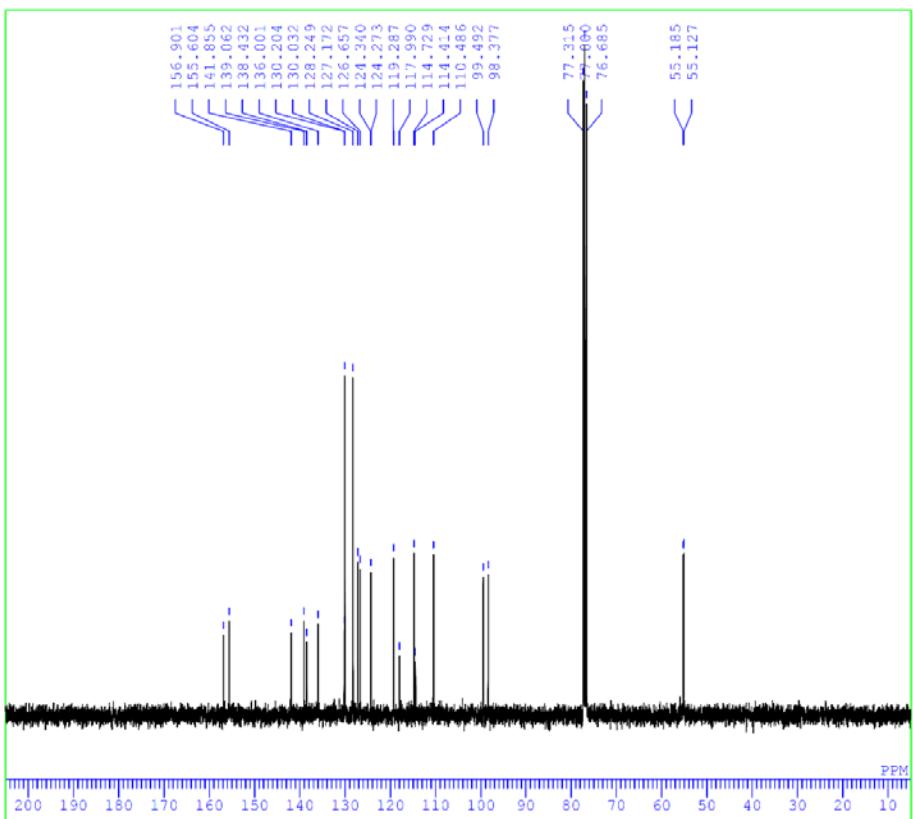
DFILE 1,2-dimethoxybenzene c-1.als
COMNT
DATIM 2017-09-15 11:41:46
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
OBNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

```





3aC

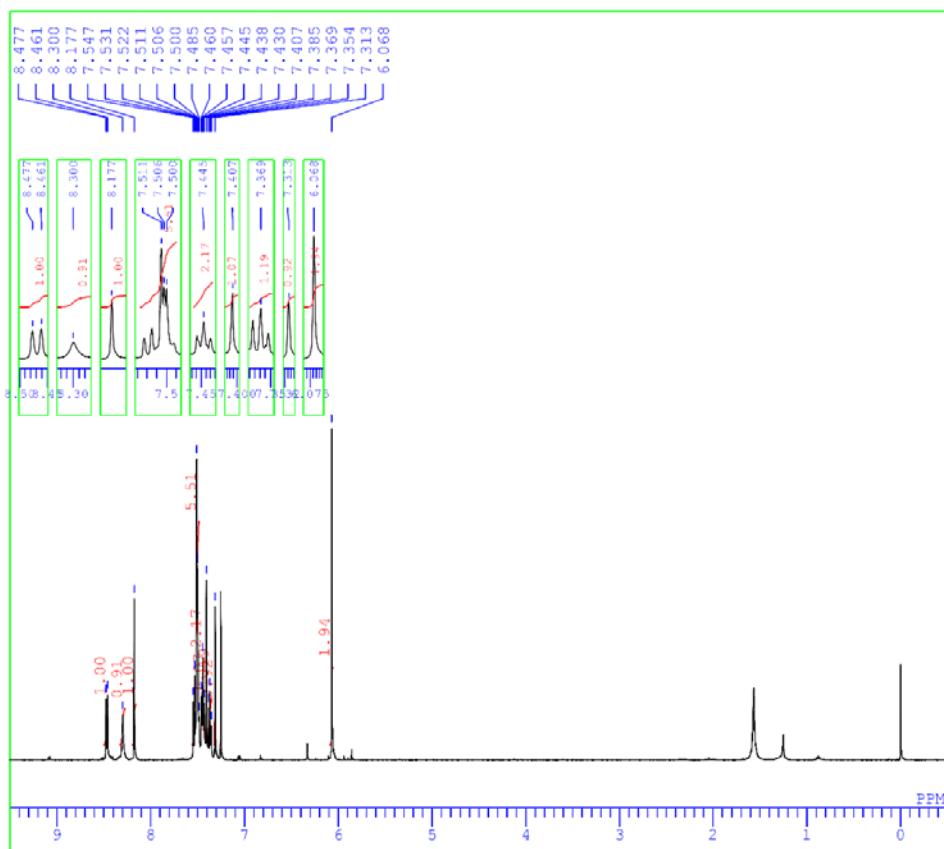


3aC

```

DFILE 1,3-dimethoxybenzene_Proton-1.
single_pulse
DATIM 2016-07-16 11:23:37
OBNUC 1H
EXMOD proton.jxp
OBFRQ      399.78 MHz
QBSET       4.19 kHz
OBFIN      7.29 Hz
POINT      13107
FREQU      7598.78 Hz
SCANS        8
ACQTM       1.7249 sec
PD          5.0000 sec
PW1         3.06 usec
IRNUC 1H
CTEMP      19.7 c
SLVNT CDCL3
EXREF      0.00 ppm
BF          0.12 Hz
RGAIN      36

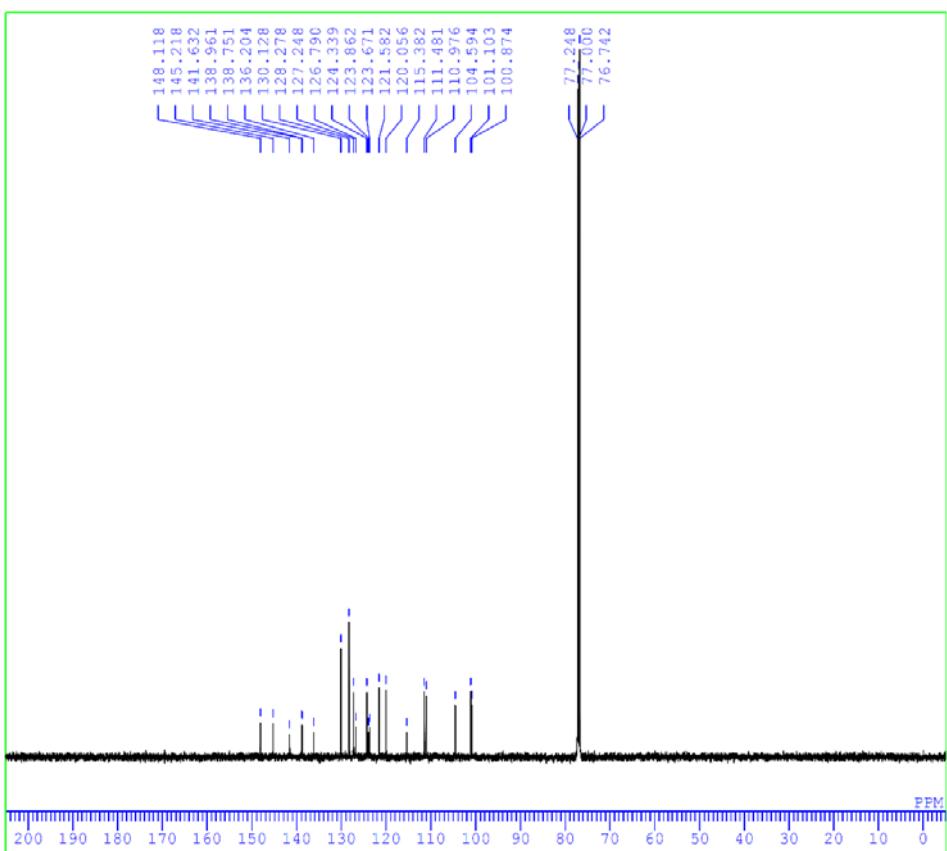
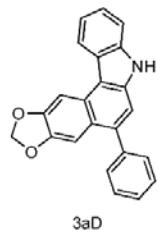
```



```

DFILE 1,3-benzodioxole h-1.als
COMNT single_pulse
DATIM 2016-11-02 17:10:21
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 48

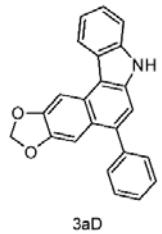
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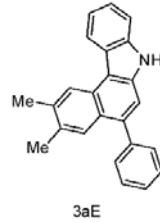
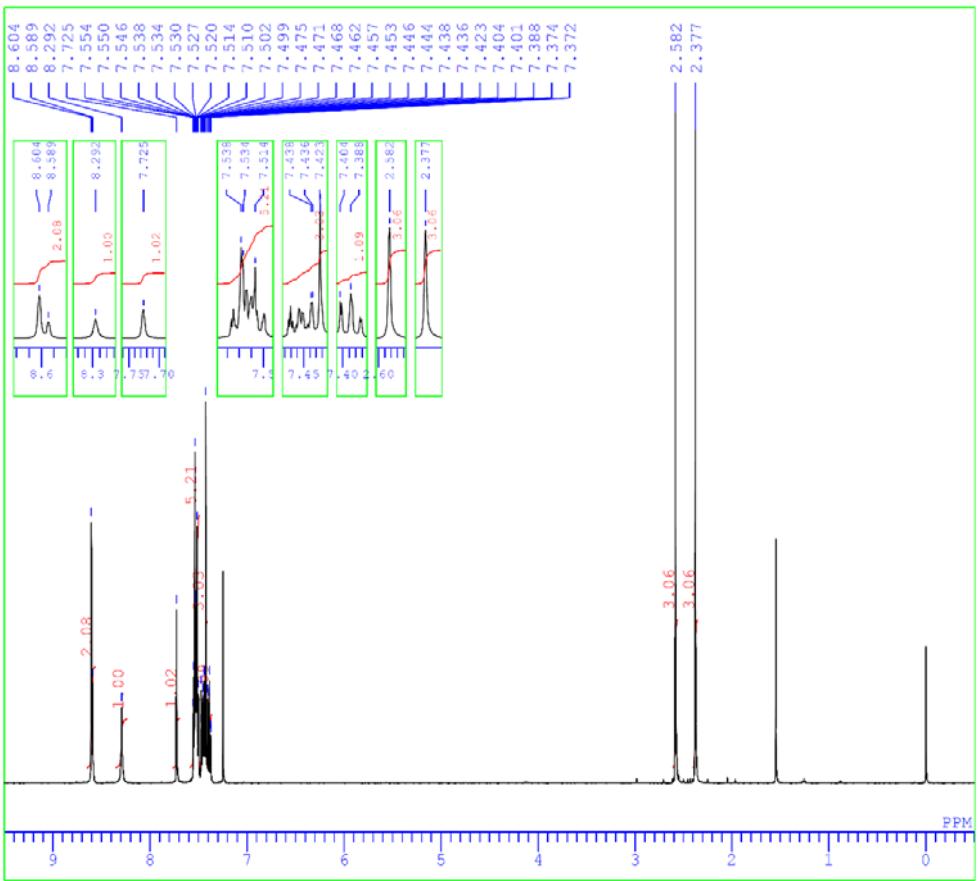


```

DFILE 1,3-benzodioxole c-1.als
COMNT single_pulse_dec
DATIM 2016-11-02 18:01:44
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58

```

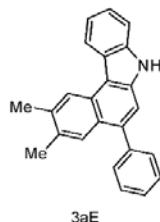
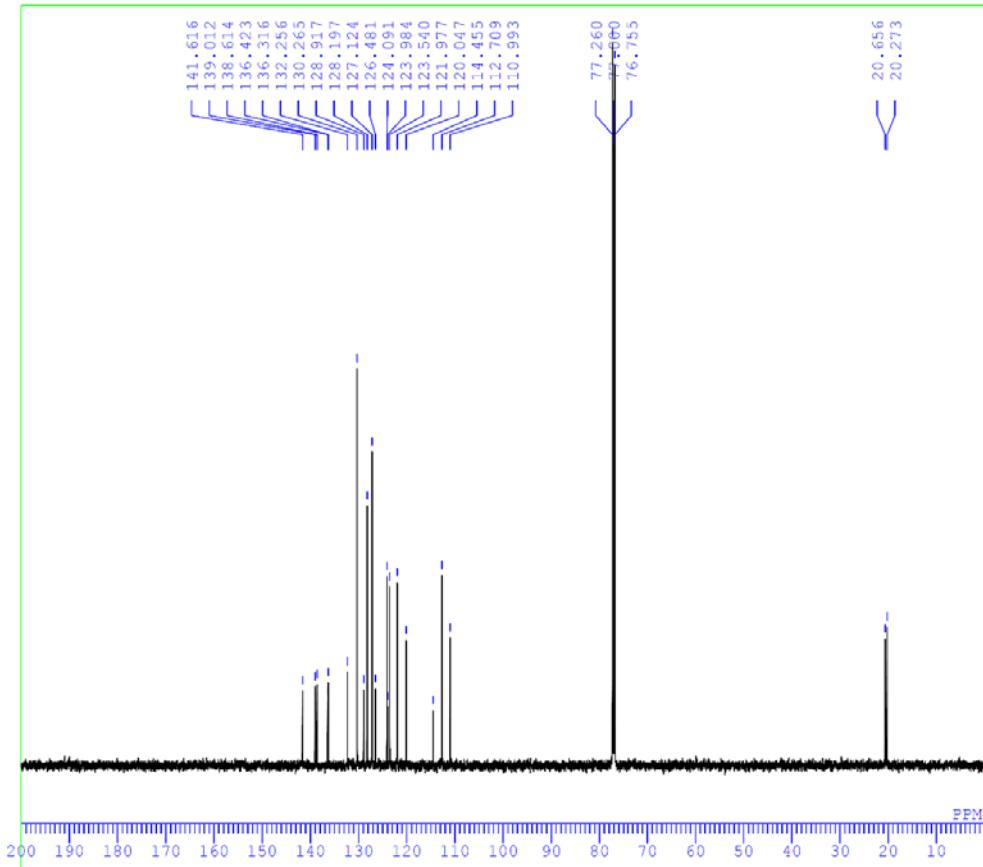


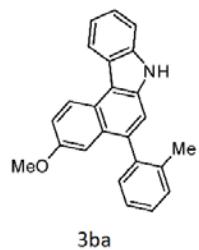
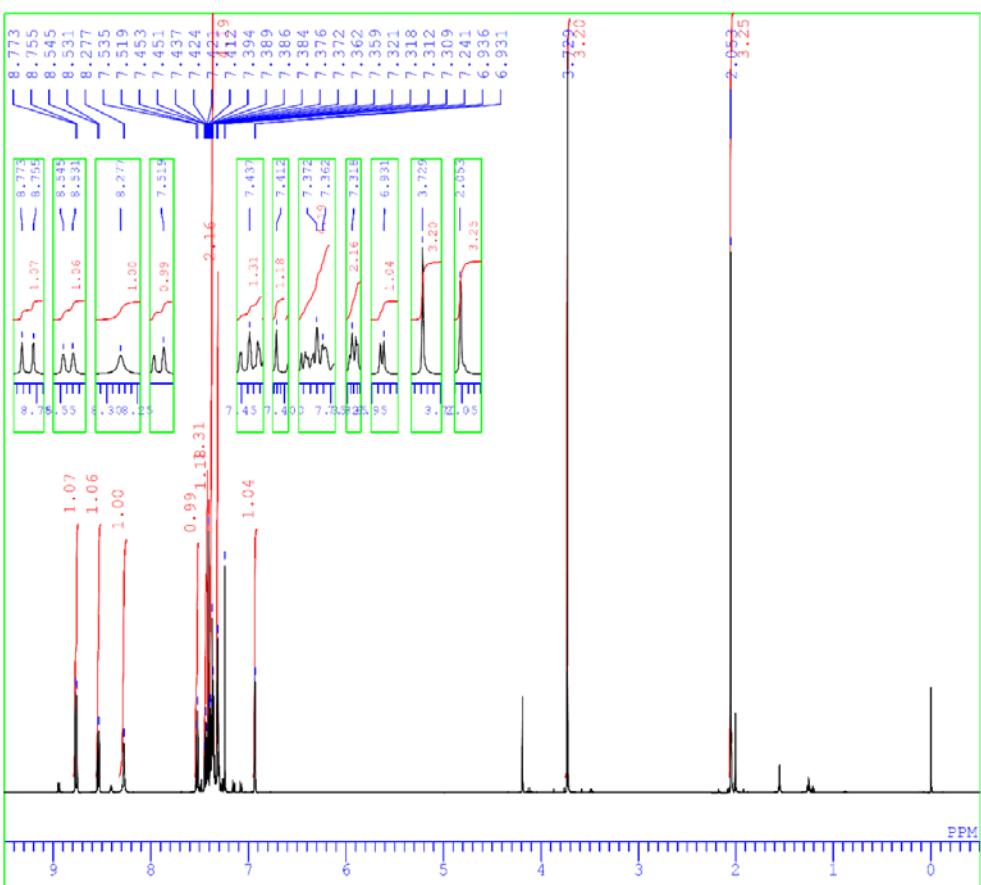


```

DFILE o-xylene c-1.als
COMNT
DATIM 2018-03-08 17:05:18
OBNUC 13C
EXMOD single_pulse_dec
OBFREQ 125.77 MHz
OBSET 7.87 kHz
OBSFIN 4.21 Hz
PCINT 13107
FREQU 25252.14 Hz
SCANS 2077
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54

```

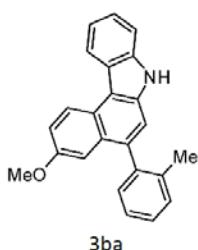
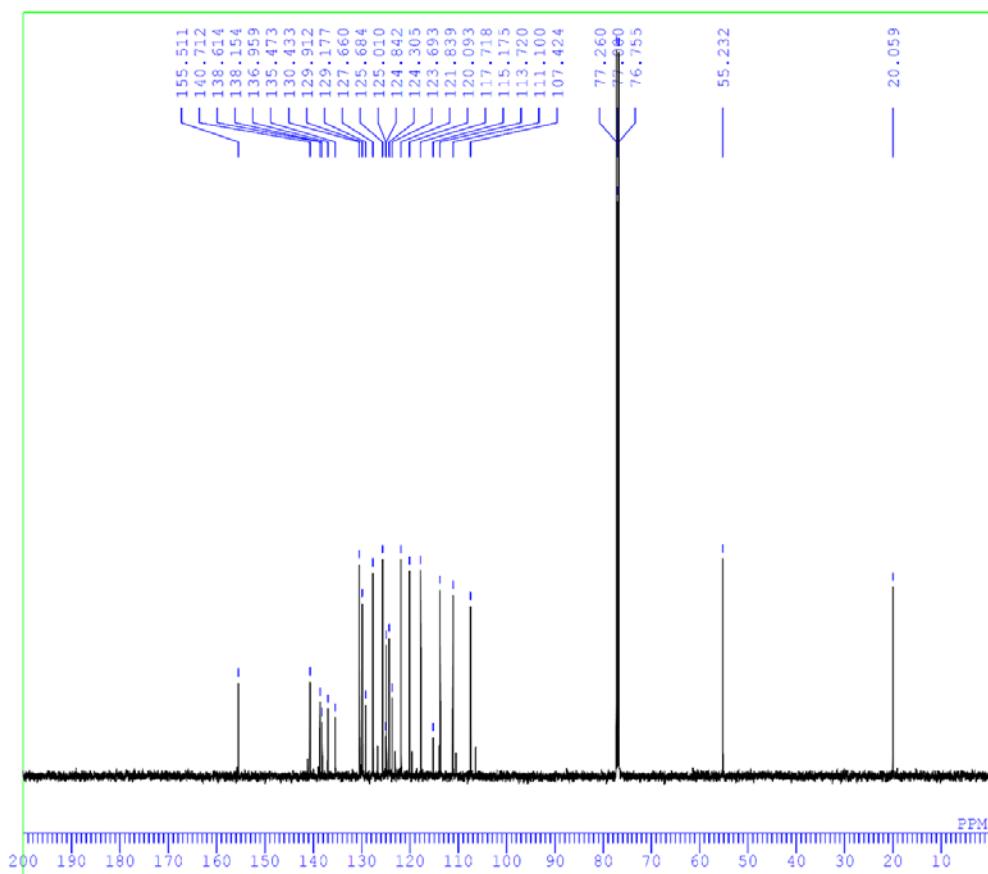


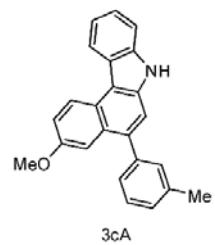
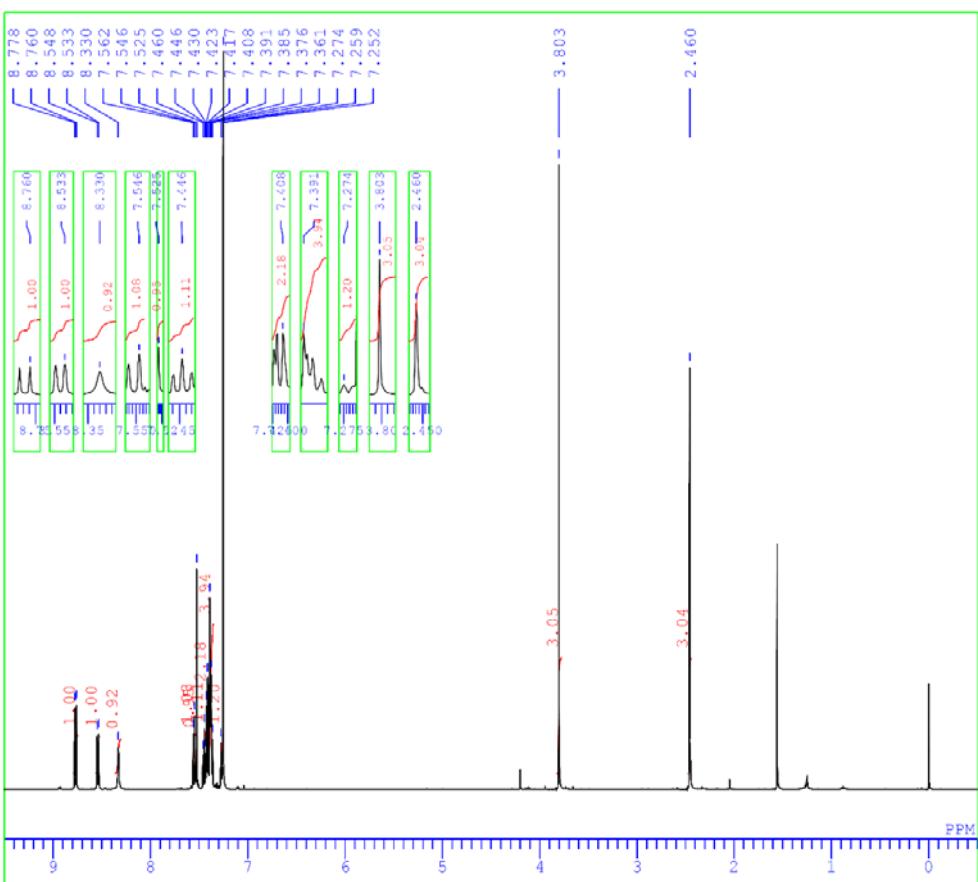


```

DFILE o-methyl c-1.als
COMNT
DATIM 2018-03-06 15:22:28
OBNUC 13C
EXMD singe_pulse dec
OBRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 13107
FREQU 25252.14 Hz
SCANS 1425
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56

```

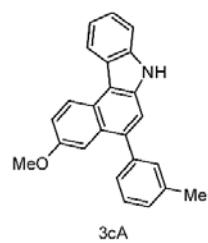
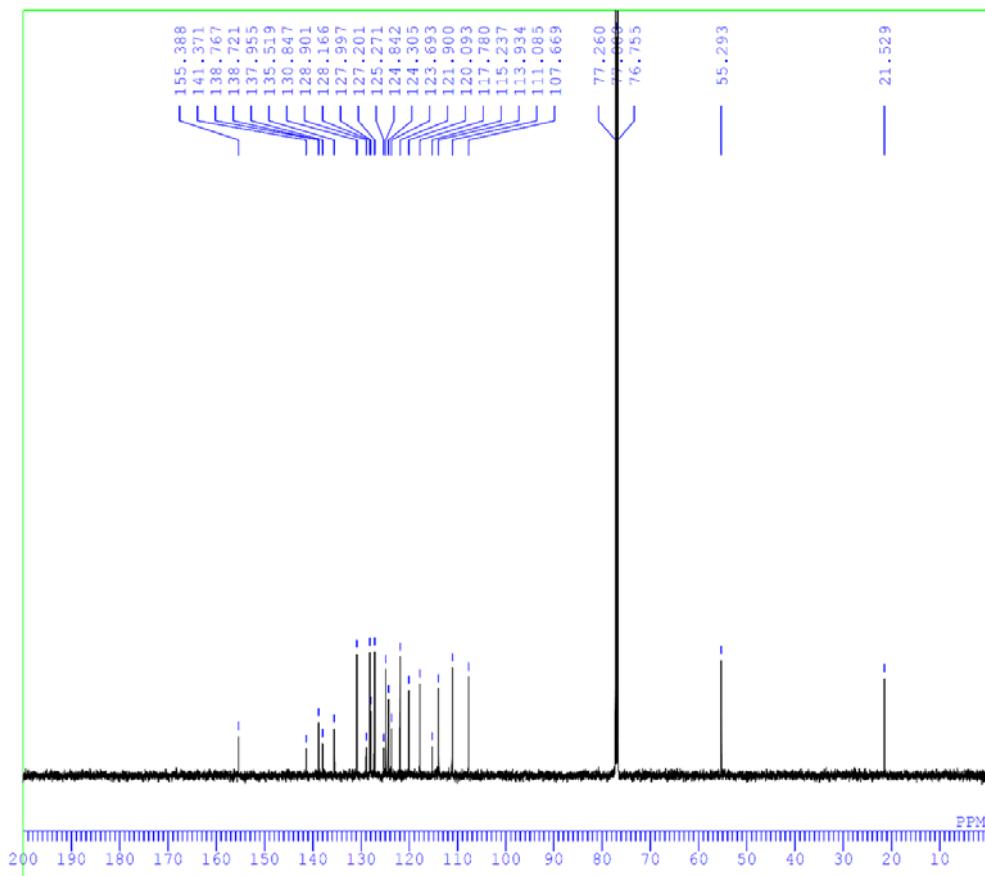


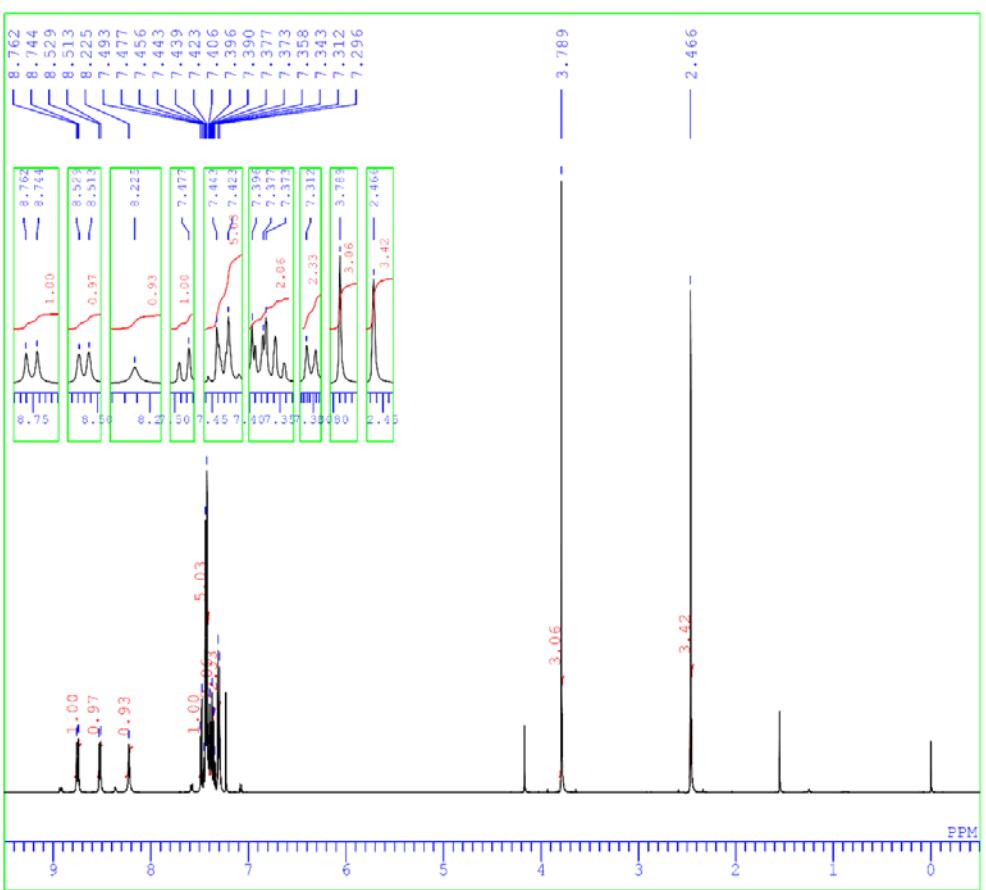


```

DFILE m-methyl c-1.als
COMNT
DATIM 2018-03-05 15:37:39
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 13107
FREQU 25252.14 Hz
SCANS 2216
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56

```

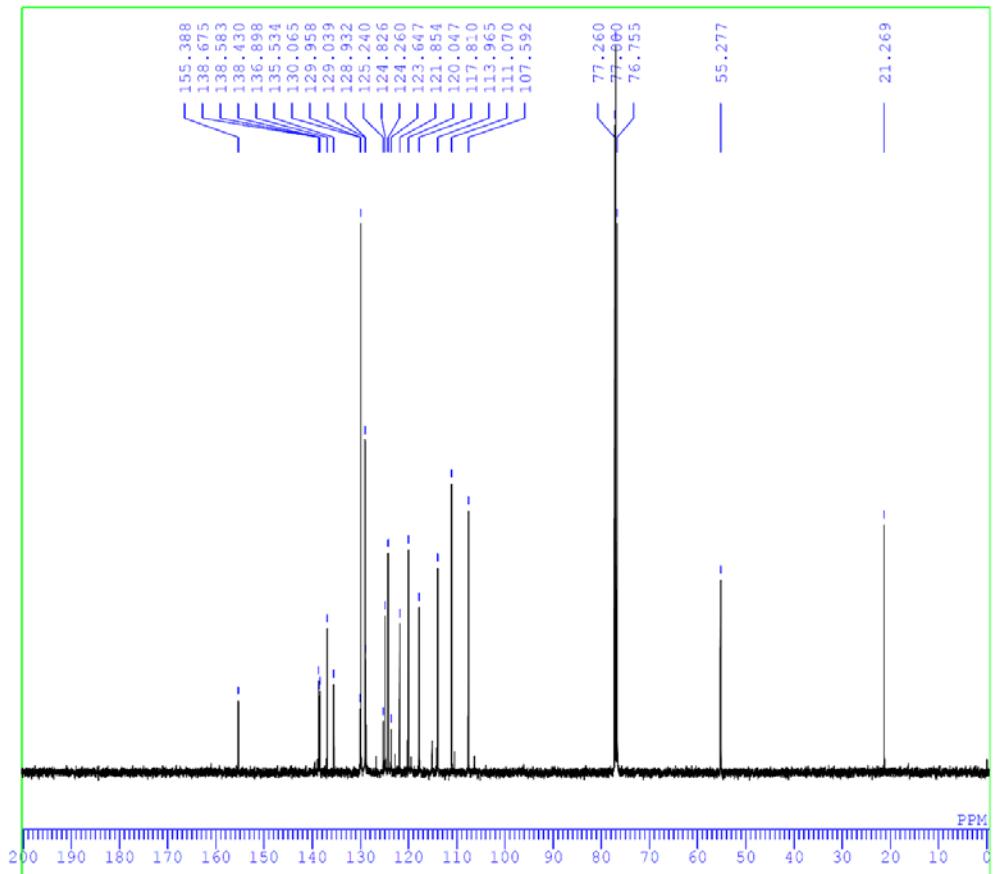
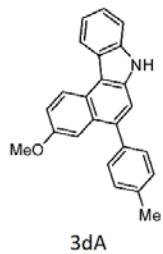




```

DFILE p-methyl_h-1.als
COMNT single pulse
DATIM 2018-03-05 16:55:01
OBNUC 1H
EXMOD single_pulse.ex2
OBFREQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PWL 7.15 usec
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

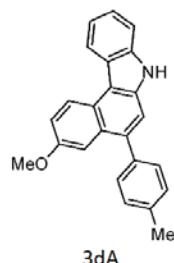
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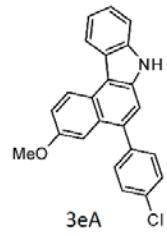
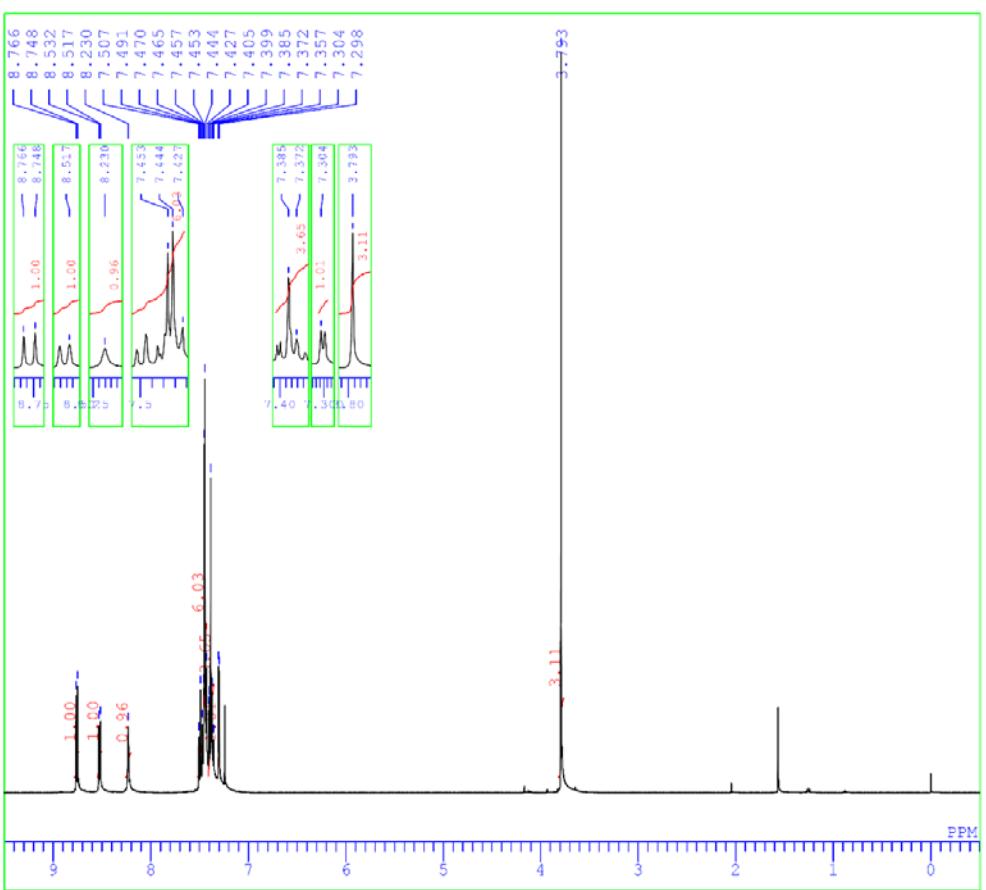


```

DFILE p-methyl c-1.als
COMNT
DATIM 2018-03-05 17:35:48
OBNUC 13C
EXMOD single_pulse_dec
OBFREQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 13107
FREQU 25252.14 Hz
SCANS 1567
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58

```

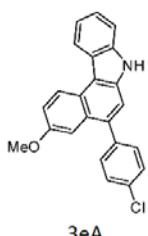
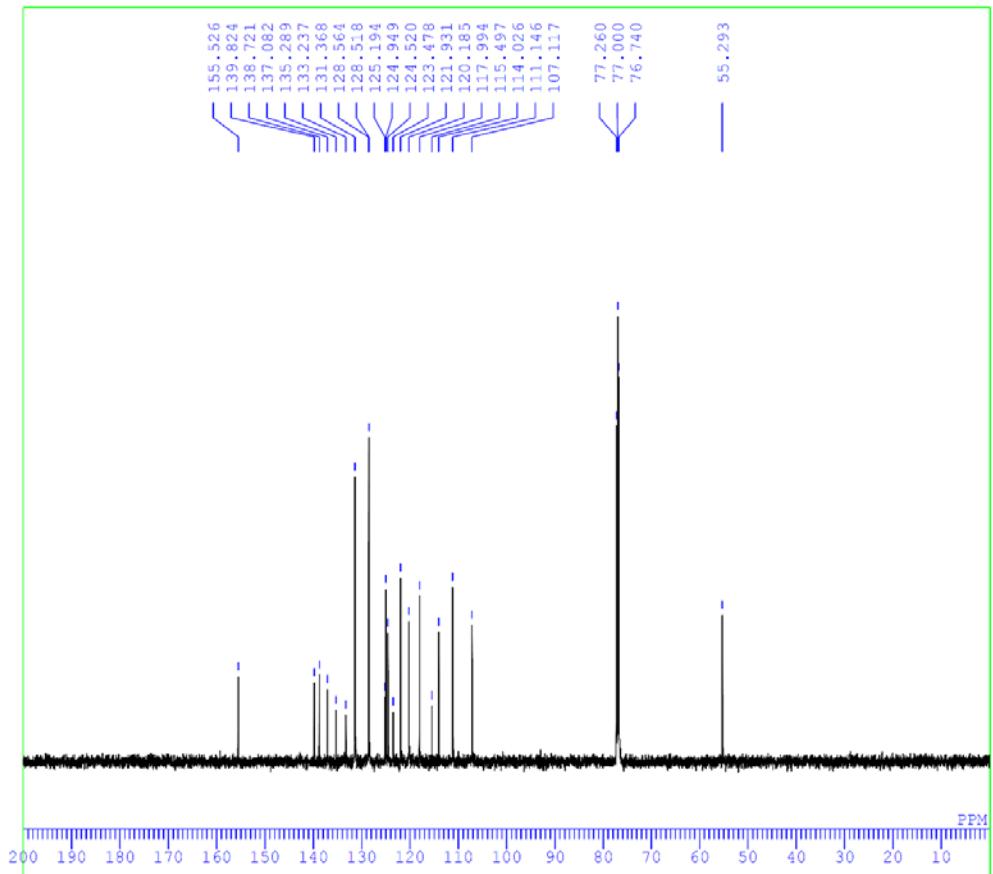


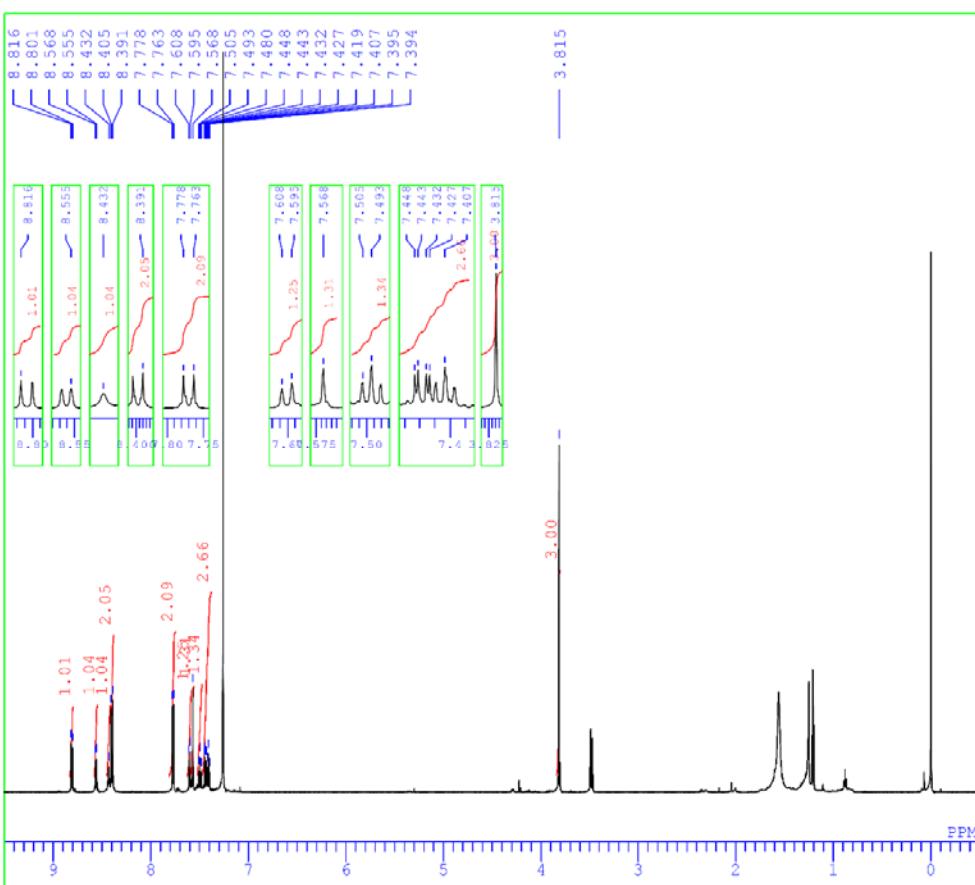


```

DFILE chloro c-1.als
COMNT
DATIM 2018-02-21 15:10:38
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 13107
FREQU 25252.14 Hz
SCANS 616
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56

```

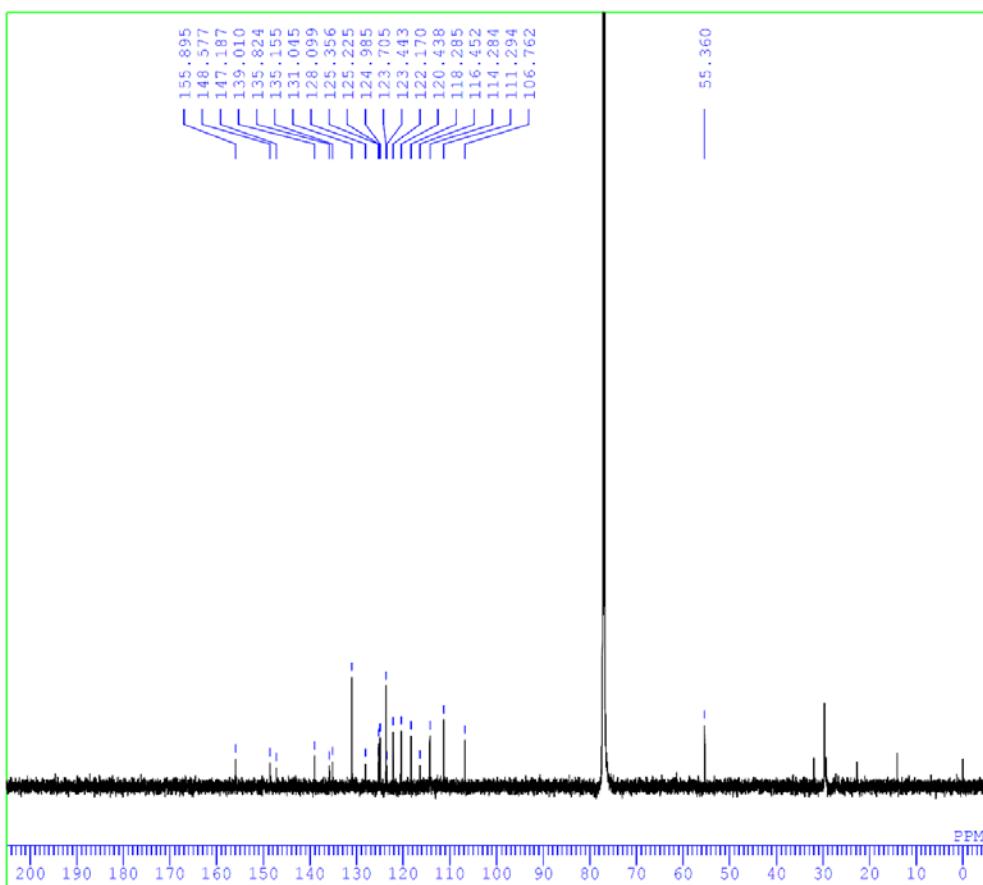
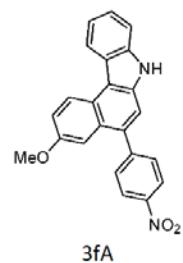




```

DFILE nitro_h-1.als
COMNT
DATIM 2018-03-08 14:09:33
OBNUC 1H
EXMOD zg30
OBFRQ 600.13 MHz
OBSET 3.00 kHz
OBFIN 0.65 Hz
POINT 16384
FREQU 8992.81 Hz
SCANS 128
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 24.9 c
SLVNT CDCl3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 574

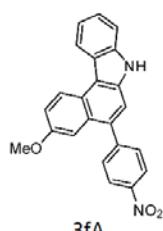
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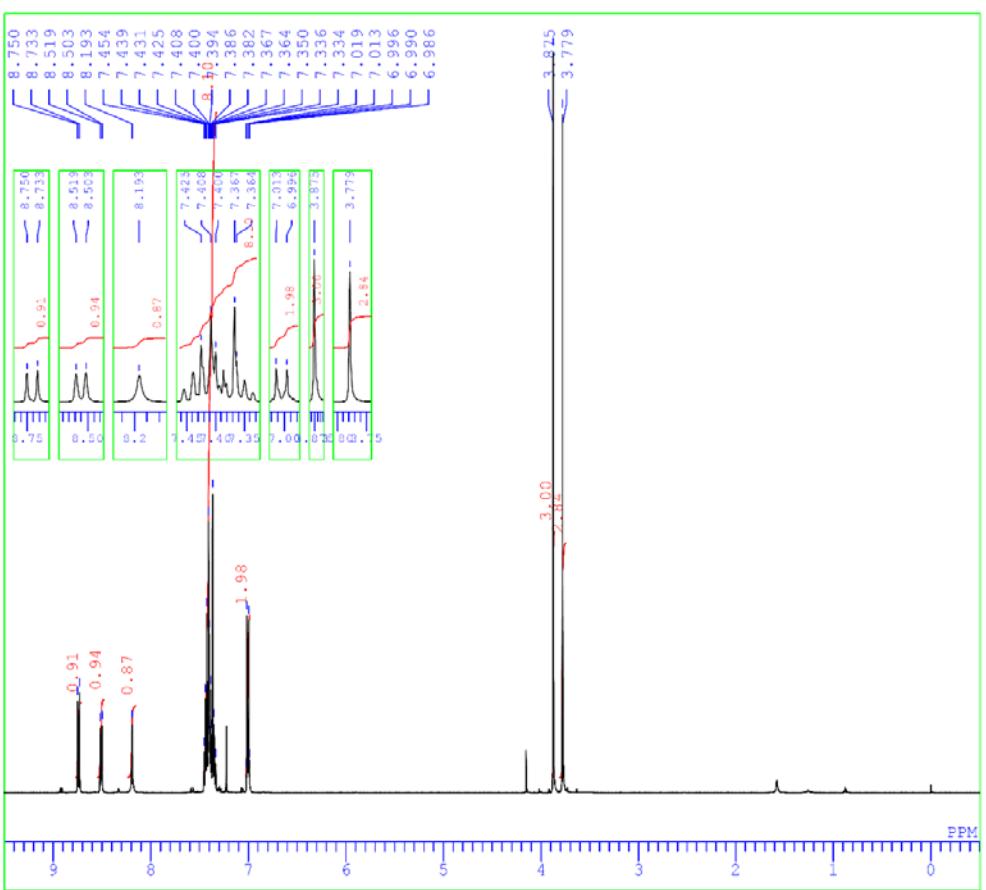


```

DFILE nitro_c-1.als
COMNT
DATIM 2018-03-23 06:58:45
OBNUC 13C
EXMOD zgpg30
OBFRQ 150.91 MHz
OBSET 7.89 kHz
OBFIN 8.81 Hz
POINT 32768
FREQU 35971.22 Hz
SCANS 80000
ACQTM 0.0000 sec
PD 0.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 24.9 c
SLVNT CDCl3
EXREF 219.18 ppm
BF 1.20 Hz
RGAIN 4597

```

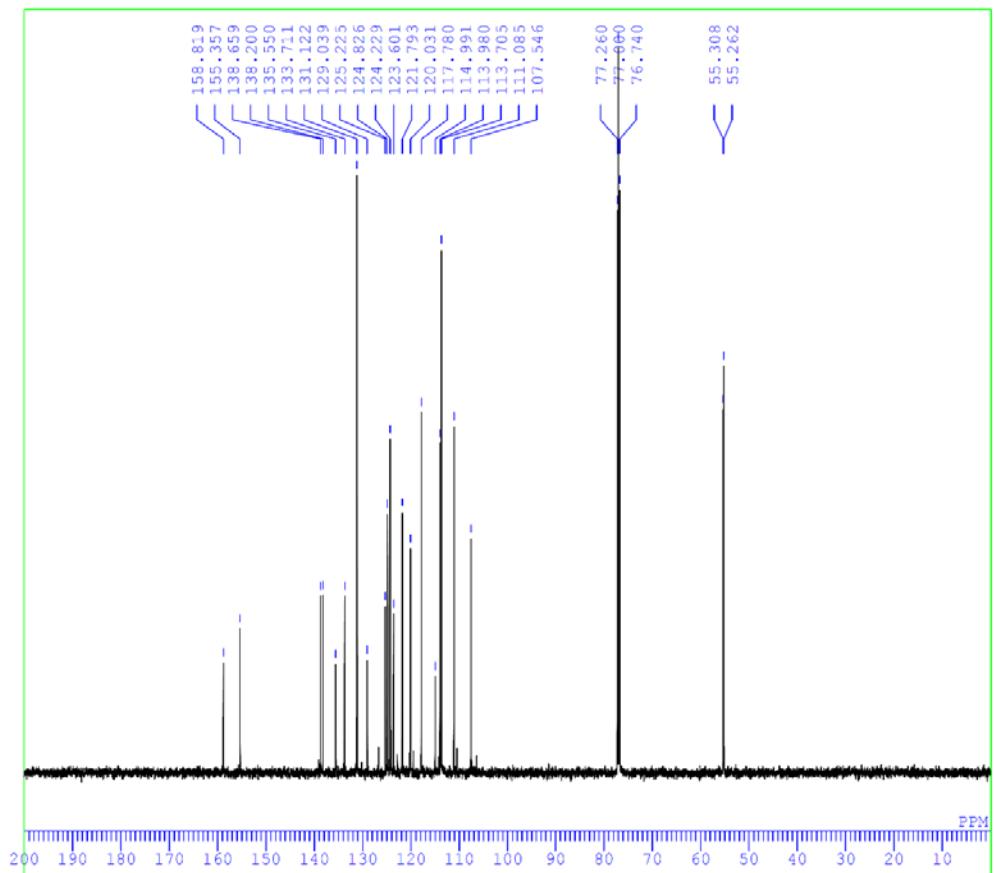
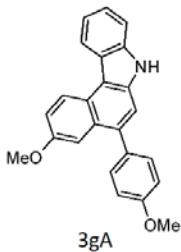




```

DFILE methoxy_h-1.als
COMNT single pulse
DATIM 2018-03-01 16:39:44
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40

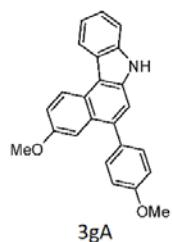
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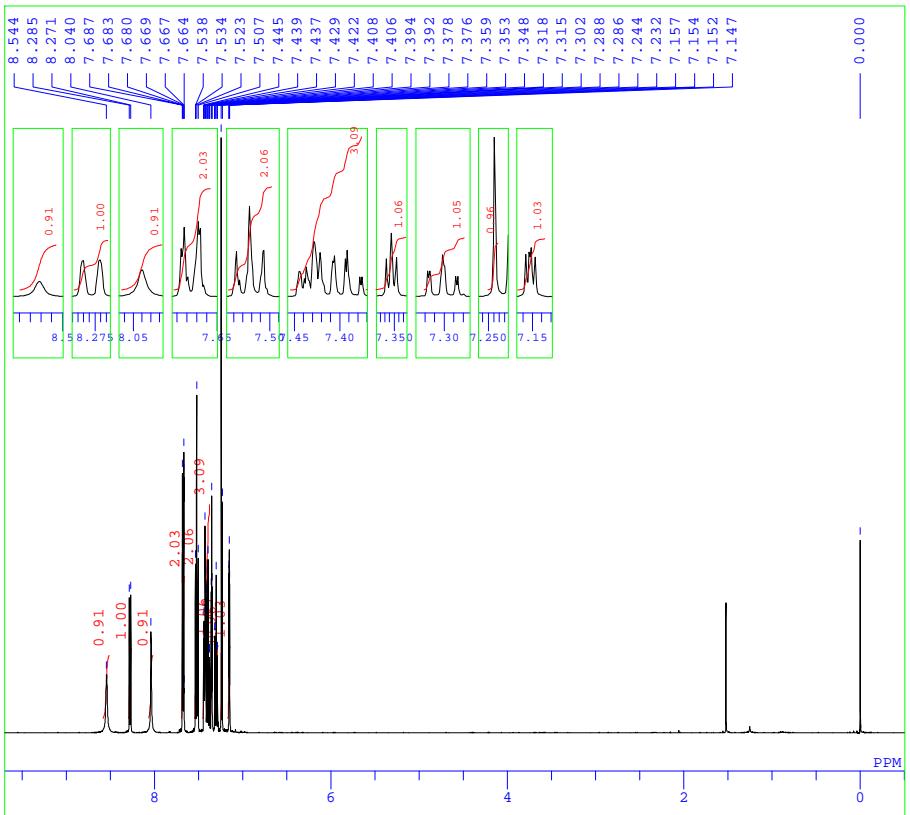


```

DFILE methoxy c-1.als
DATIM 2018-03-01 17:13:56
OBNUC 13C
EXMOD single_pulse dec
OFREQ 125.77 MHz
OBSET 7.87 KHz
OEFIN 4.21 Hz
POINT 13107
FREQU 25252.14 Hz
SCANS 1332
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

```

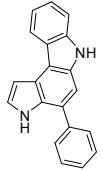




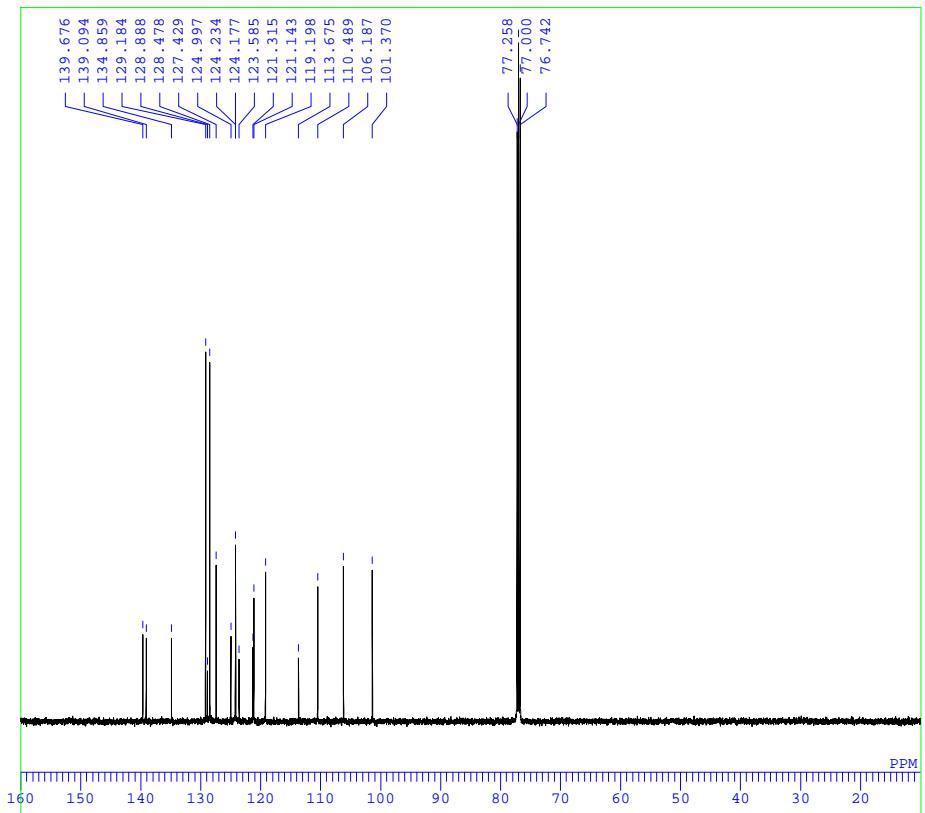
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DFILE 6aA H-1.als
COMNT single_pulse
DATIM 2017-02-23 21:40:53
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

```



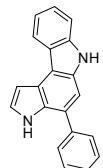
6aA



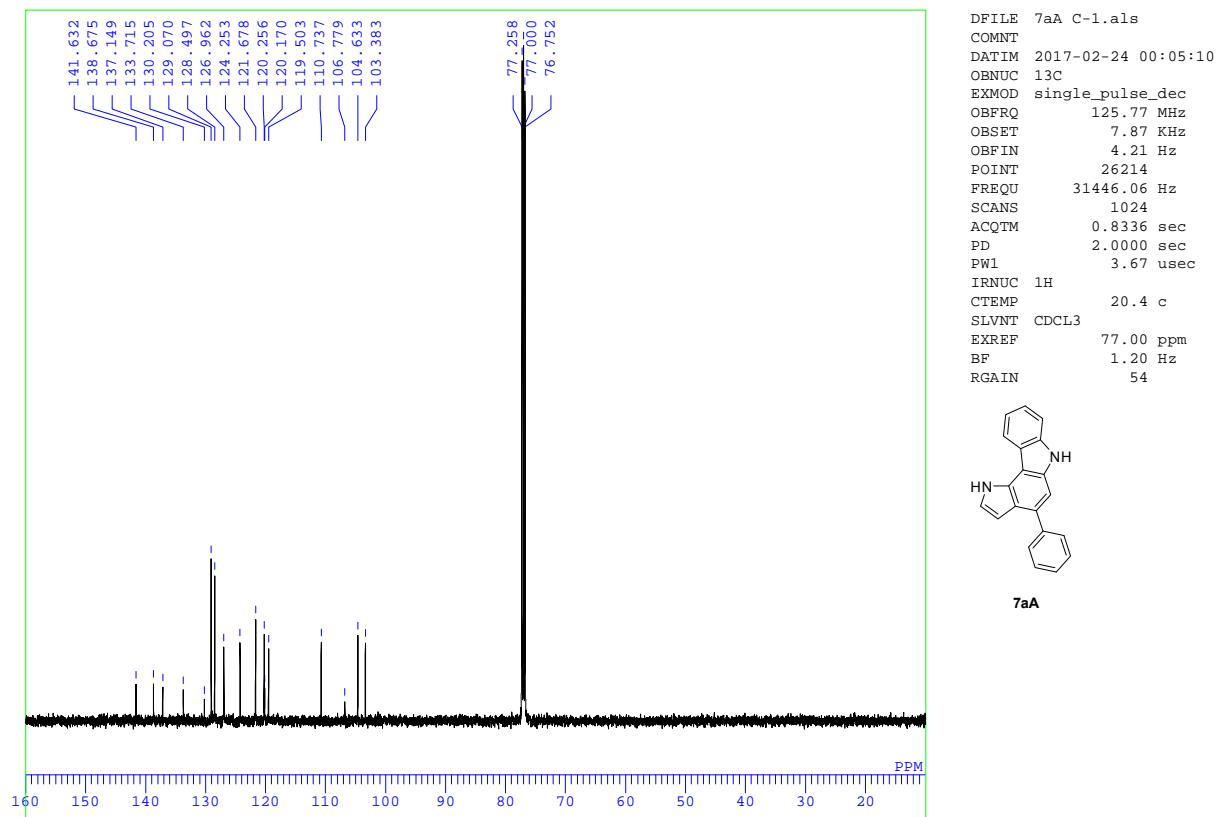
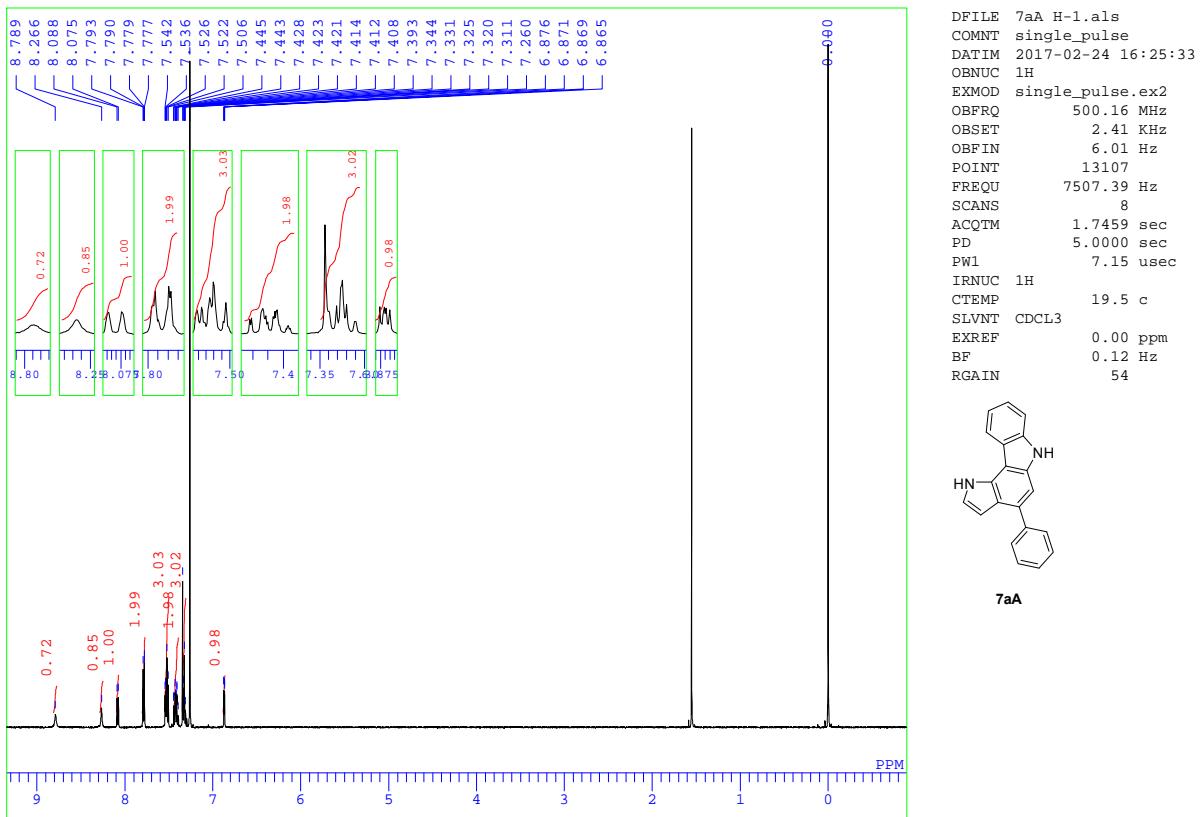
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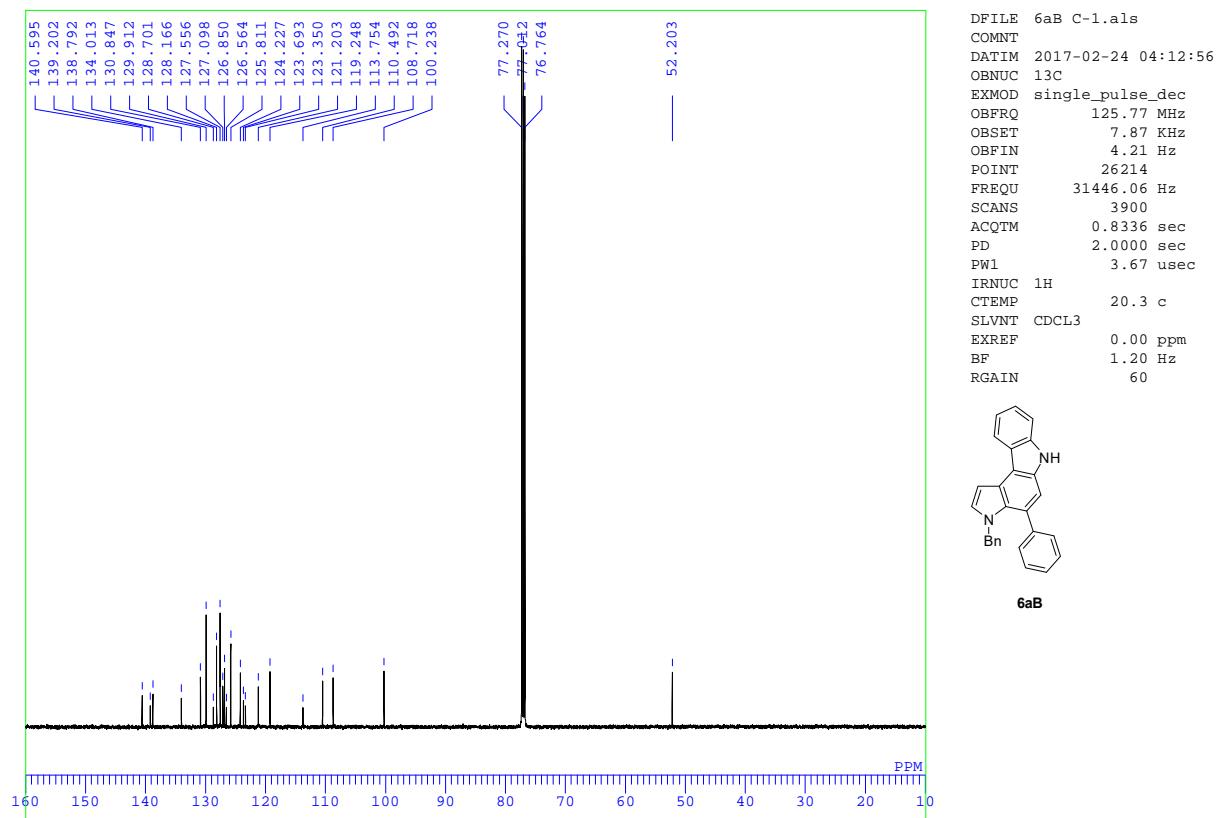
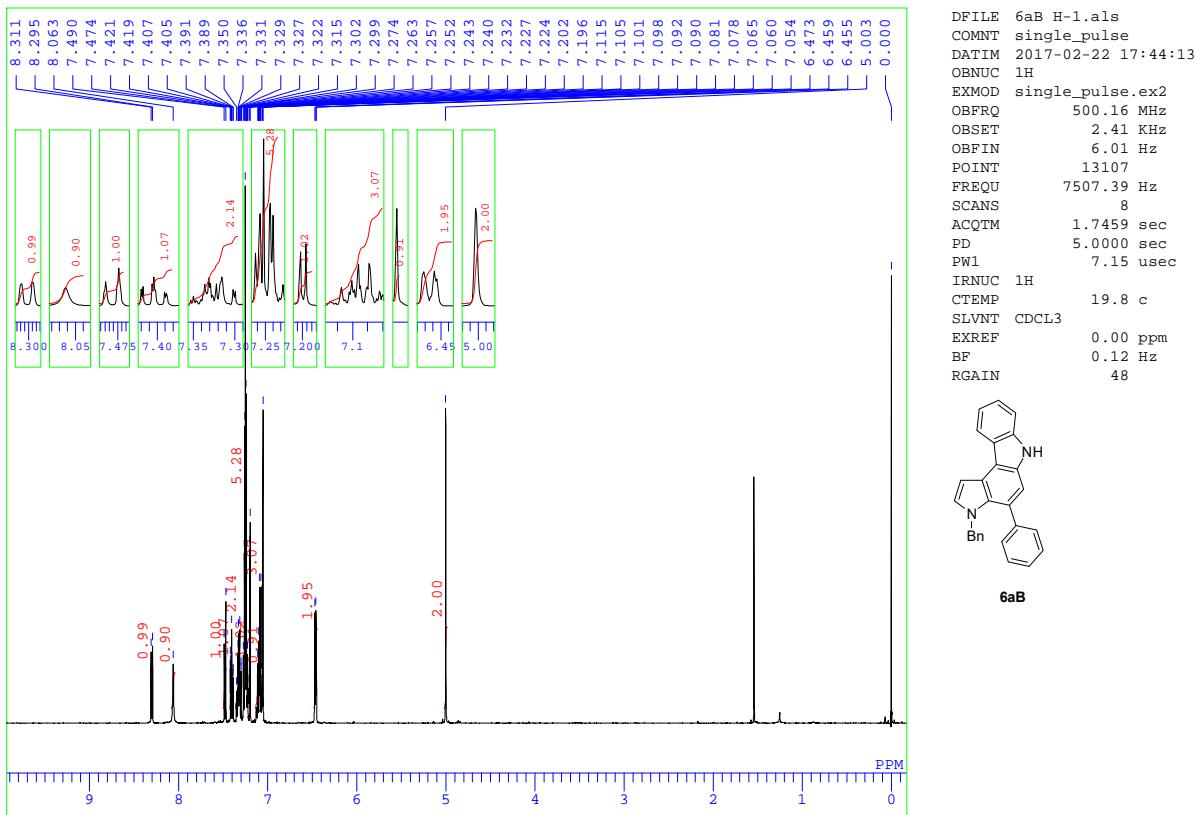
DFILE 6aA C-1.als
COMNT
DATIM 2017-02-24 01:01:33
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

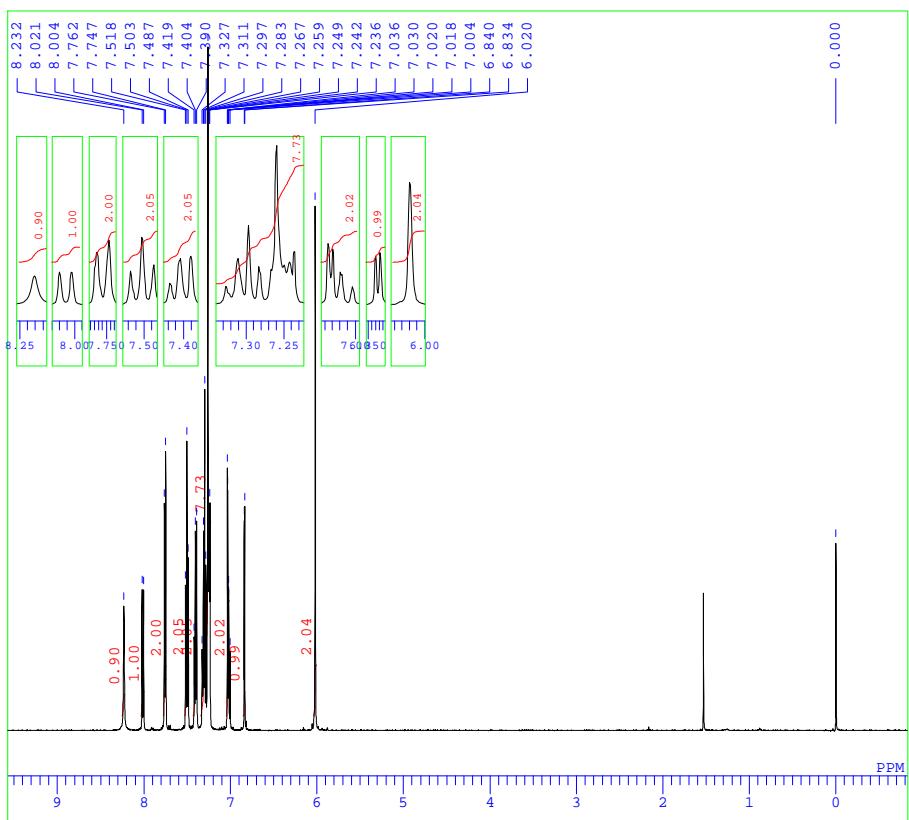
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6aA



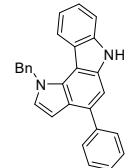




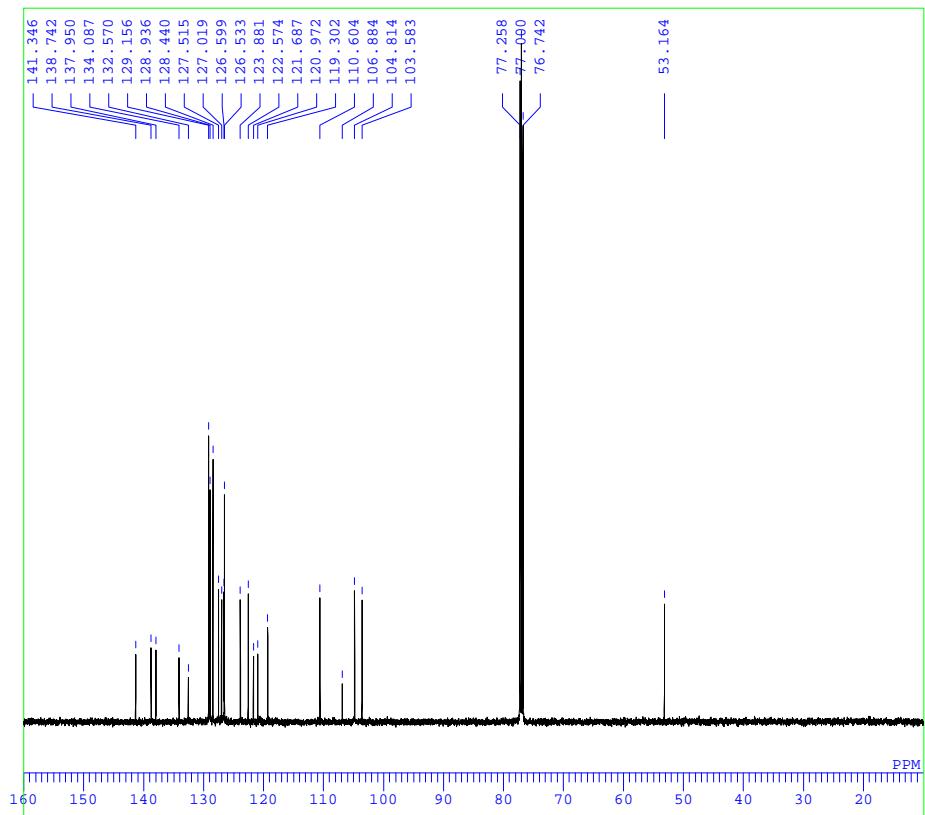
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DFILE 7aB H-1.als
COMNT single_pulse
DATIM 2017-02-22 17:49:26
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ      500.16 MHz
OBSET       2.41 kHz
OBFIN       6.01 Hz
POINT        13107
FREQU     7507.39 Hz
SCANS         8
ACQTM      1.7459 sec
PD          5.0000 sec
PW1          7.15 usec
IRNUC    1H
CTEMP      19.8 c
SLVNT    CDCL3
EXREF      0.00 ppm
BF          0.12 Hz
RGAIN       44

```



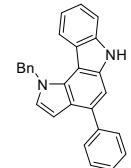
7aB



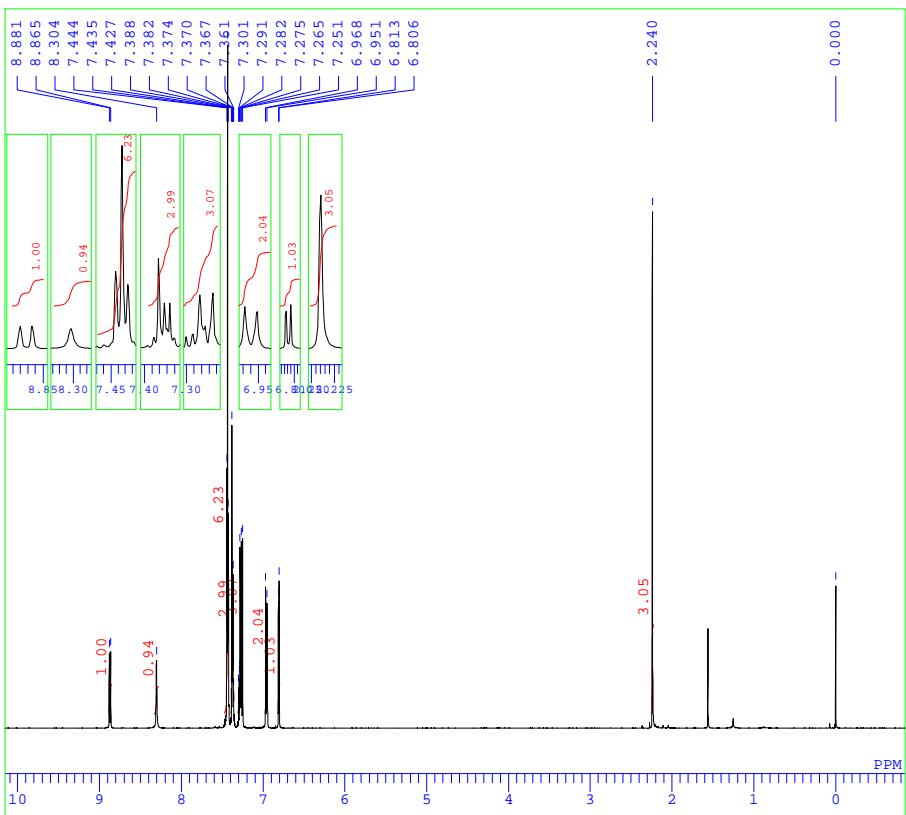
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DFILE 7aB C-1.als
COMNT
DATIM 2017-02-24 05:06:42
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

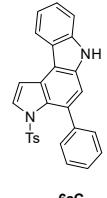
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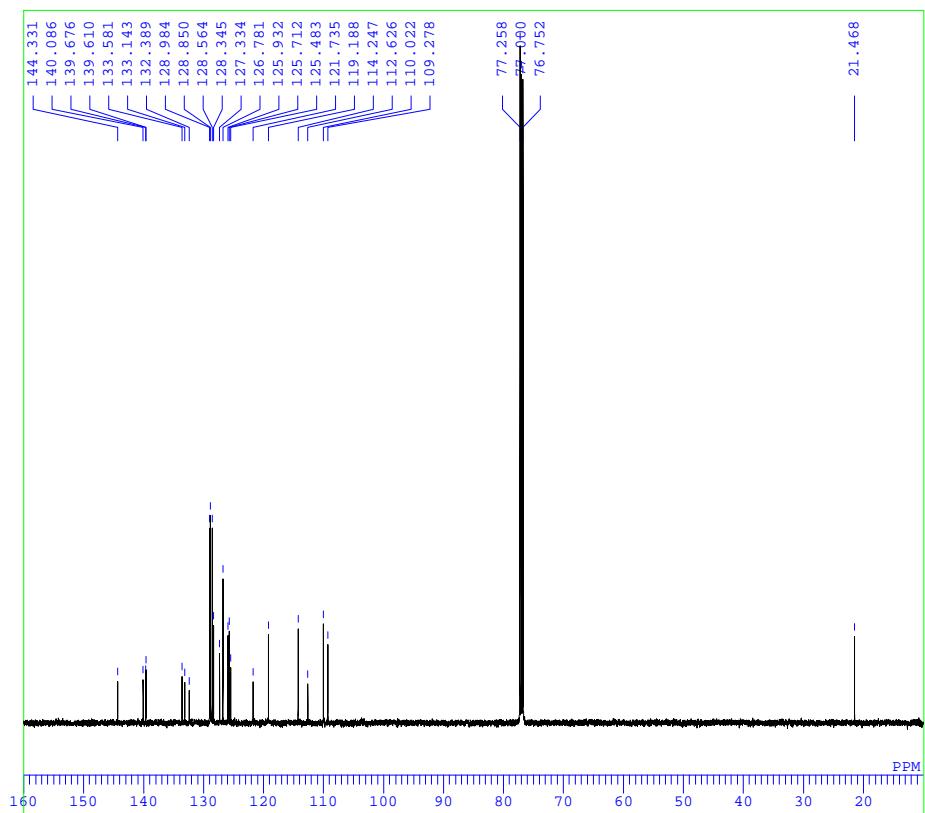
7aB



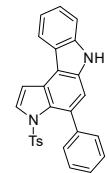
DFILE 6aC H-1.als
 COMNT single_pulse
 DATIM 2017-02-22 17:53:52
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRQ 500.16 MHz
 OBSET 2.41 kHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 7.15 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL3
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 44



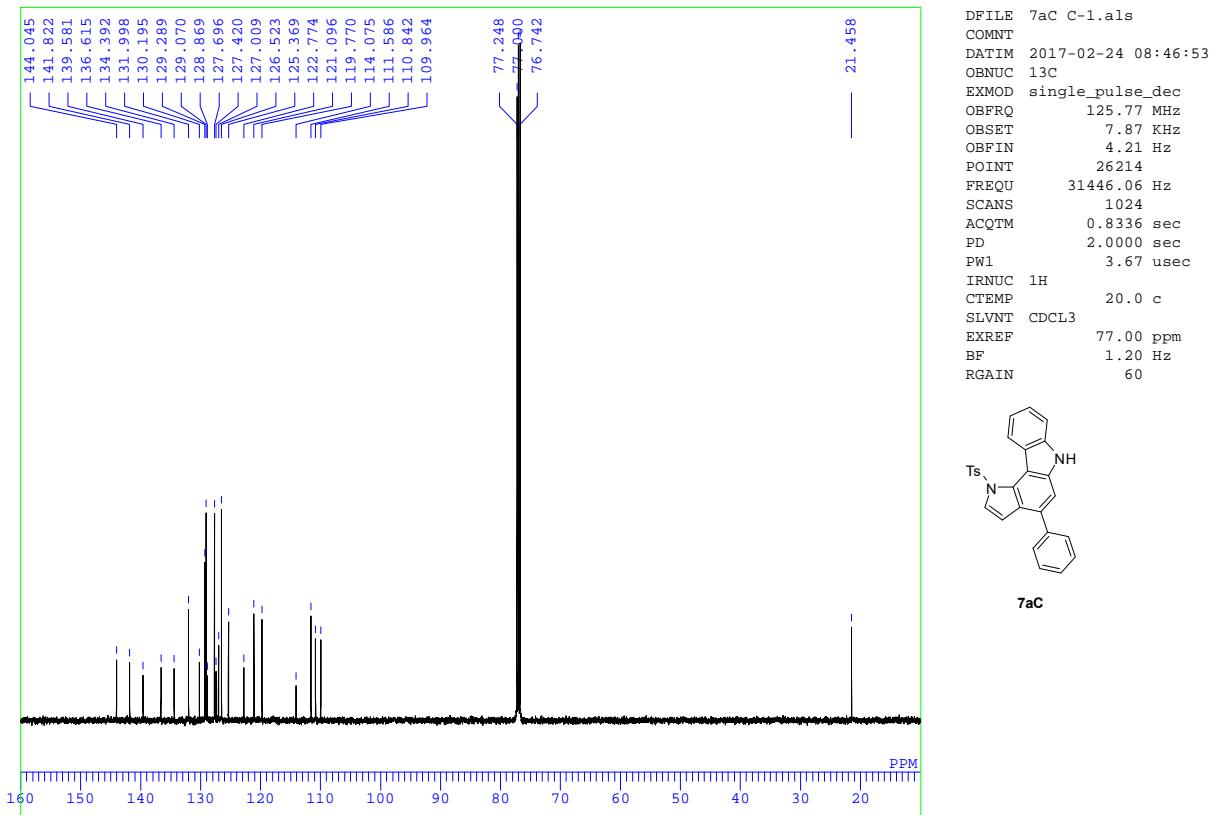
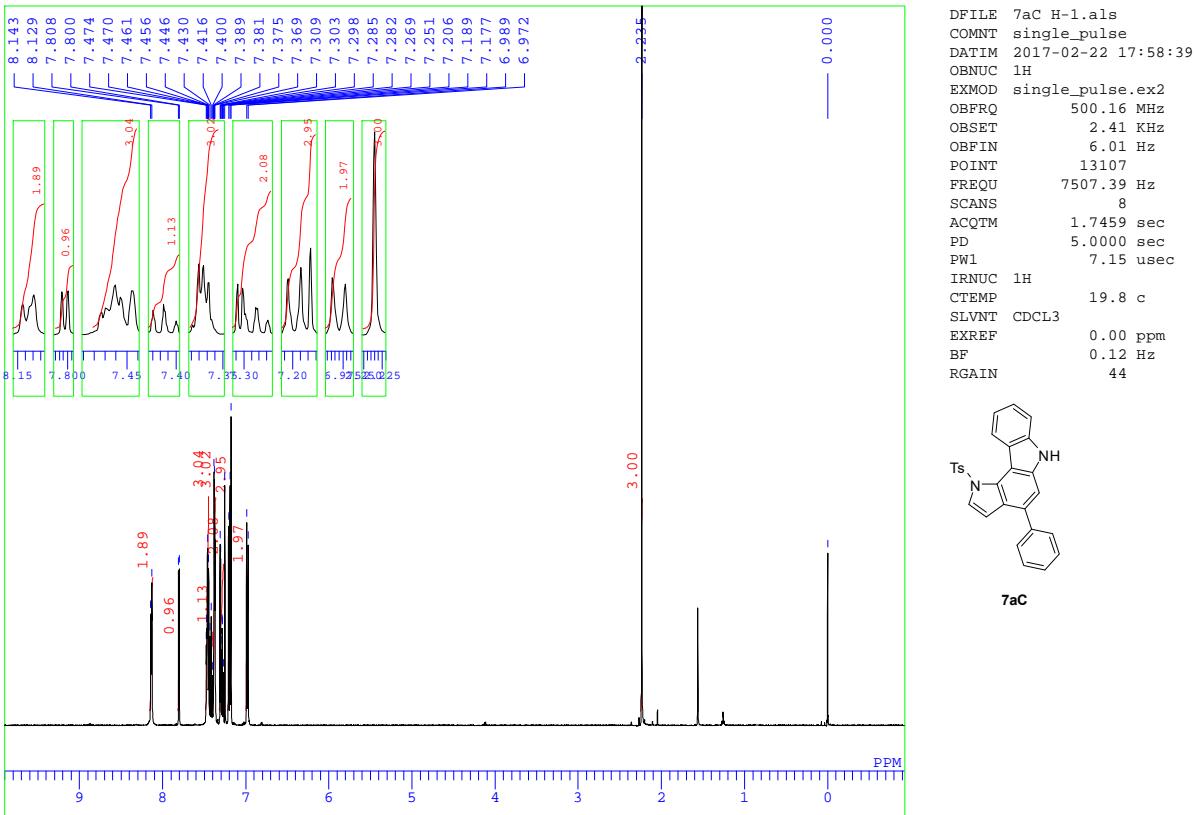
6aC

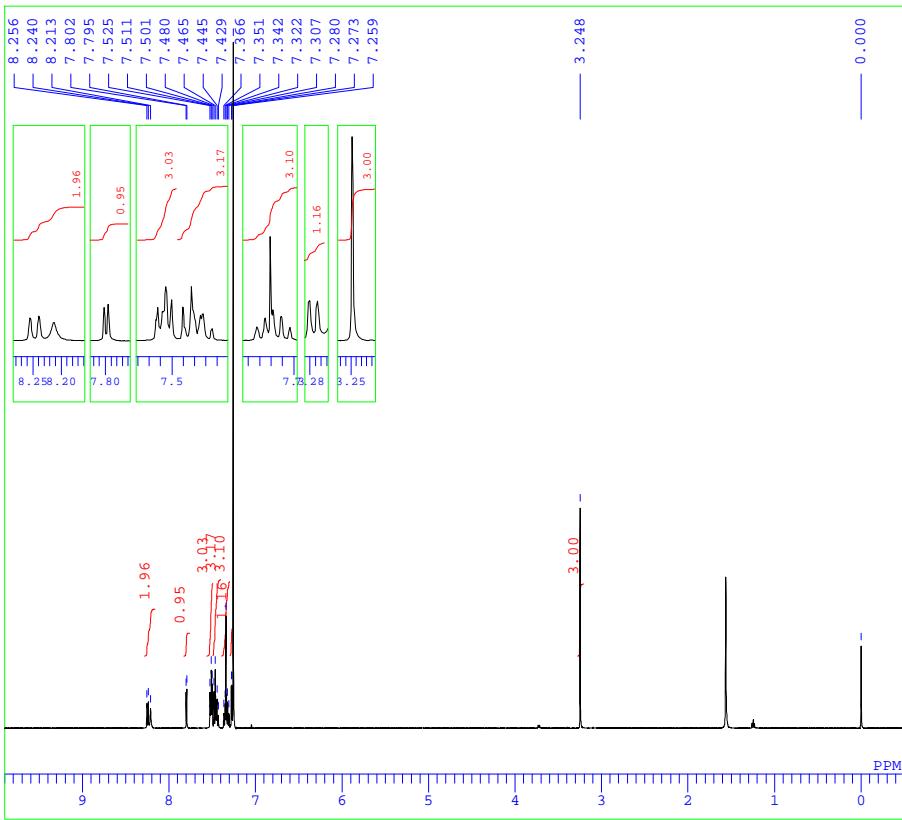


DFILE 6aC C-1.als
 COMNT
 DATIM 2017-02-24 07:51:52
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 kHz
 OBFIN 4.21 Hz
 POINT 26214
 FREQU 31446.06 Hz
 SCANS 1024
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 20.0 c
 SLVNT CDCL3
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



6aC

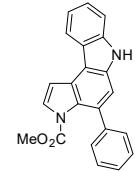




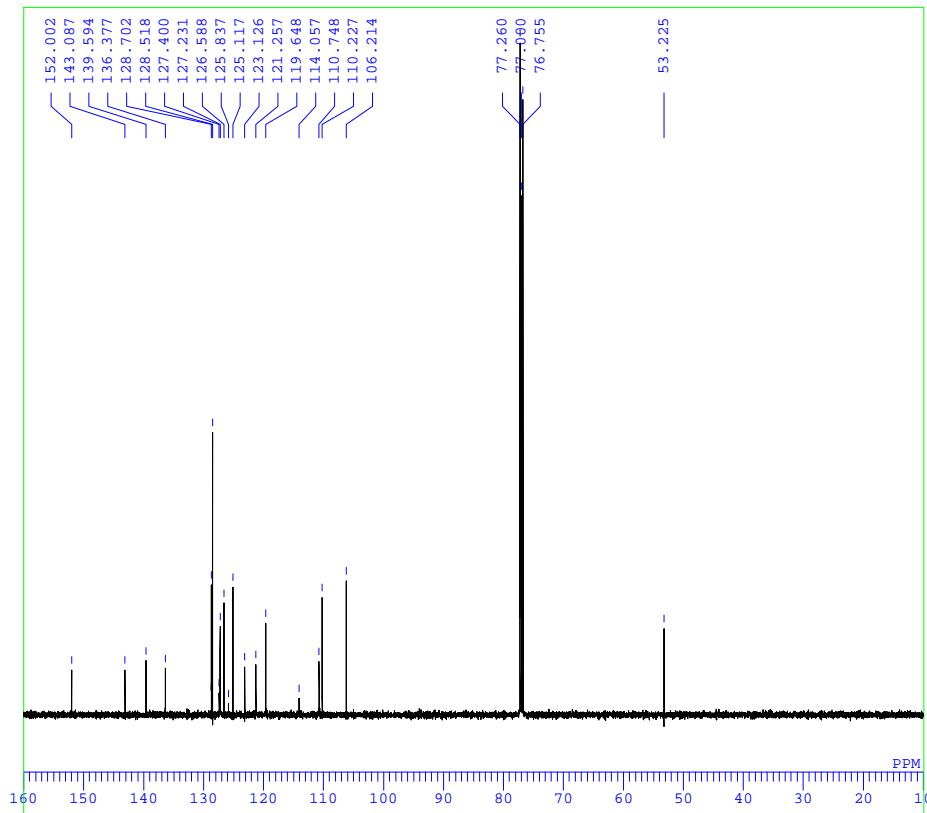
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DFILE 6aD.als
COMNT single_pulse
DATIM 2018-04-27 19:42:40
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 21.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

```



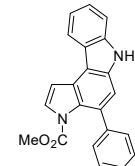
6aD



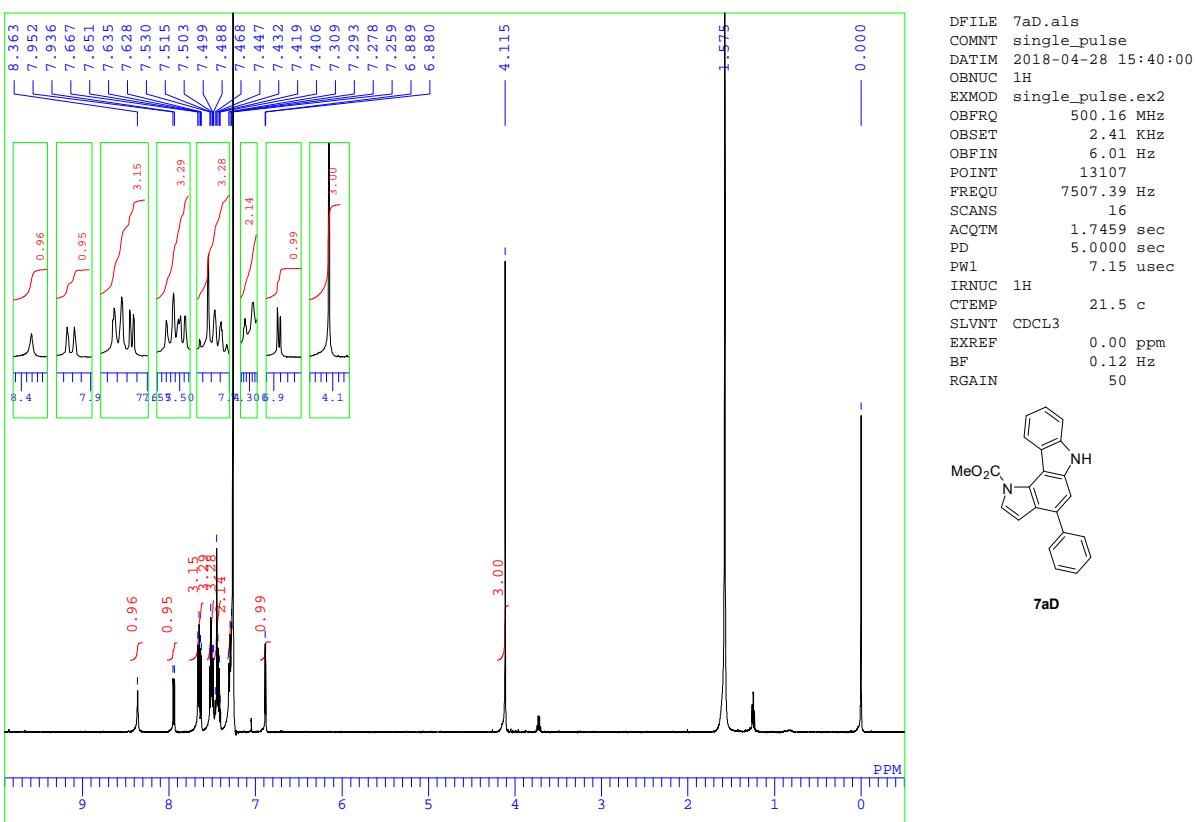
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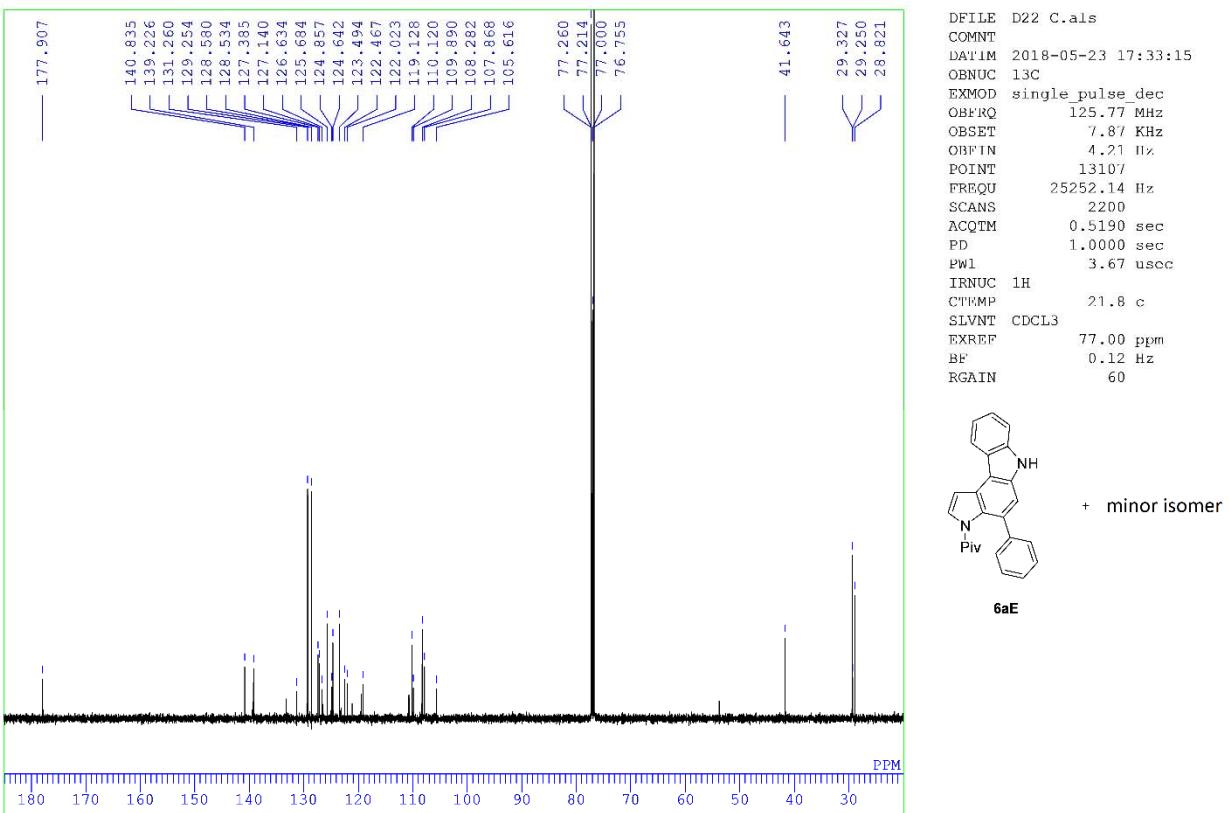
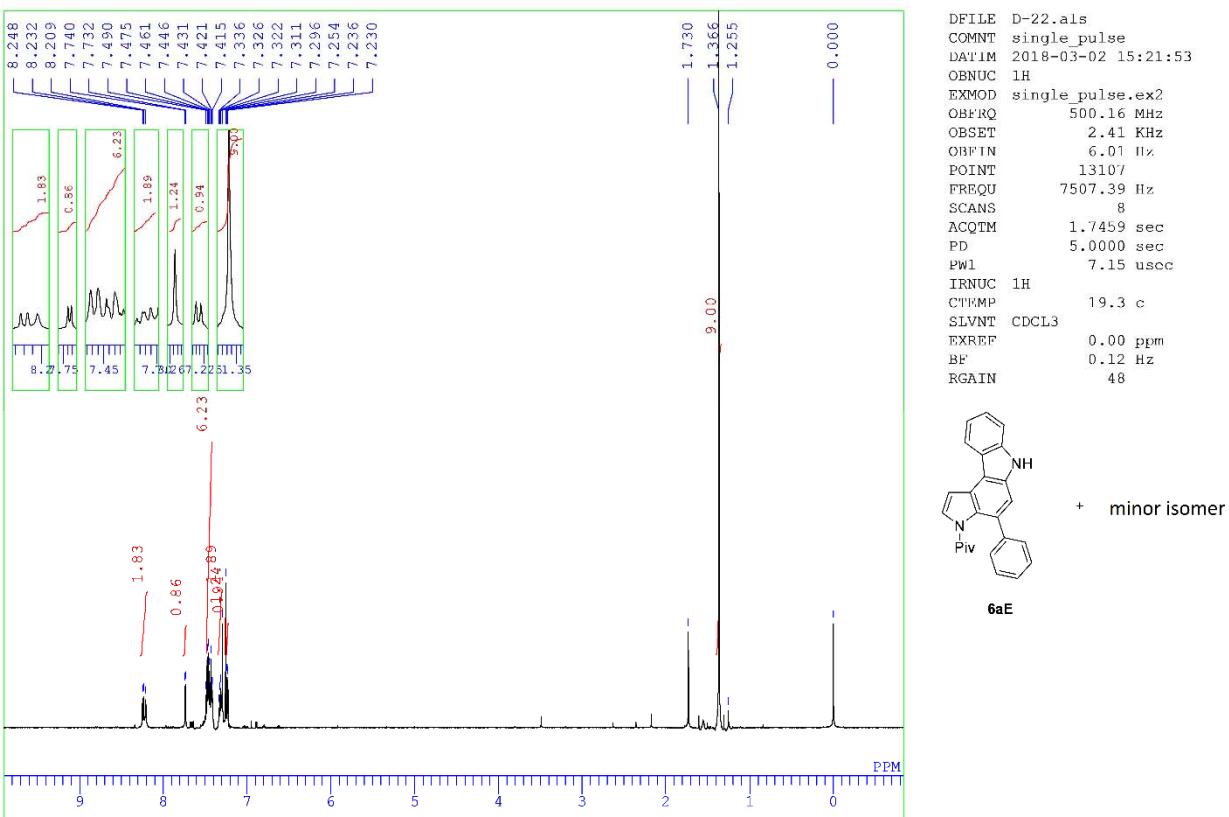
DFILE 6aD Cc-1.als
COMNT
DATIM 2018-04-29 18:34:57
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 13107
FREQU 25252.14 Hz
SCANS 3507
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

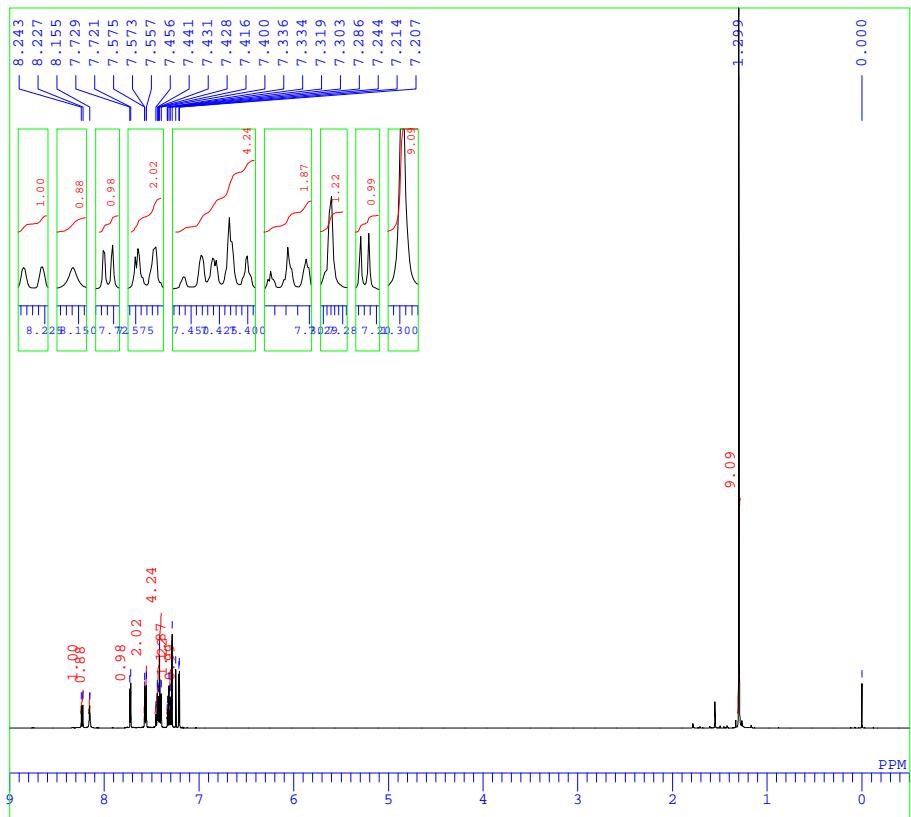
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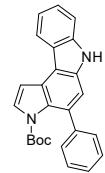
6aD



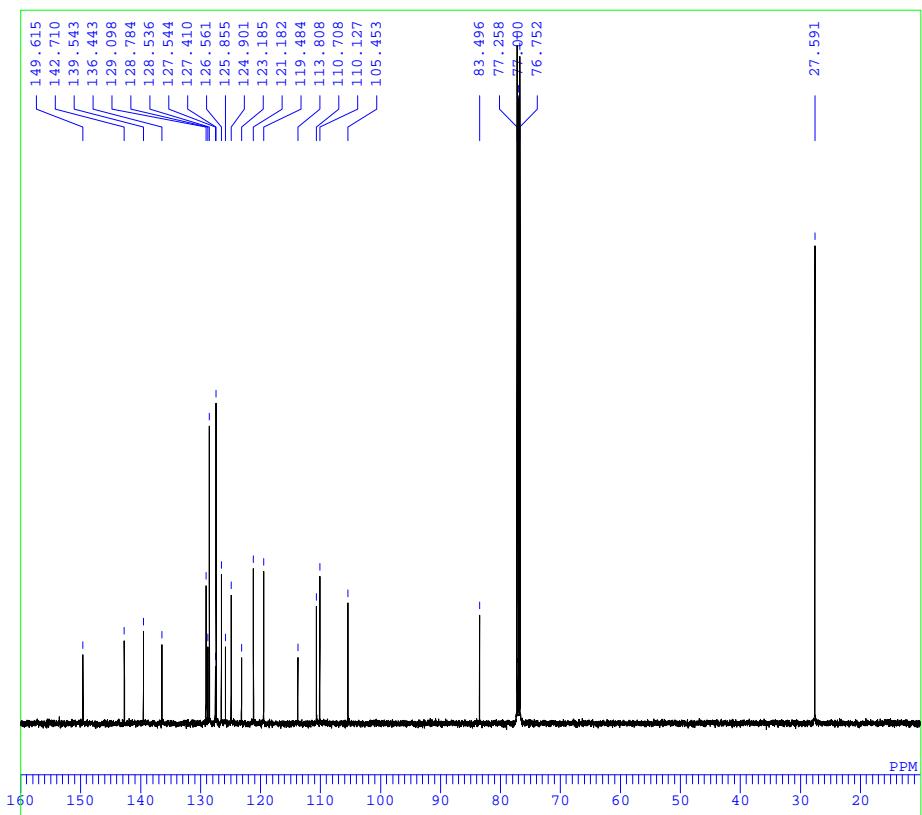




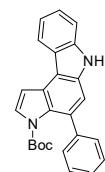
DFILE 6aF H-1.als
 COMNT single_pulse
 DATIM 2017-02-24 16:33:32
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRQ 500.16 MHz
 OBSET 2.41 kHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 7.15 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 44



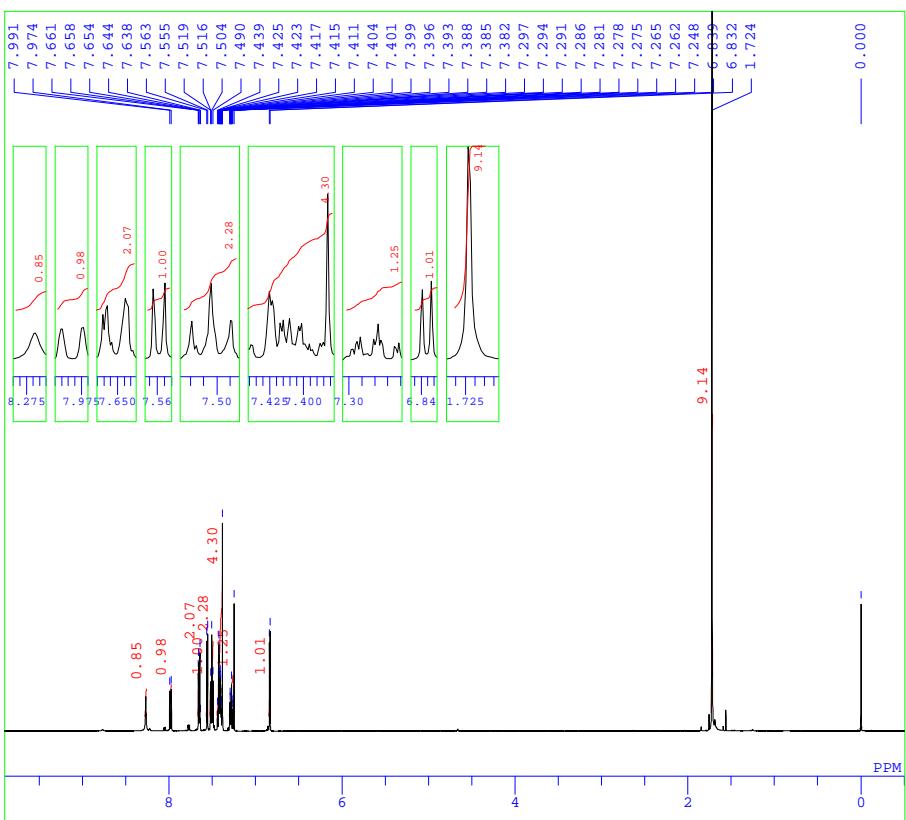
6aF



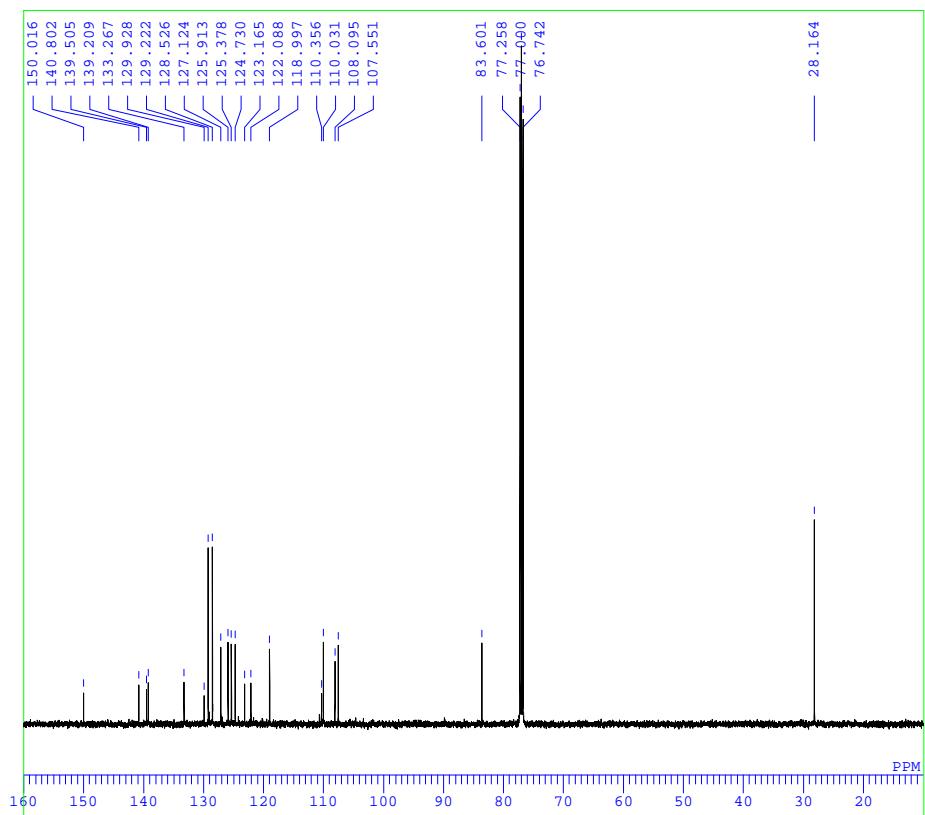
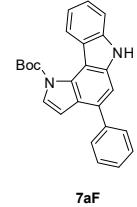
DFILE 6aF C-1.als
 COMNT
 DATIM 2017-02-24 06:01:34
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 kHz
 OBFIN 4.21 Hz
 POINT 26214
 FREQU 31446.06 Hz
 SCANS 1024
 ACQTM 0.8336 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 20.1 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



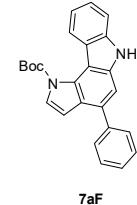
6aF

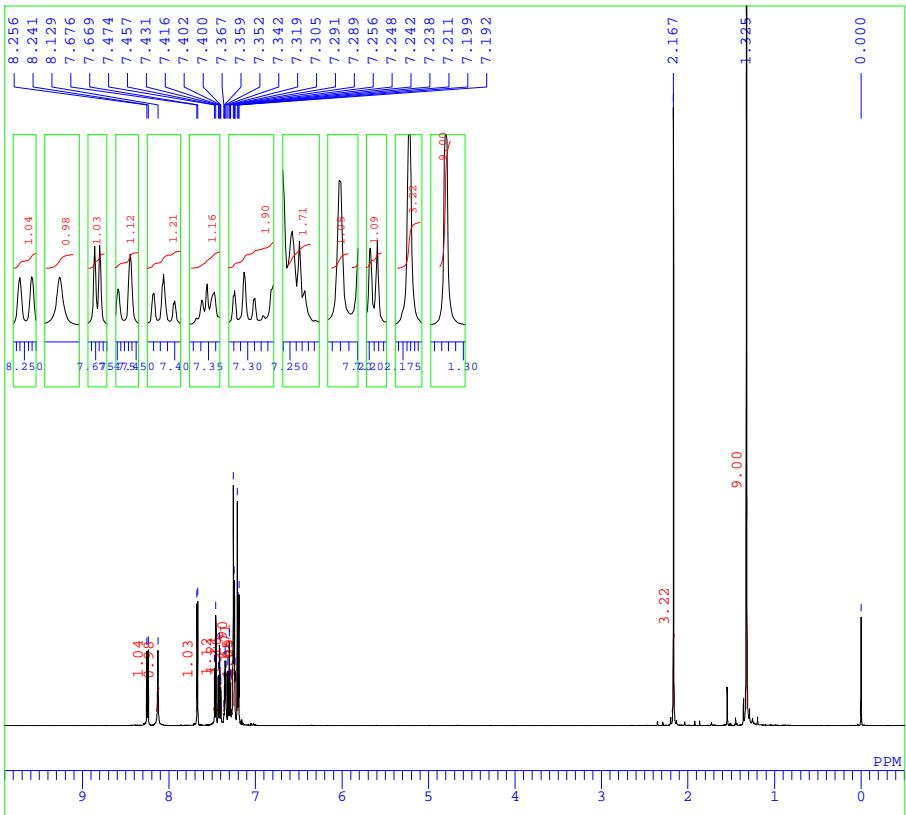


DFILE 7aF H-1.als
COMNT single_pulse
DATIM 2017-02-23 21:44:59
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44



DFILE 7aF C-1.als
COMNT
DATIM 2017-02-24 06:56:18
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

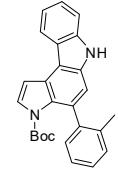




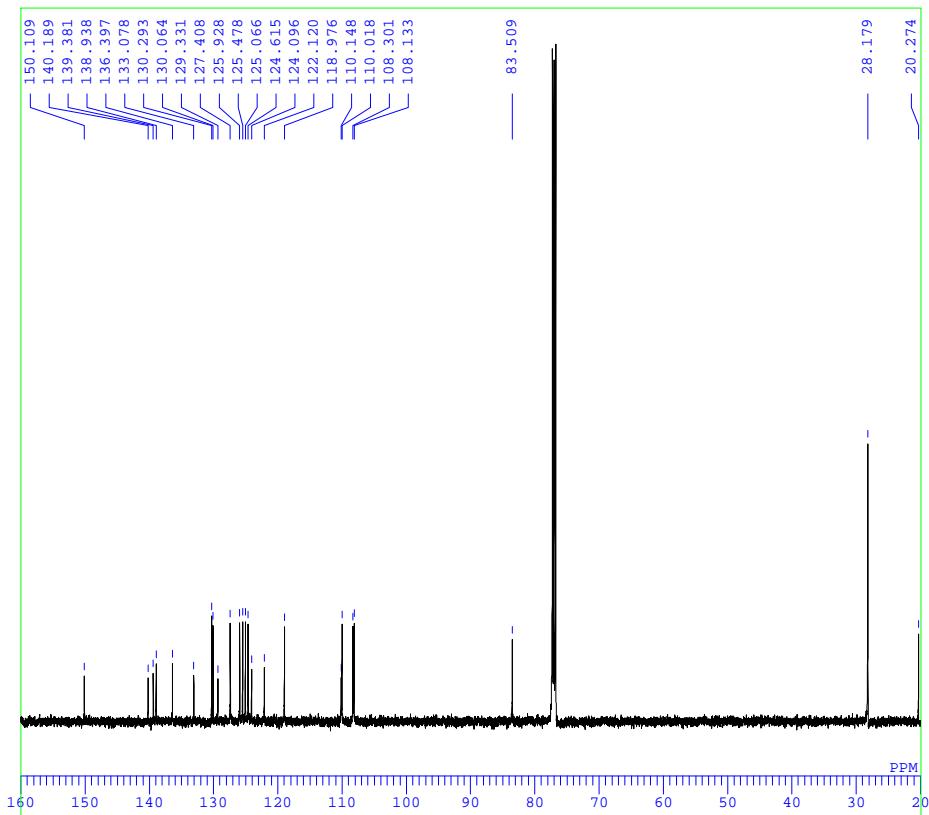
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DFILE C-34 ue H 1 26-1.als
COMNT single_pulse
DATIM 2017-01-26 21:24:44
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

```



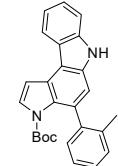
6bF



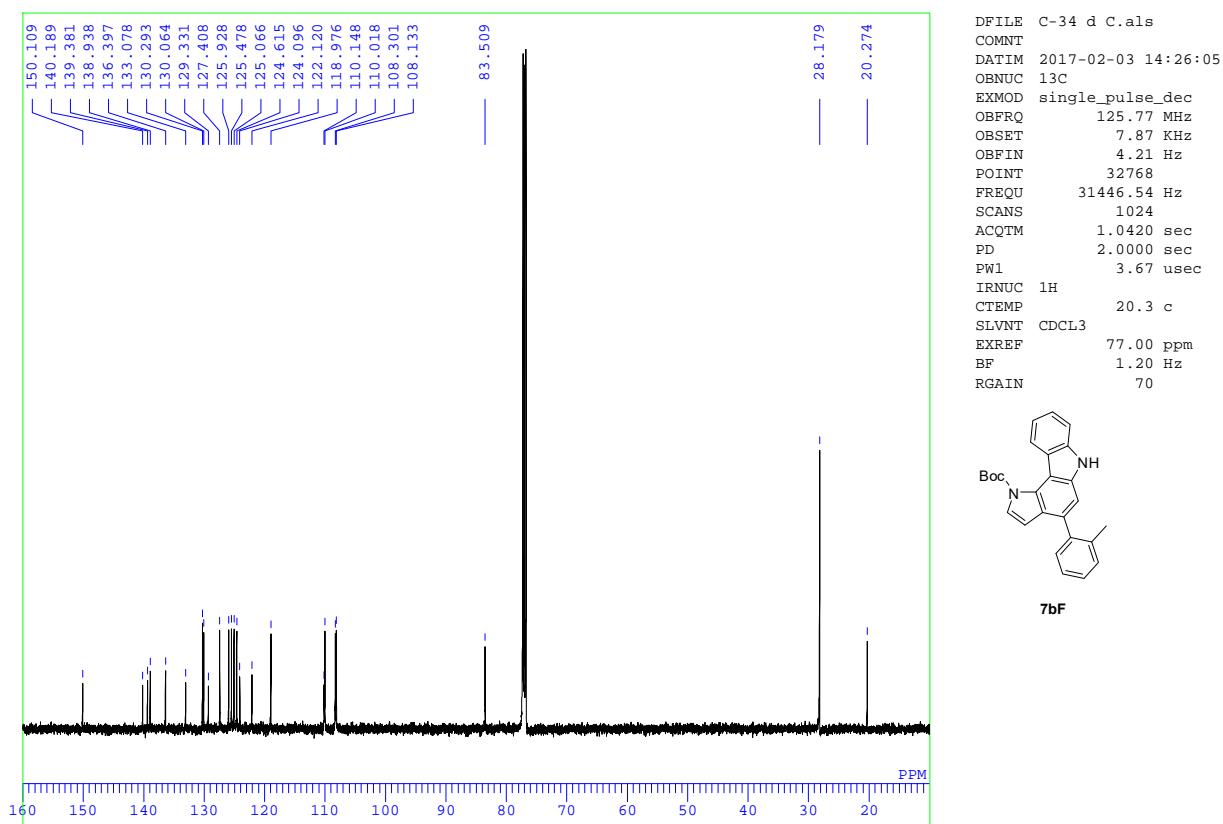
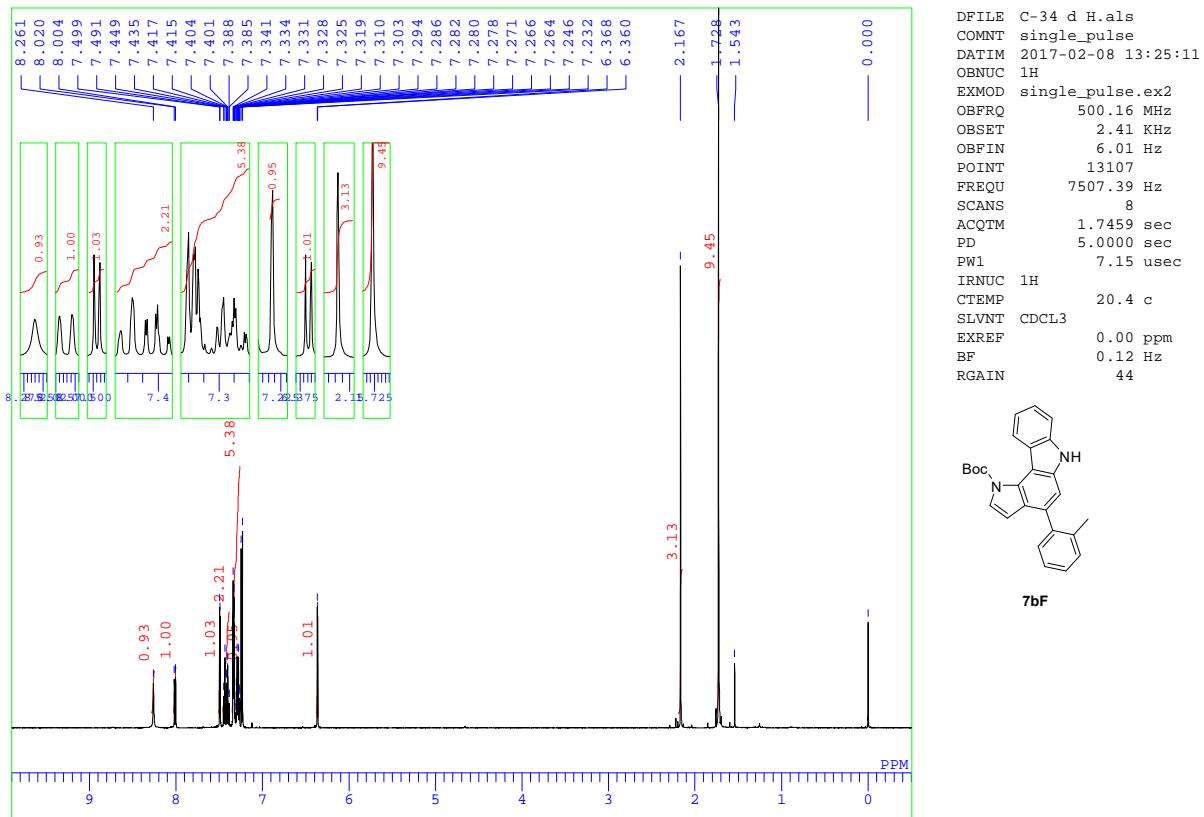
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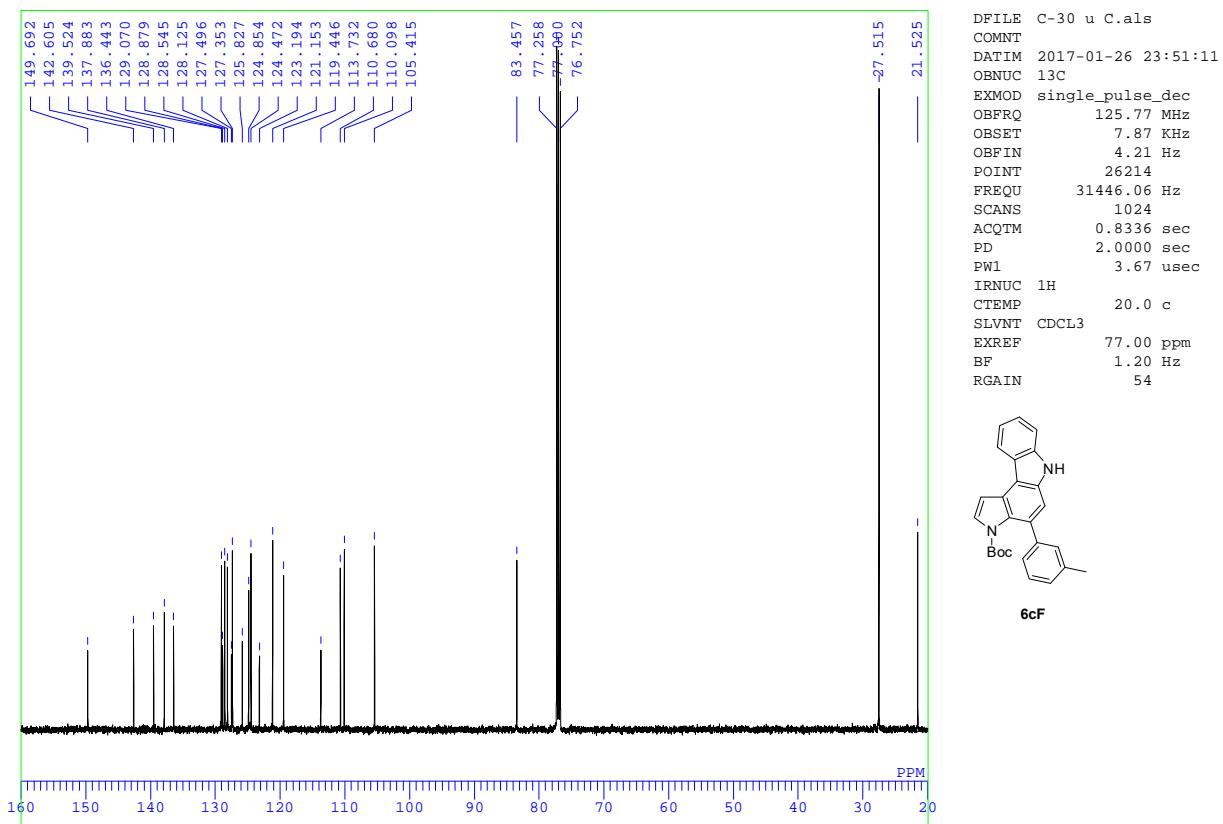
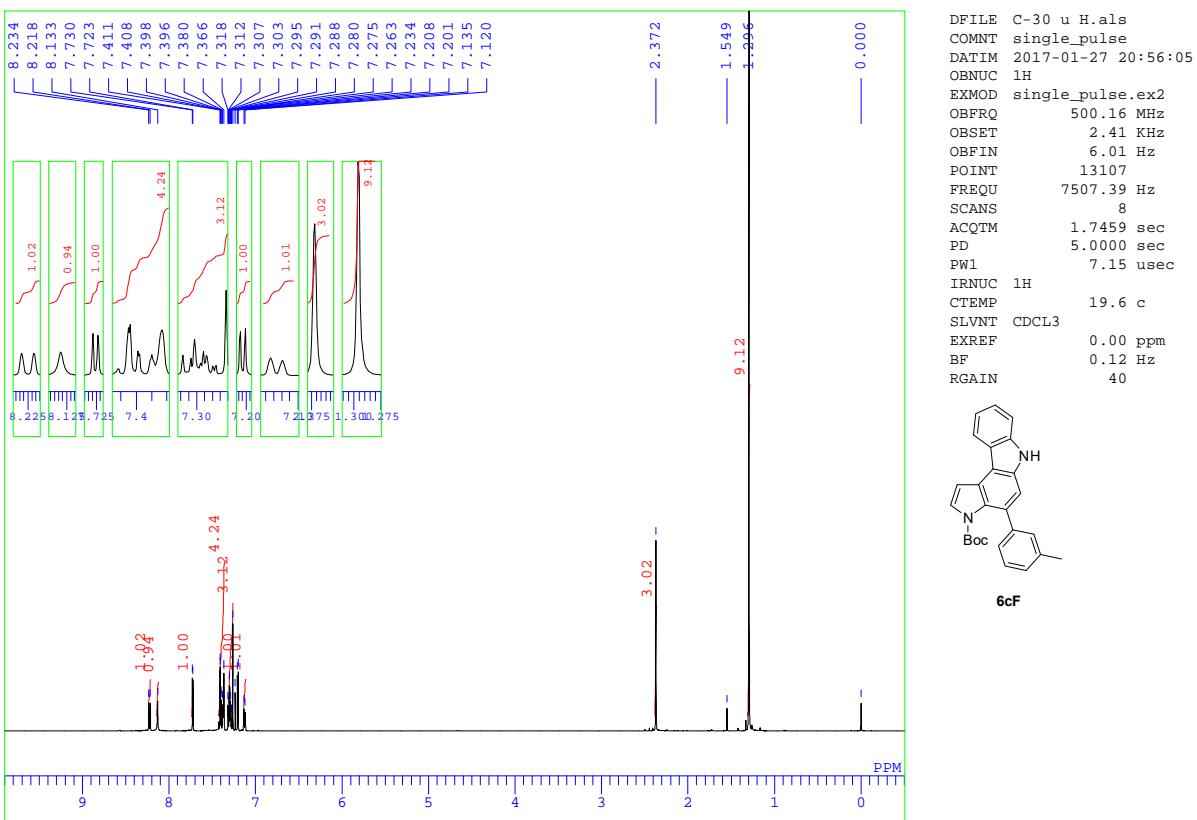
DFILE C-34 sita C 2 3-1.als
COMNT
DATIM 2017-02-03 14:26:05
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 32768
FREQU 31446.54 Hz
SCANS 1024
ACQTM 1.0420 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 70

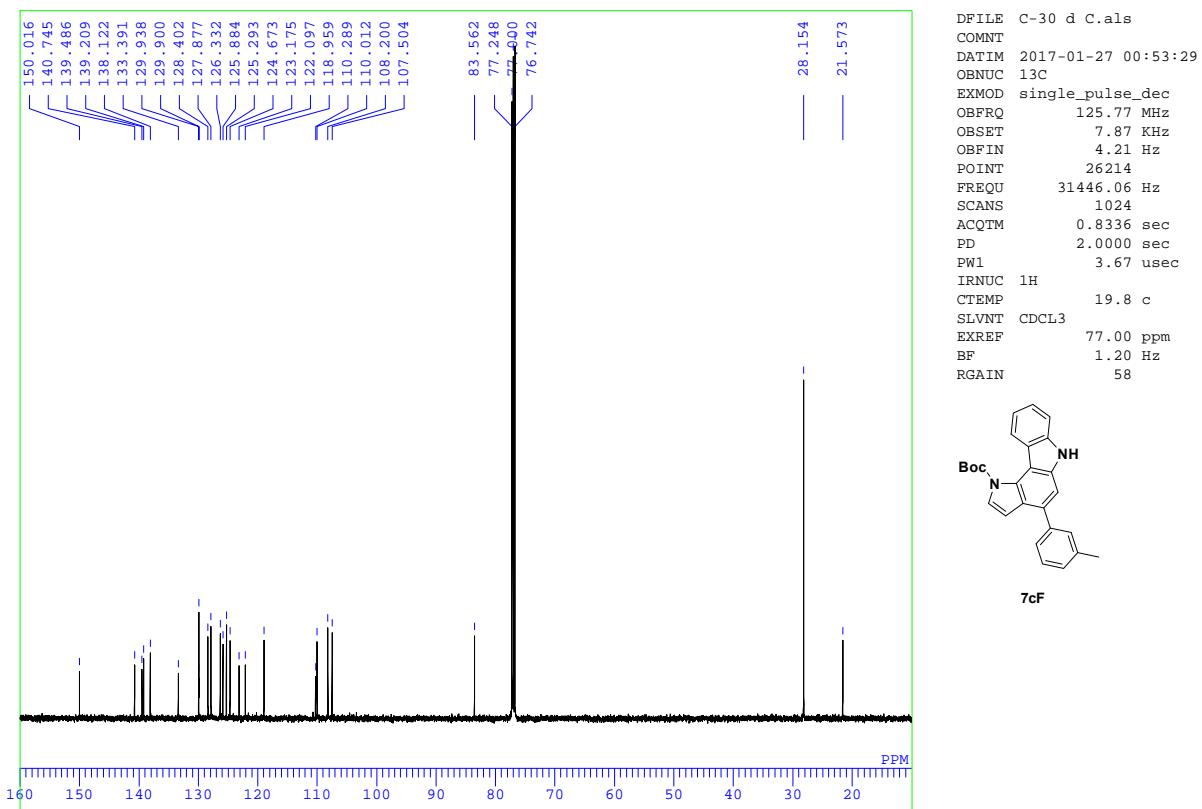
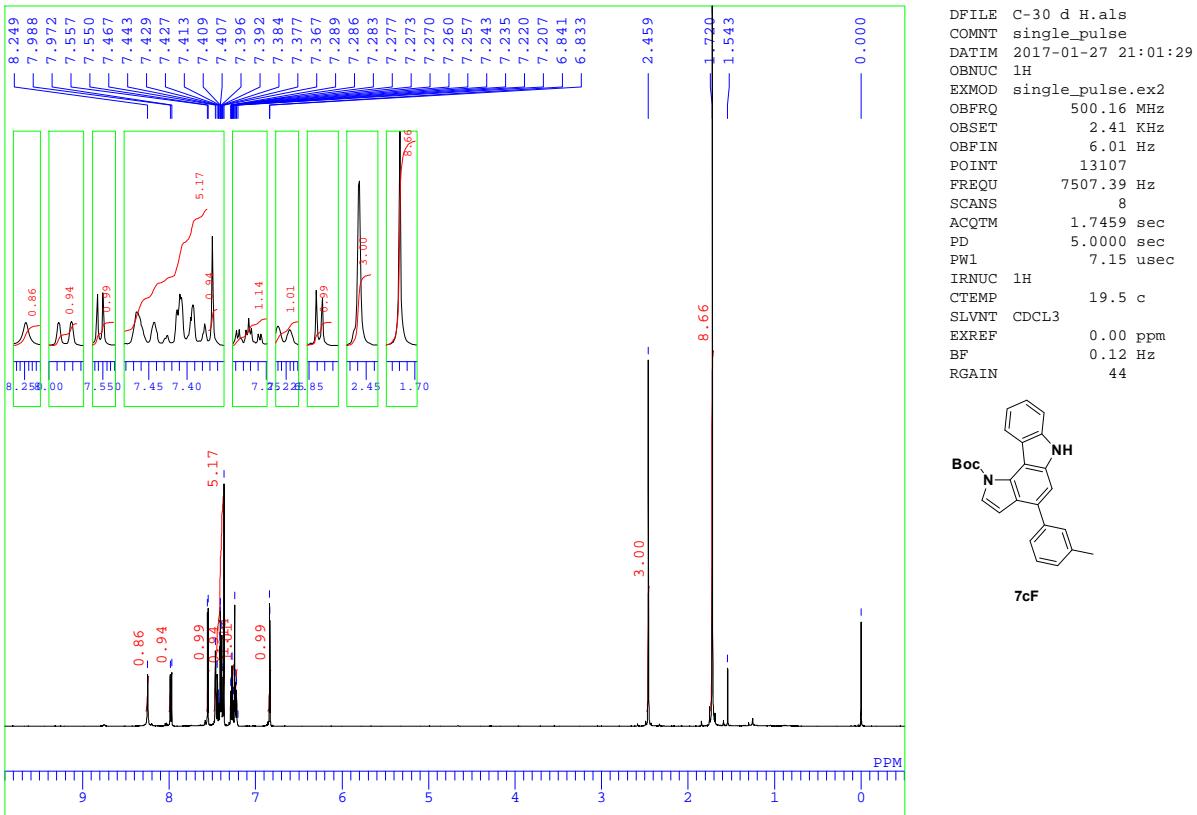
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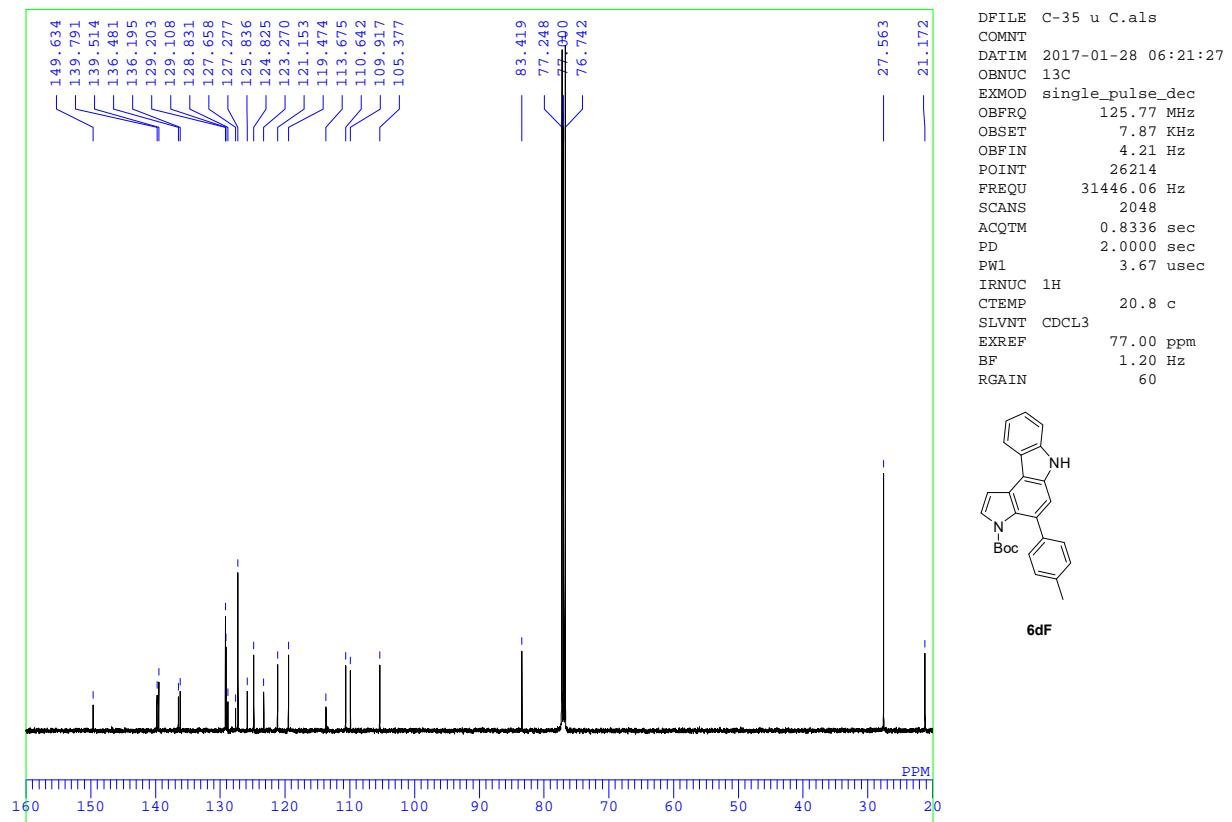
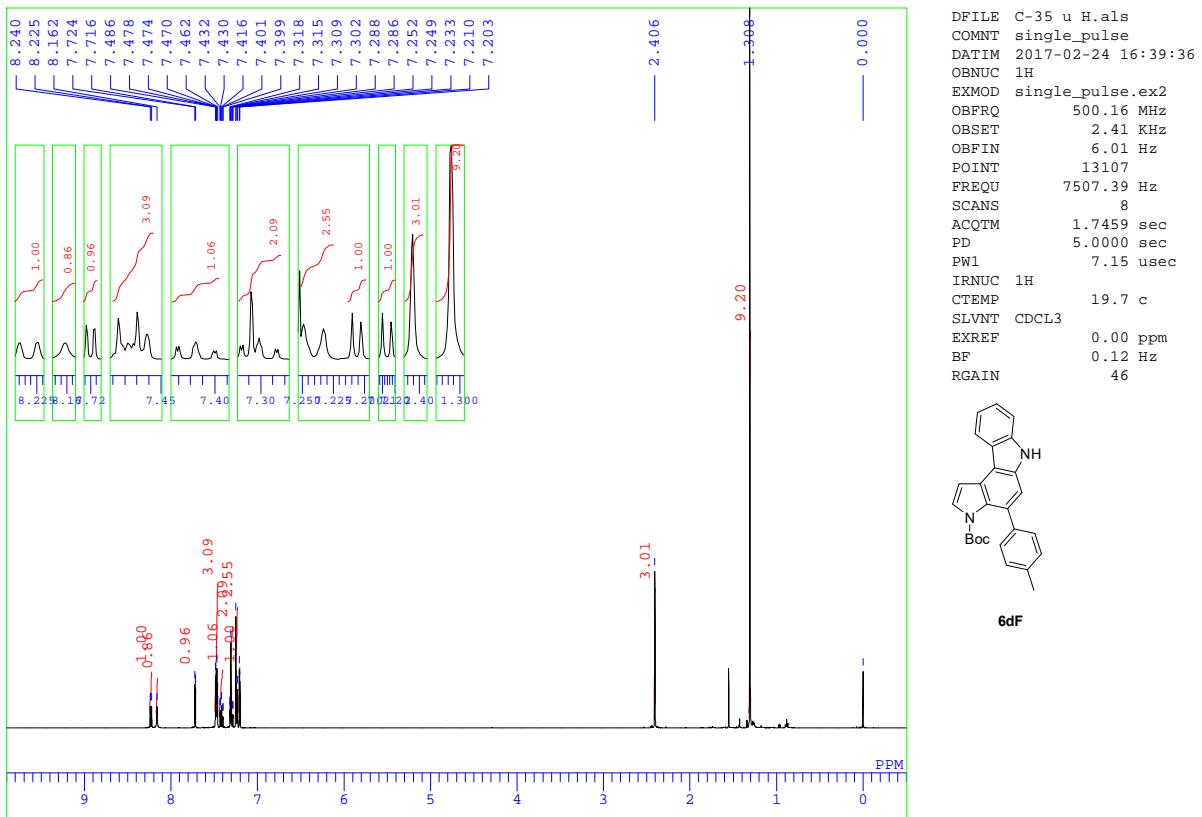


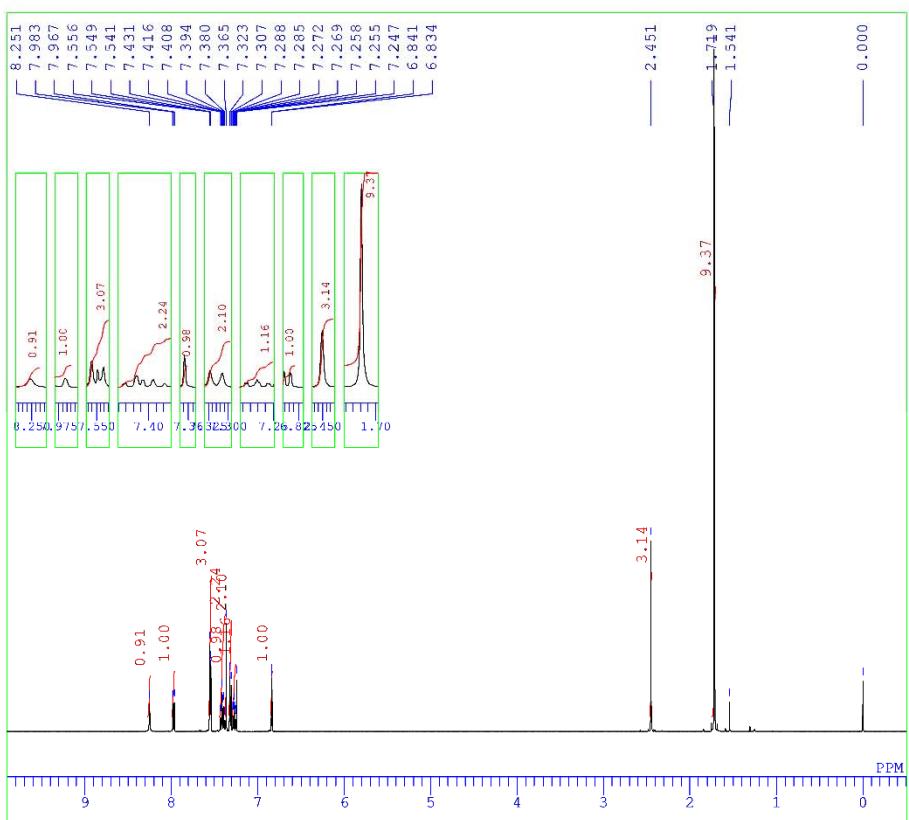
6bF







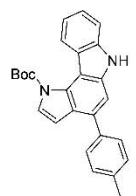
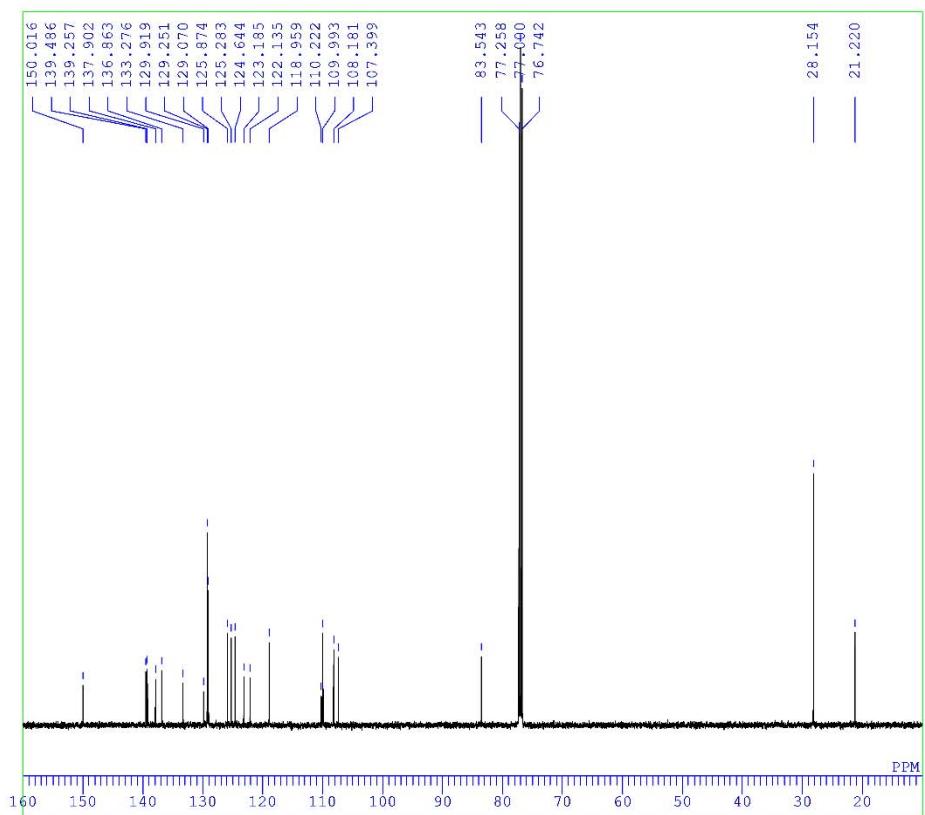
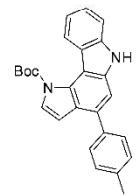


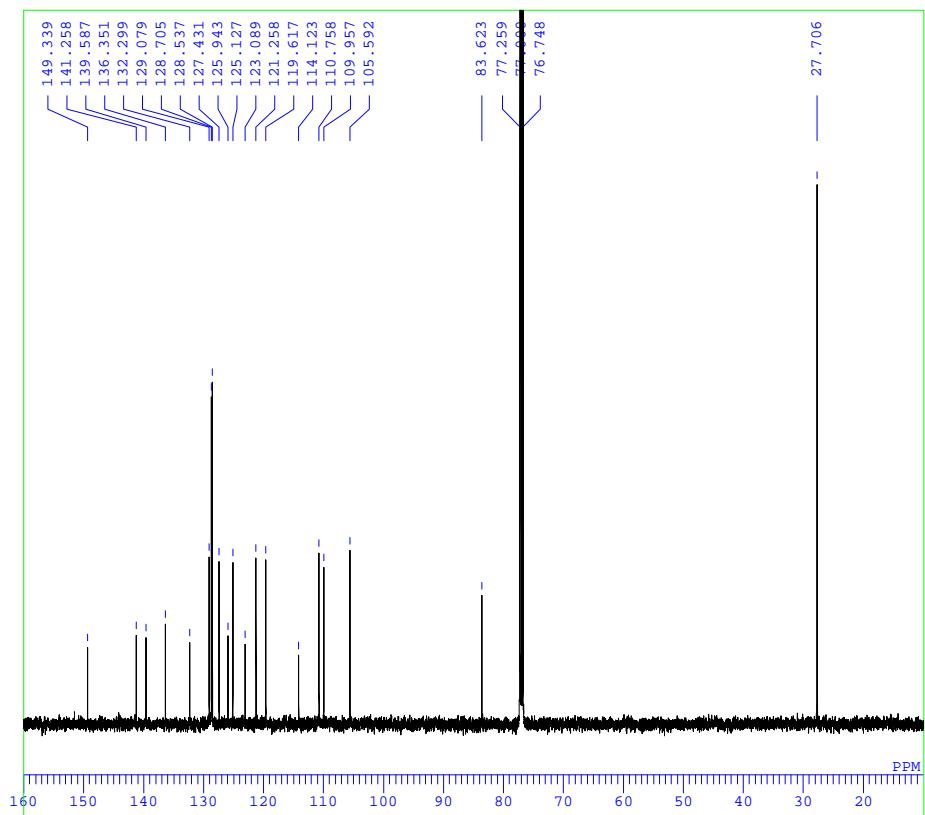
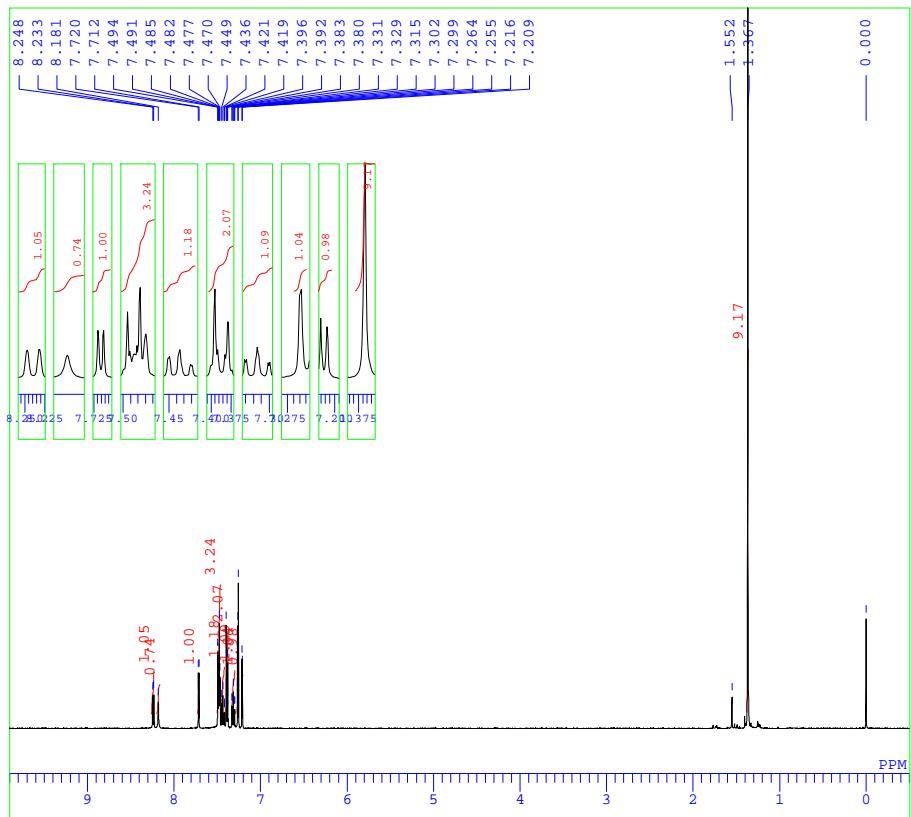


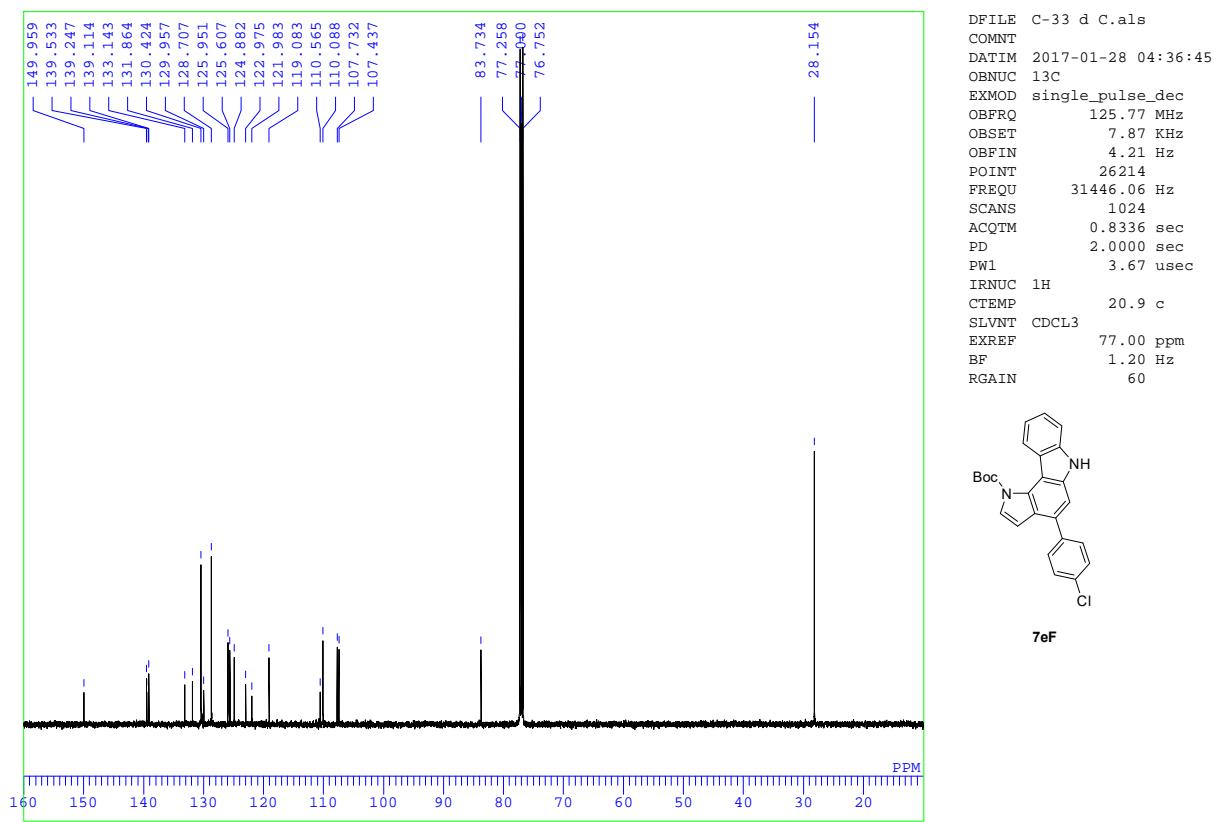
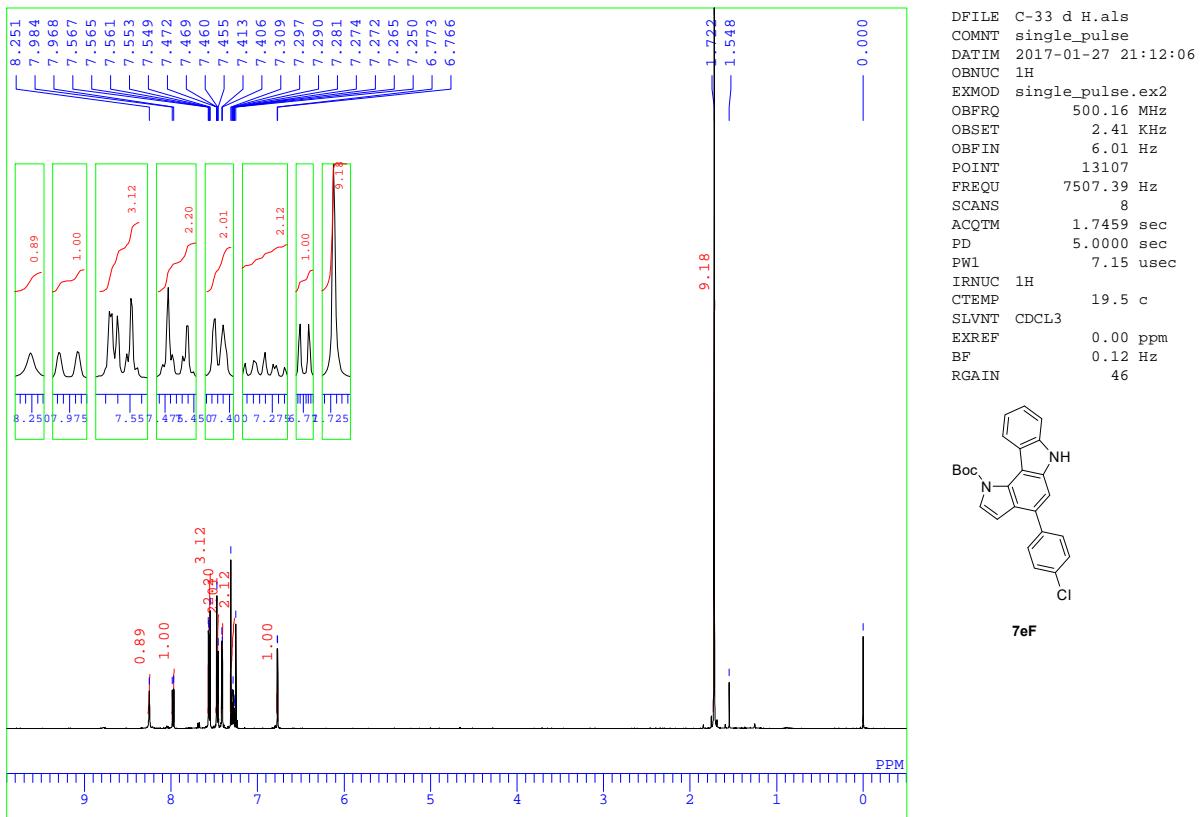
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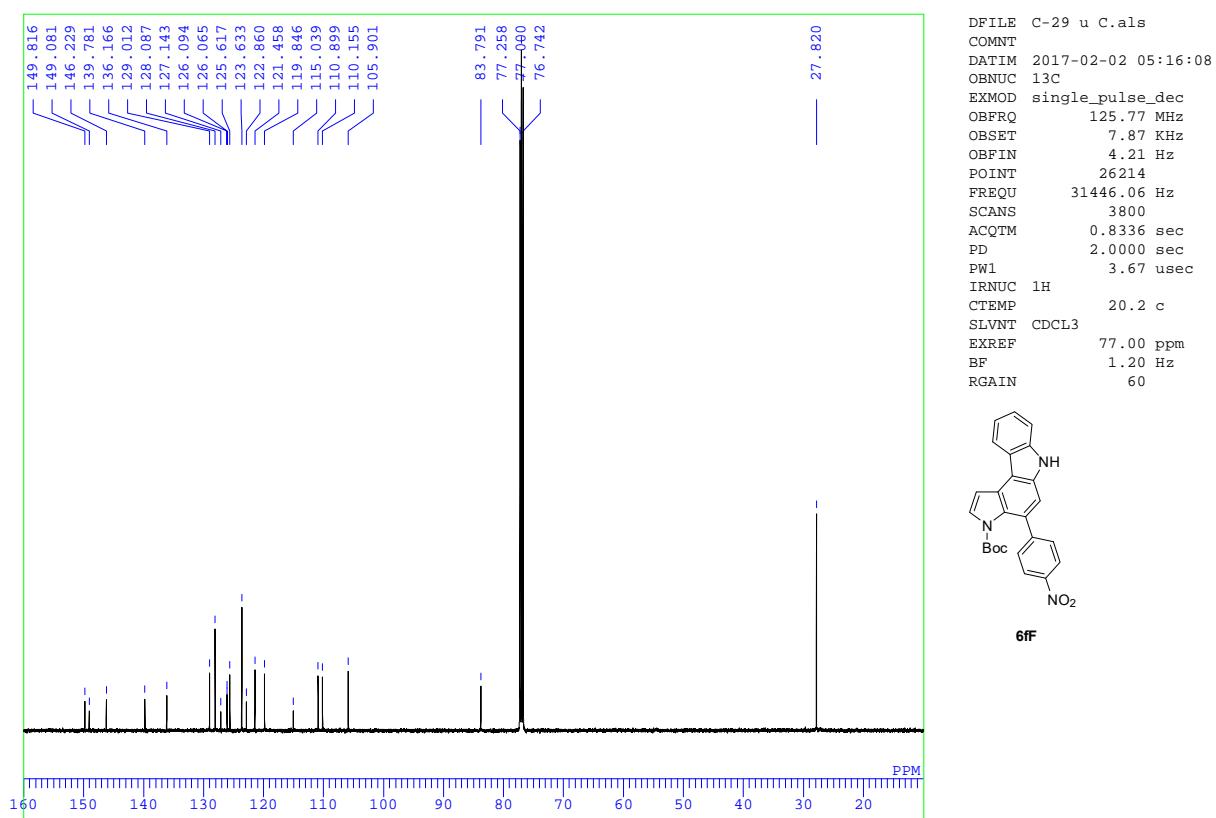
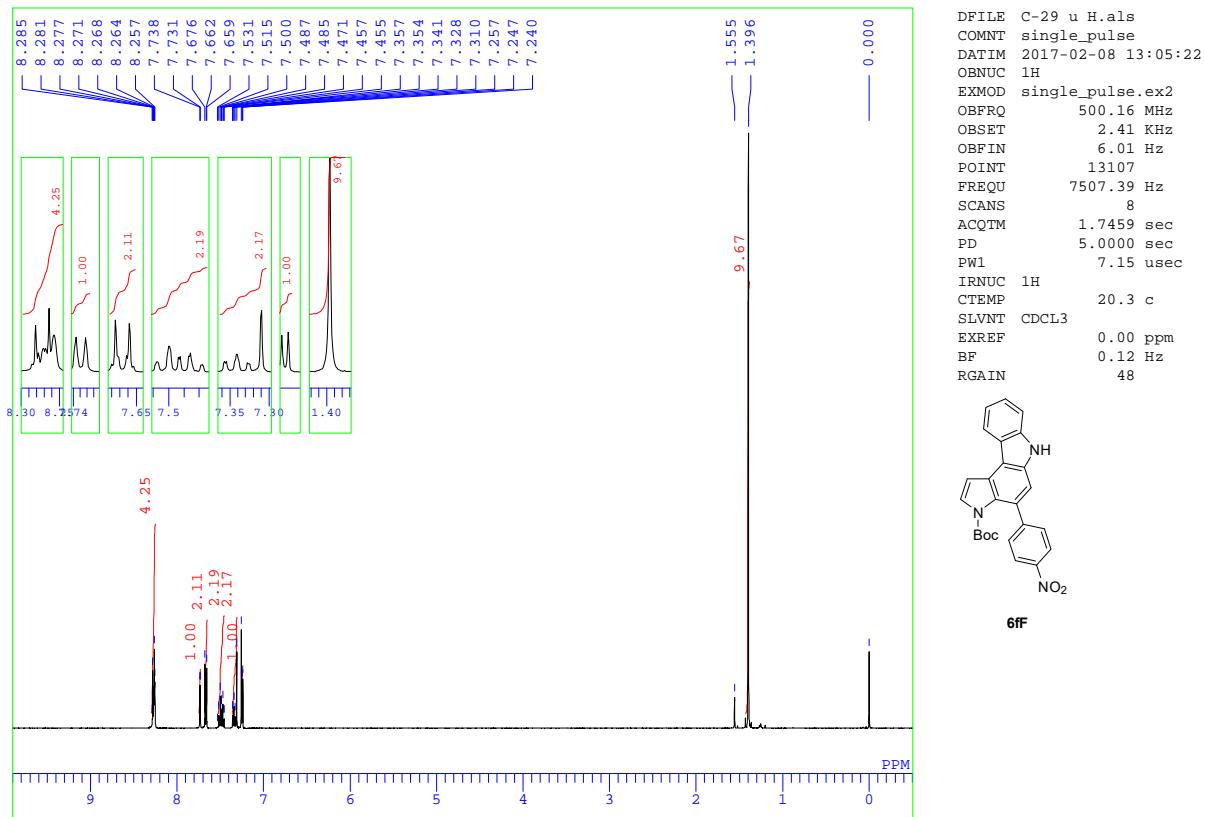
DFILE C-35 d H.als
COMNT single_pulse
DATIM 2017-01-27 21:23:20
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBPIN 6.01 Hz
POINT 1310/
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PWL 7.15 uscc
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

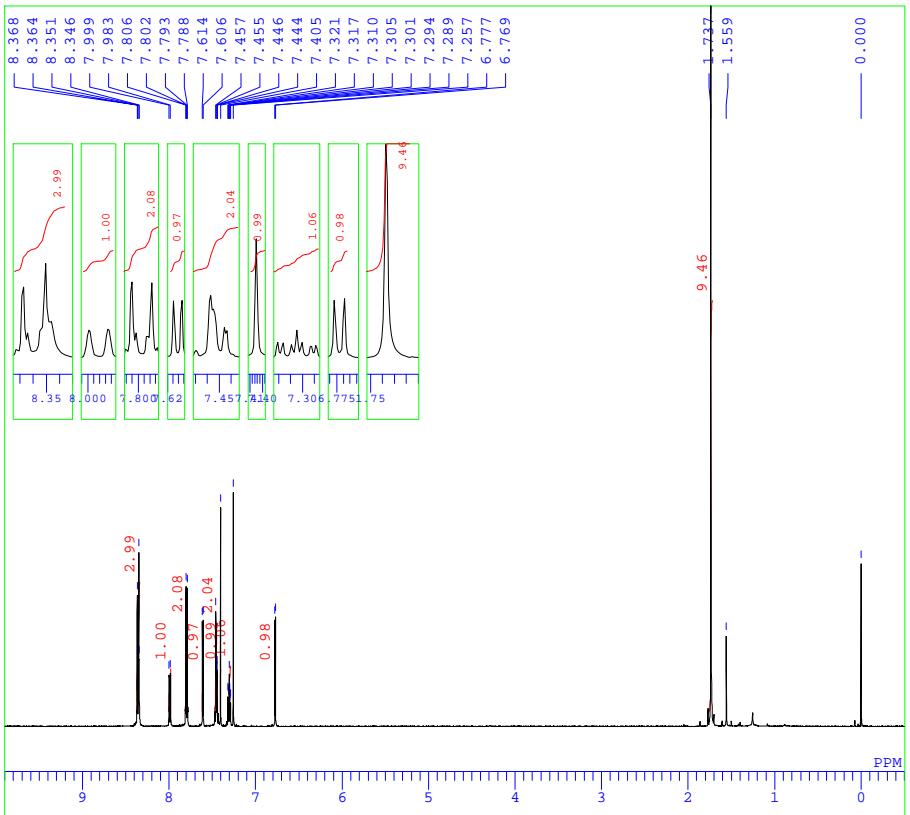
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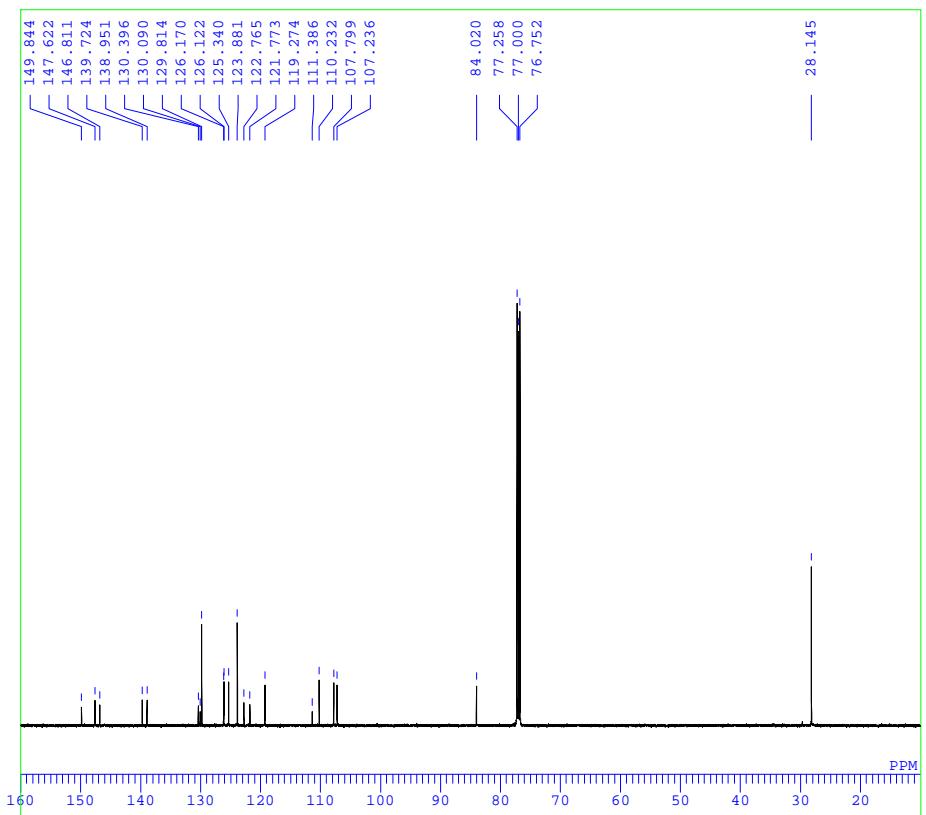
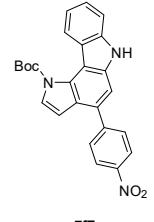




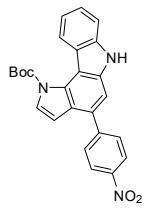


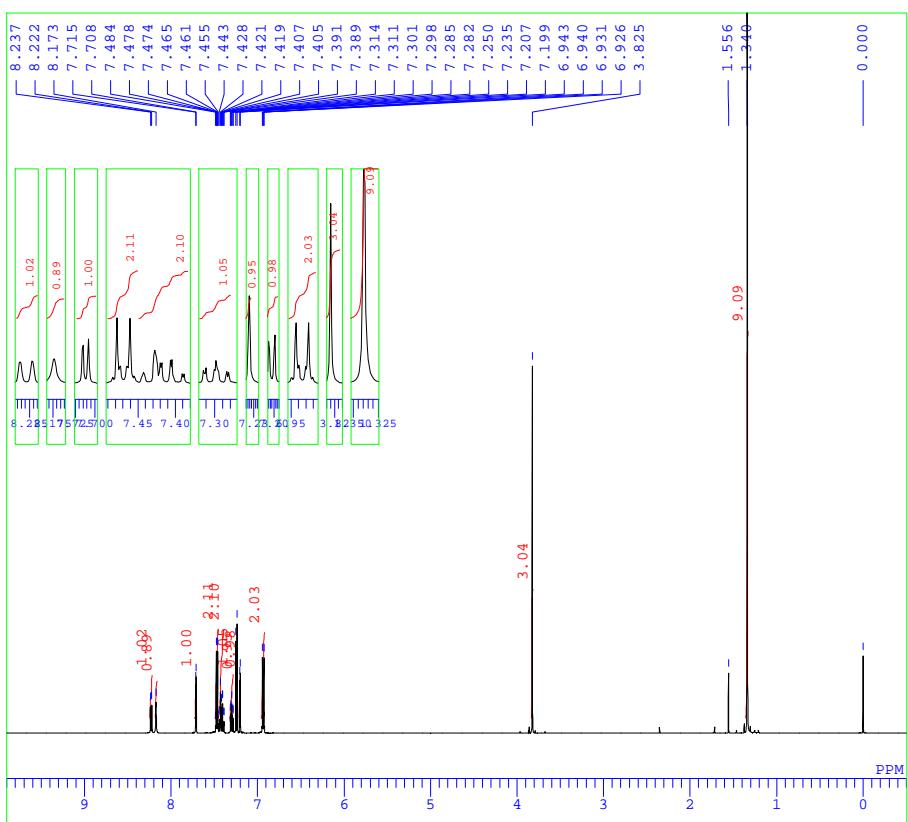


DFILE C-29 d H.als
COMNT single_pulse
DATIM 2017-02-08 13:11:43
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PWL 7.15 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL₃
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46



DFILE C-29 d C.als
COMNT
DATIM 2017-02-02 08:27:49
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 3800
ACQTM 0.8336 sec
PD 2.0000 sec
PWL 3.67 usec
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL₃
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

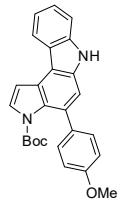




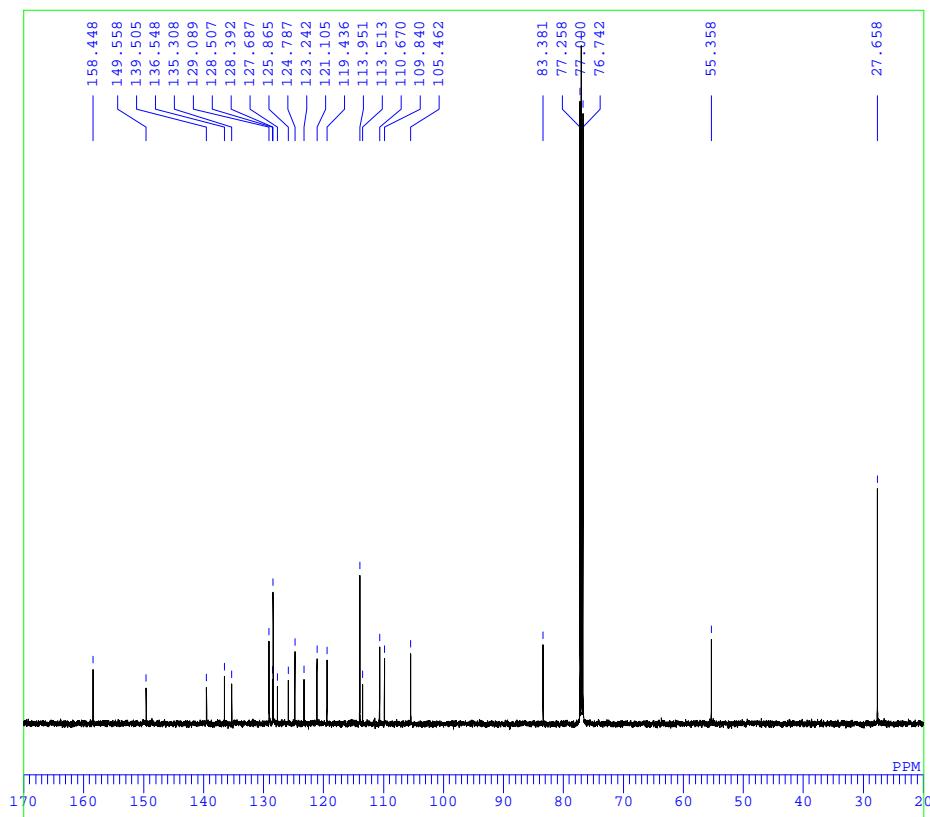
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DFILE C-32_u.H.als
COMNT single_pulse
DATIM 2017-01-27 21:07:15
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

```



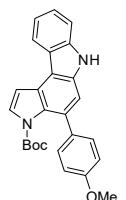
6gF



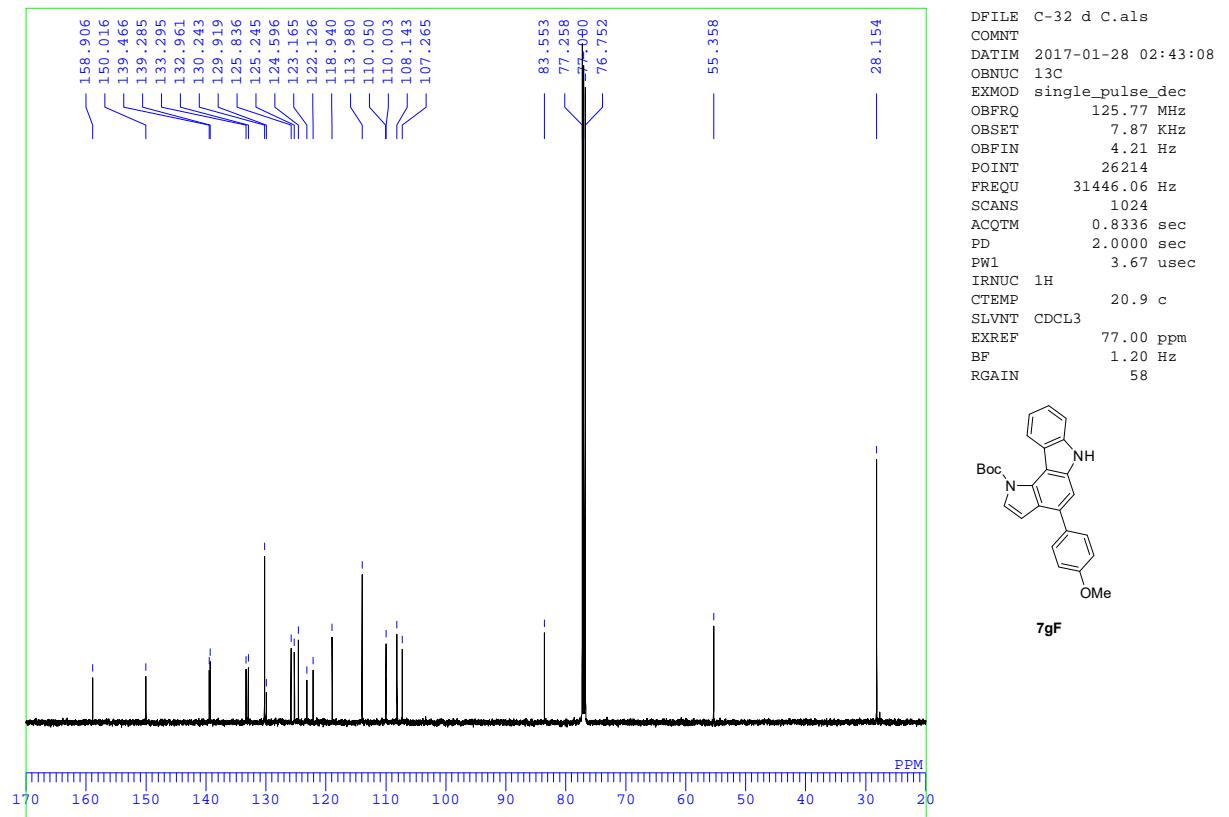
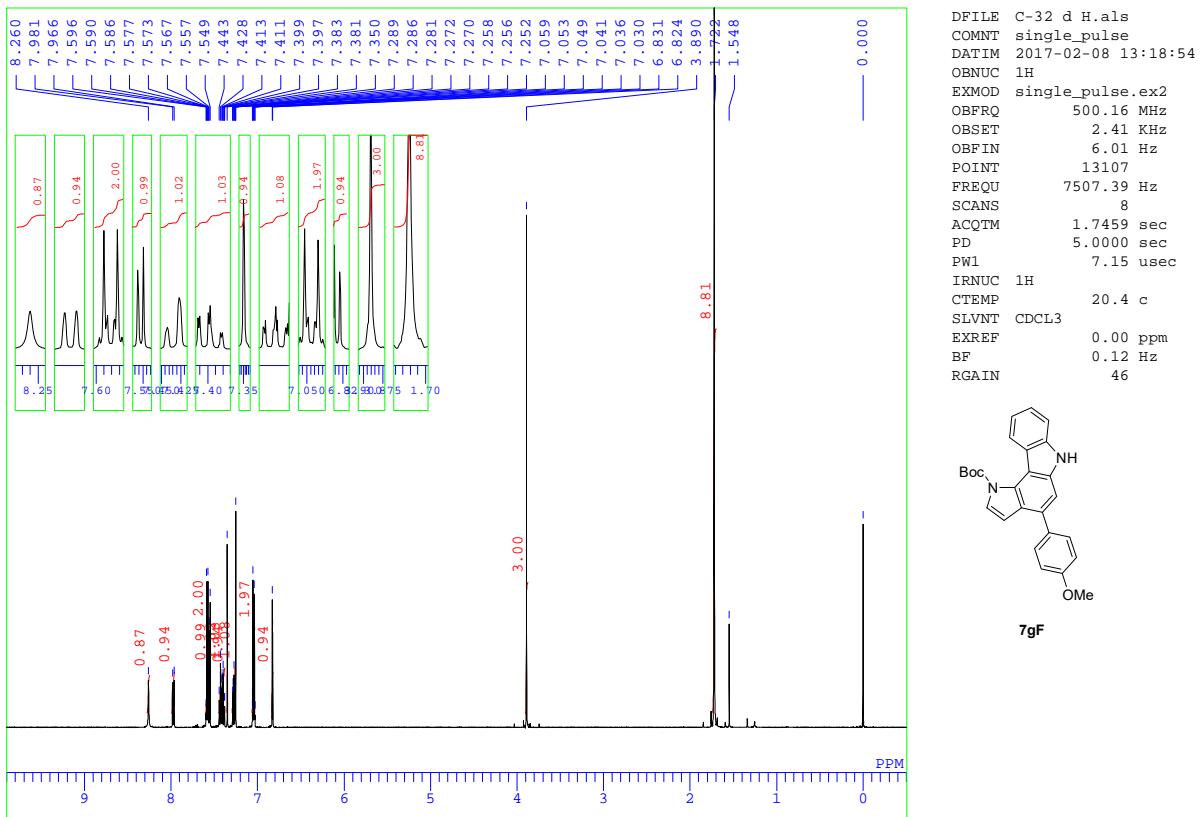
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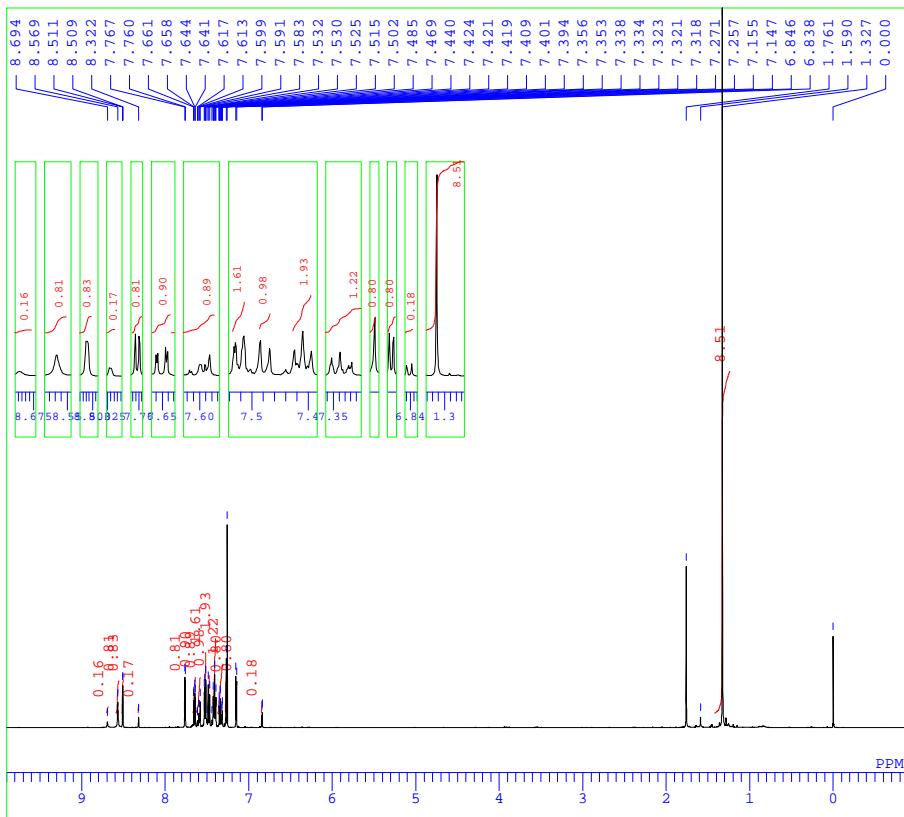
DFILE C-32 u C.als
COMNT
DATIM 2017-01-27 01:50:51
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58

```



6qF

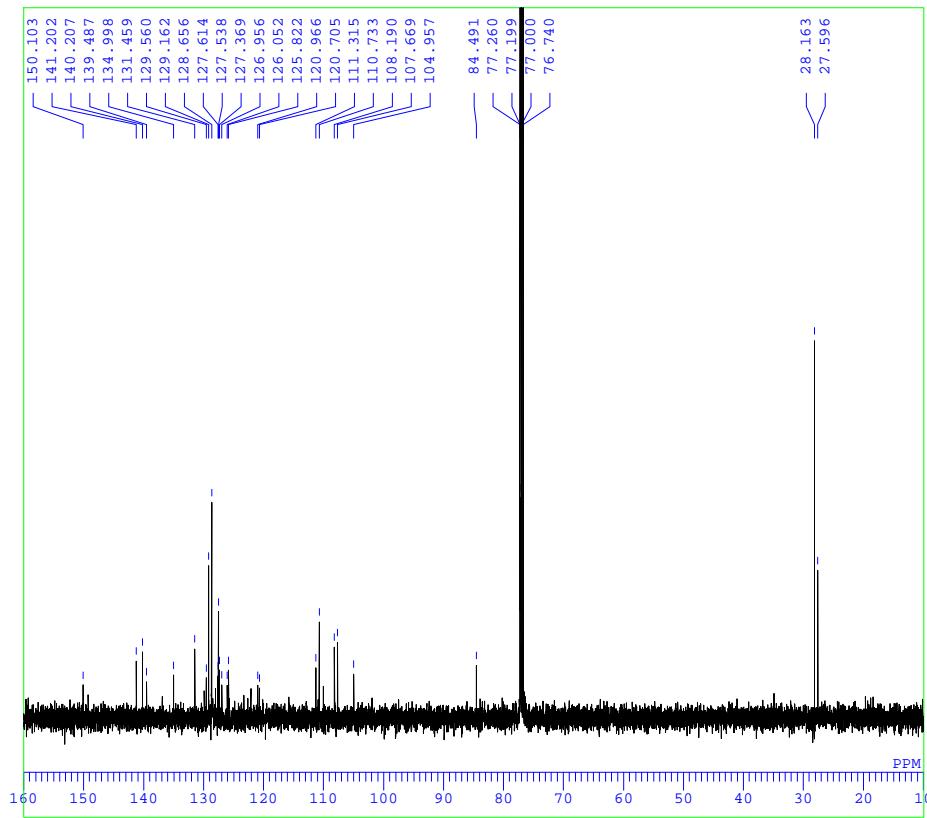
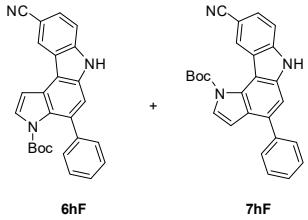




```

DFILE B-41 H.als
COMNT single_pulse
DATIM 2016-03-11 19:56:47
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTIM 1.7459 sec
PD 5.0000 sec
PW1 6.82 usec
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46

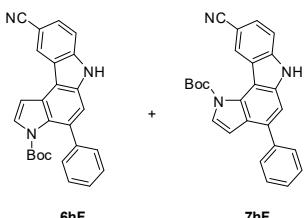
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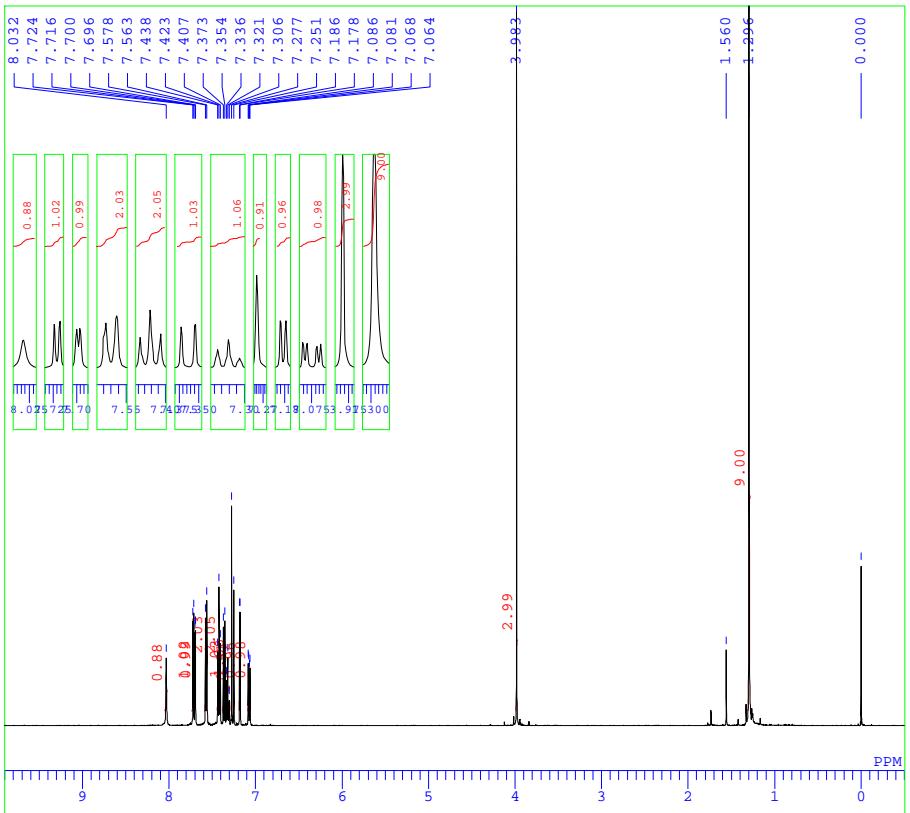


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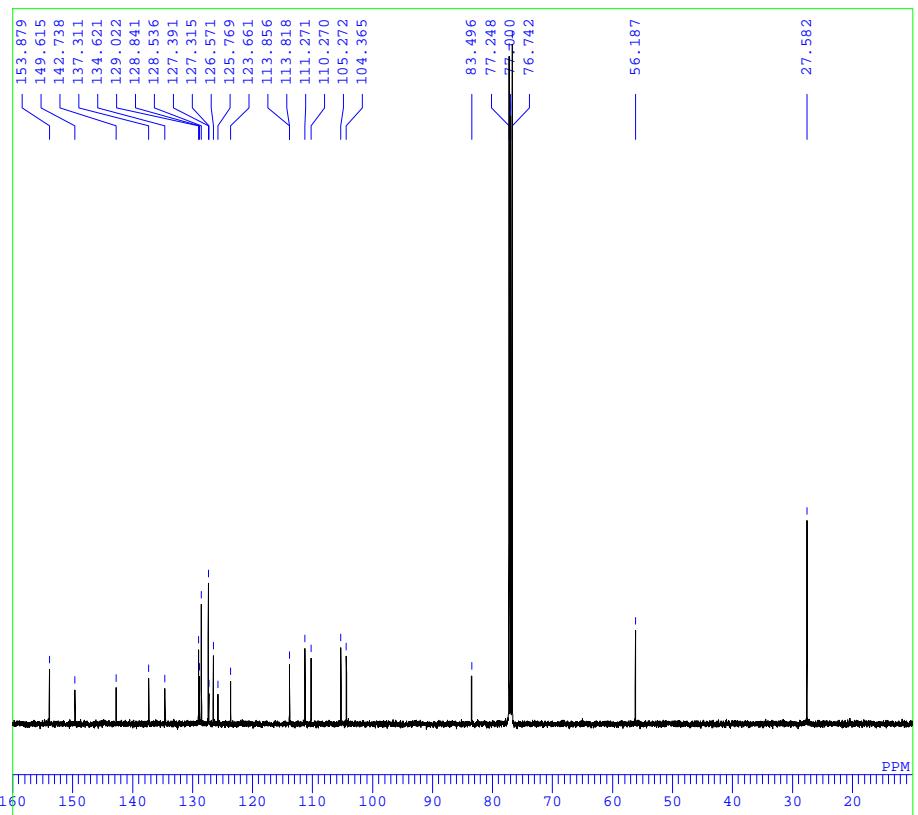
DFILE B41 C.als
COMNT
DATIM 2018-05-23 15:23:31
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 16384
FREQU 31565.66 Hz
SCANS 1926
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

```

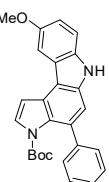




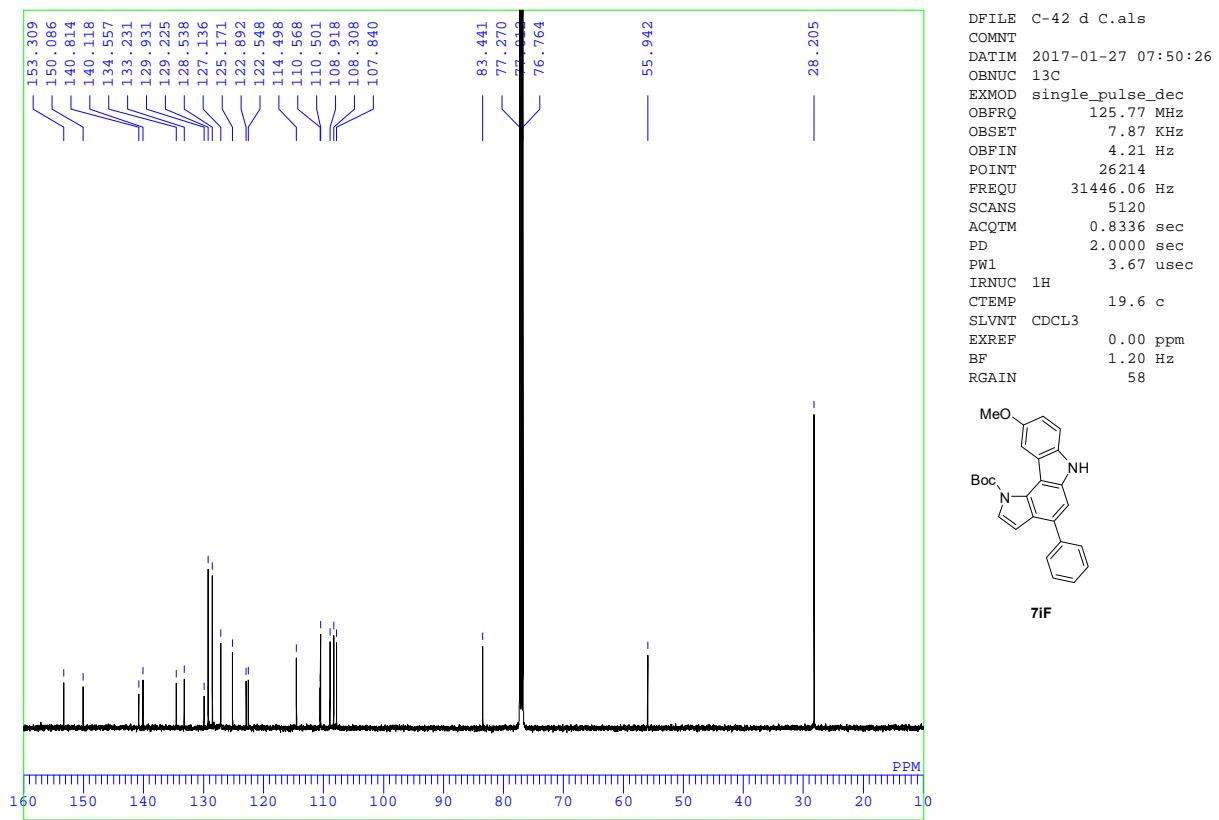
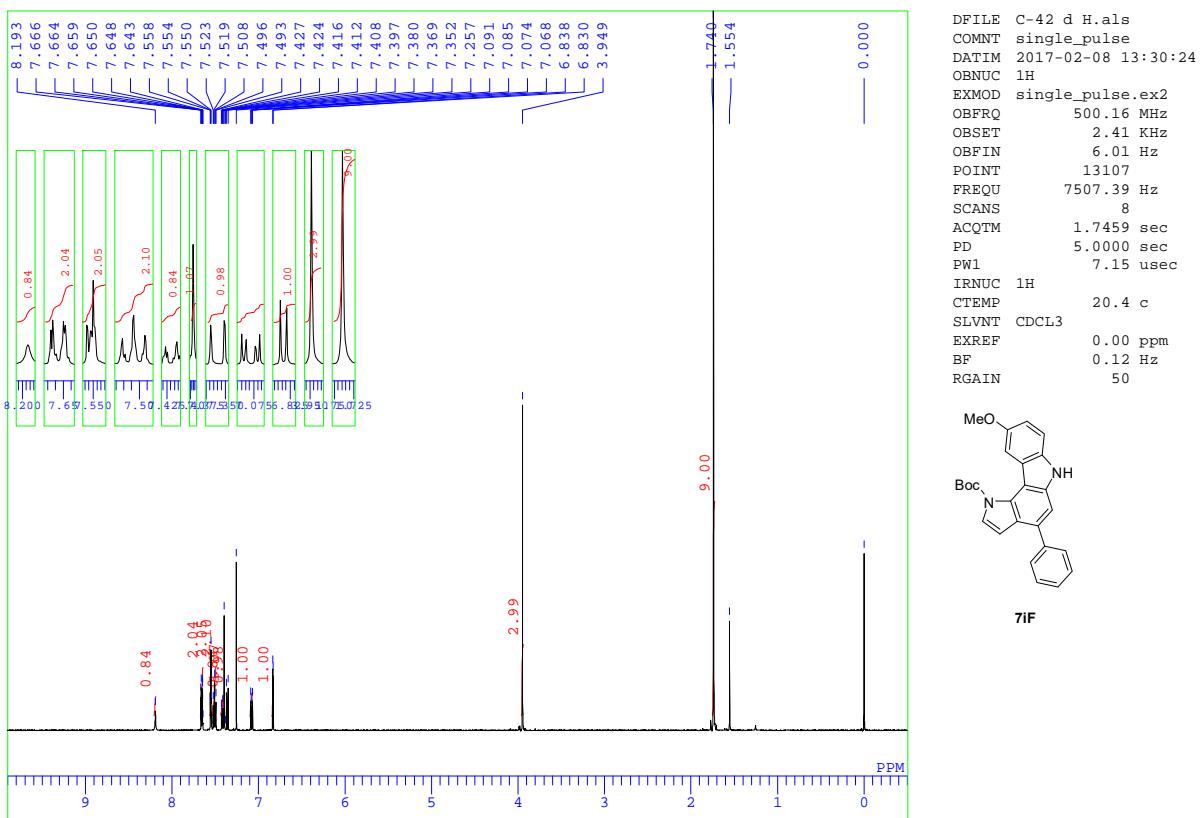
DFILE C-42 u H.als
COMNT single_pulse
DATIM 2017-01-27 21:27:46
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 kHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 7.15 usec
IRNUC 1H
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44

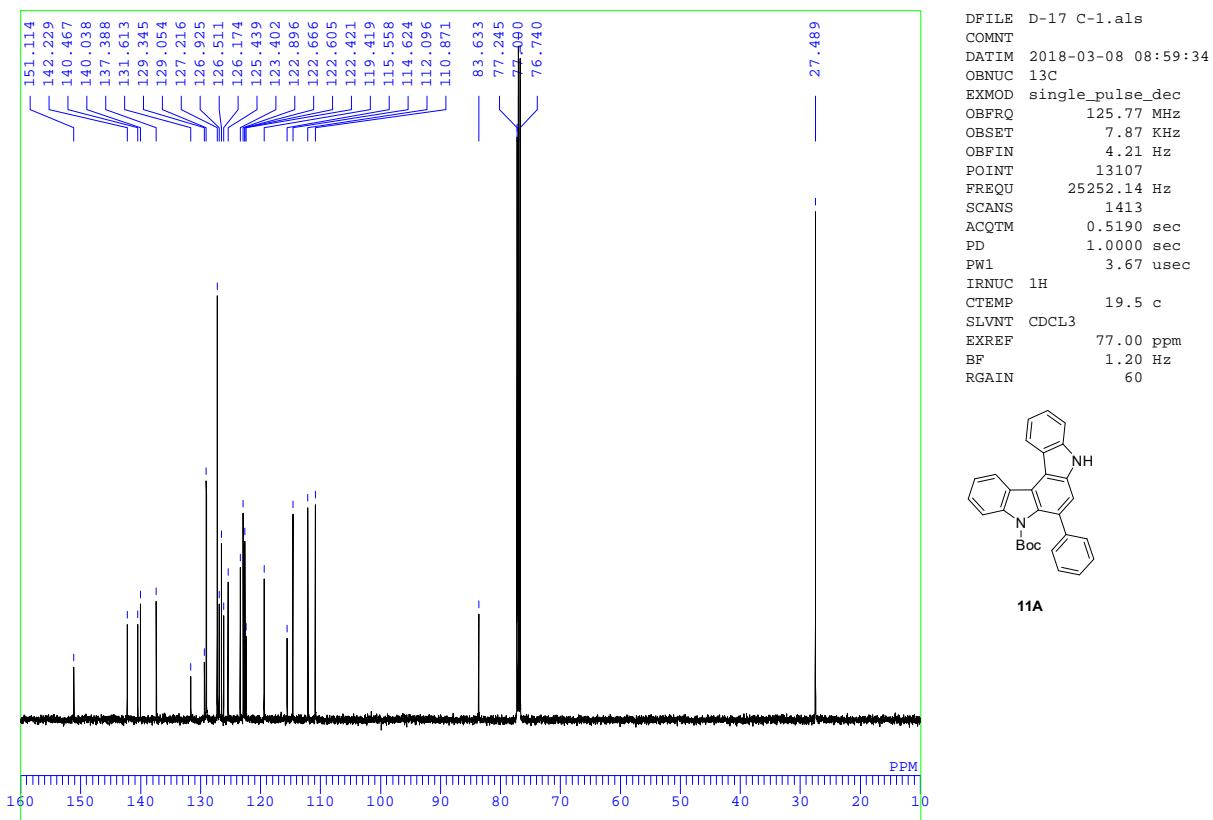
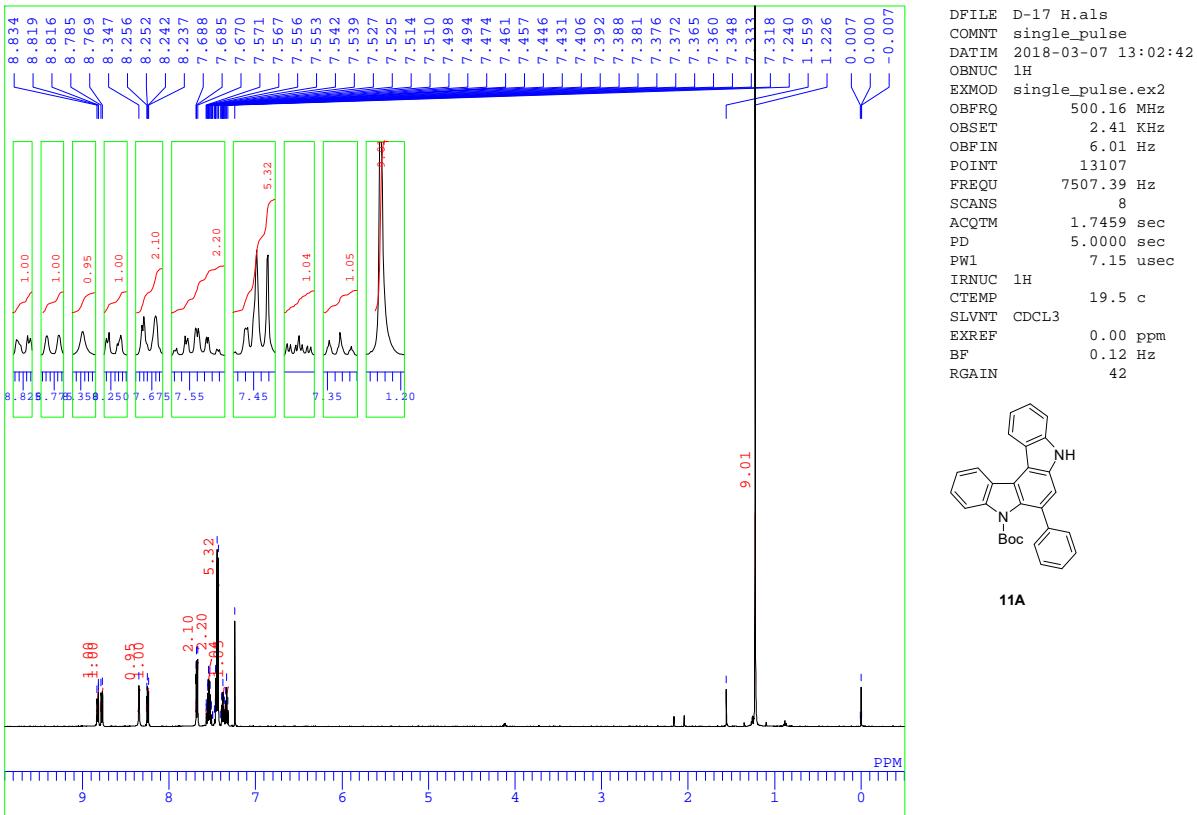


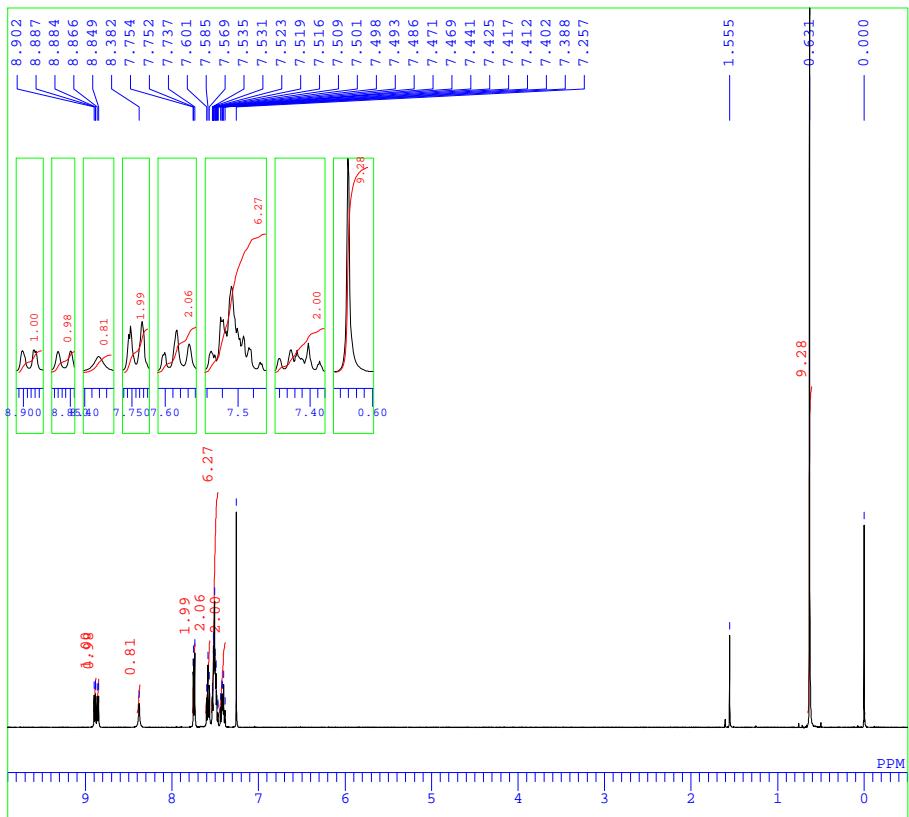
DFILE C-42 u C.als
COMNT
DATIM 2017-01-28 08:11:48
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 kHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



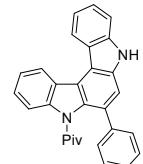
6iF



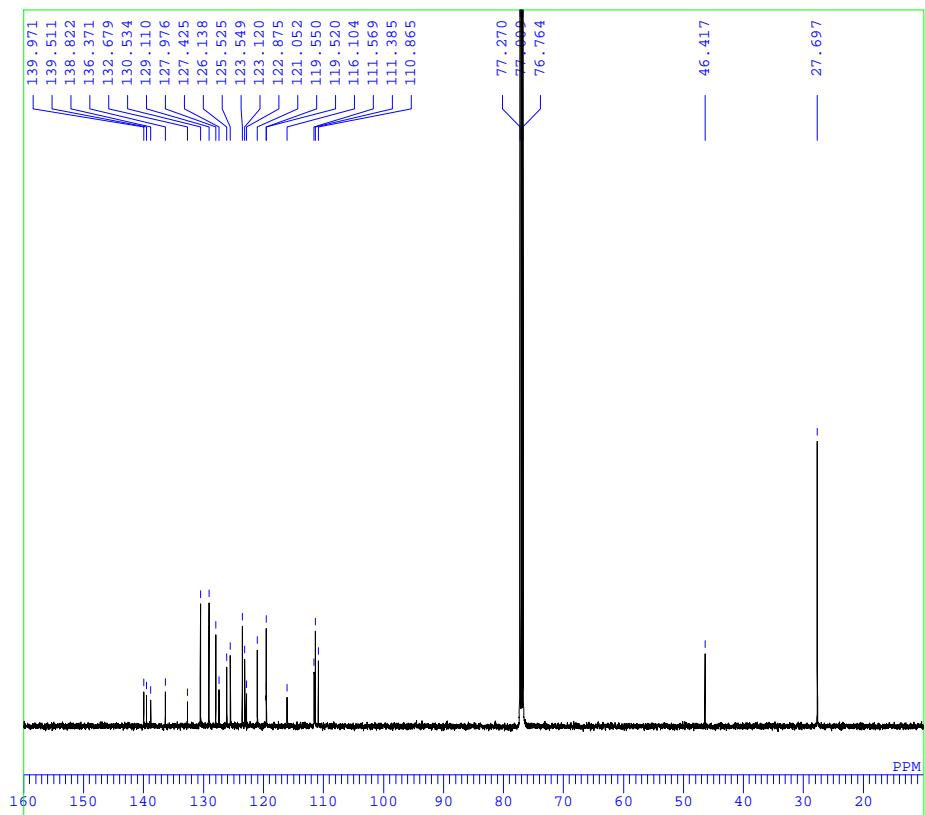




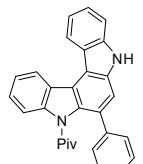
DFILE C-126 H-1.als
 COMNT single_pulse
 DATIM 2018-03-07 22:40:02
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFREQ 500.16 MHz
 OBSET 2.41 kHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 7.15 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



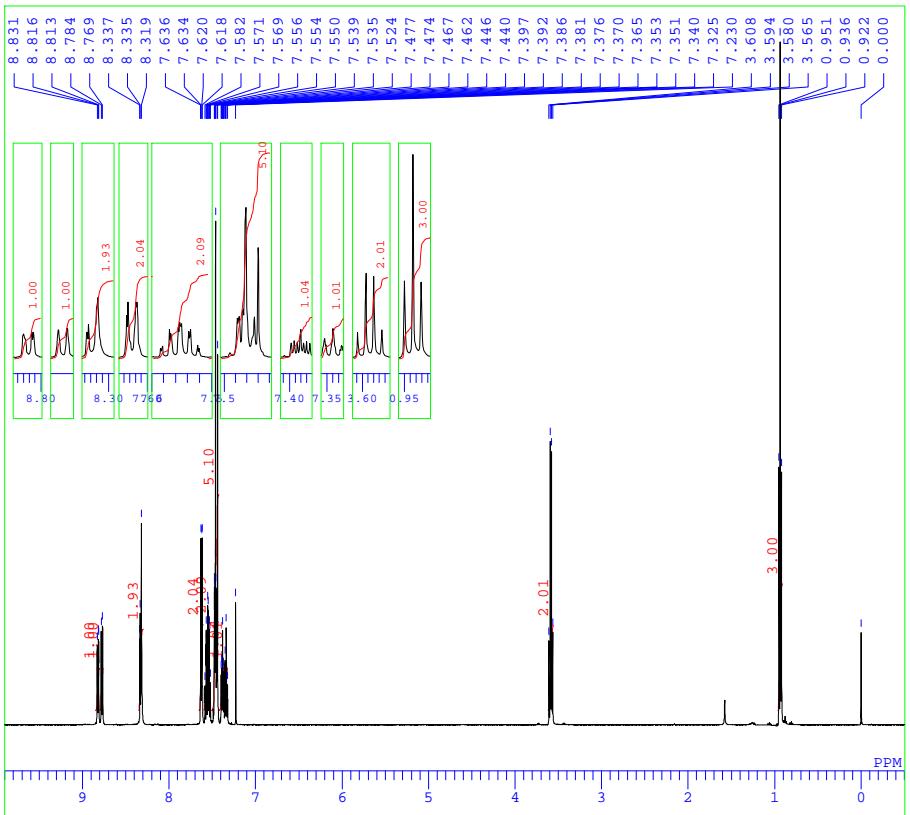
11B



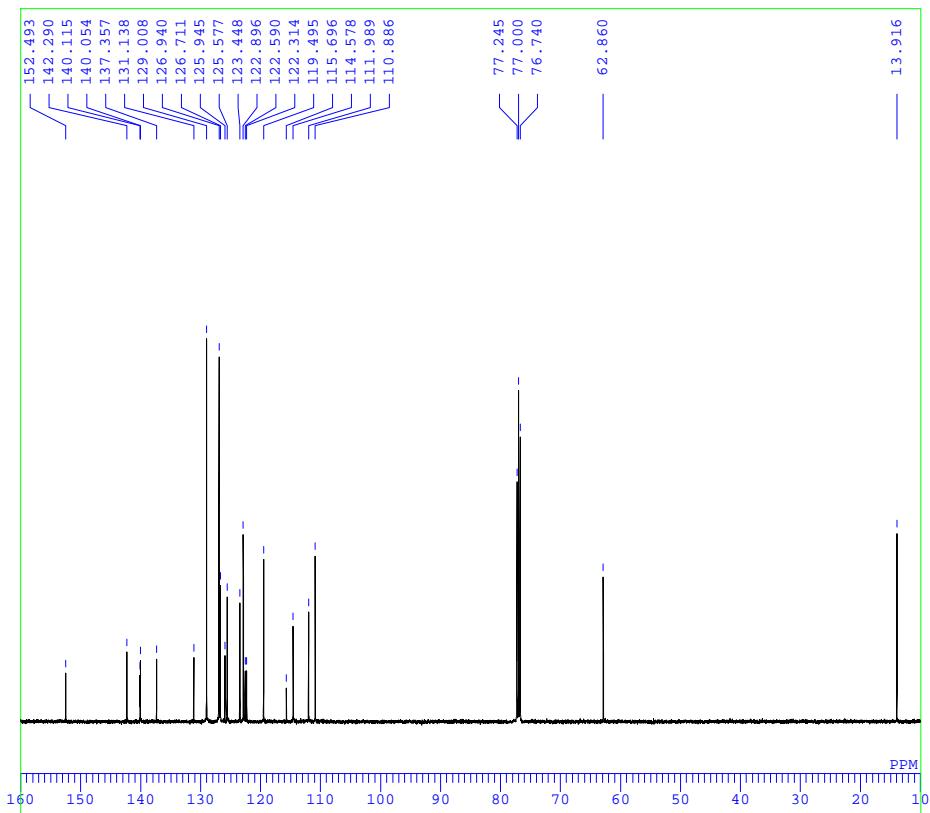
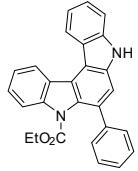
DFILE C-126 C-1.als
 COMNT
 DATIM 2018-03-08 07:14:32
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFREQ 125.77 MHz
 OBSET 7.87 kHz
 OBFIN 4.21 Hz
 POINT 13107
 FREQU 25252.14 Hz
 SCANS 7168
 ACQTM 0.5190 sec
 PD 1.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 1.20 Hz
 RGAIN 60



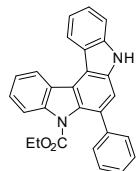
11B

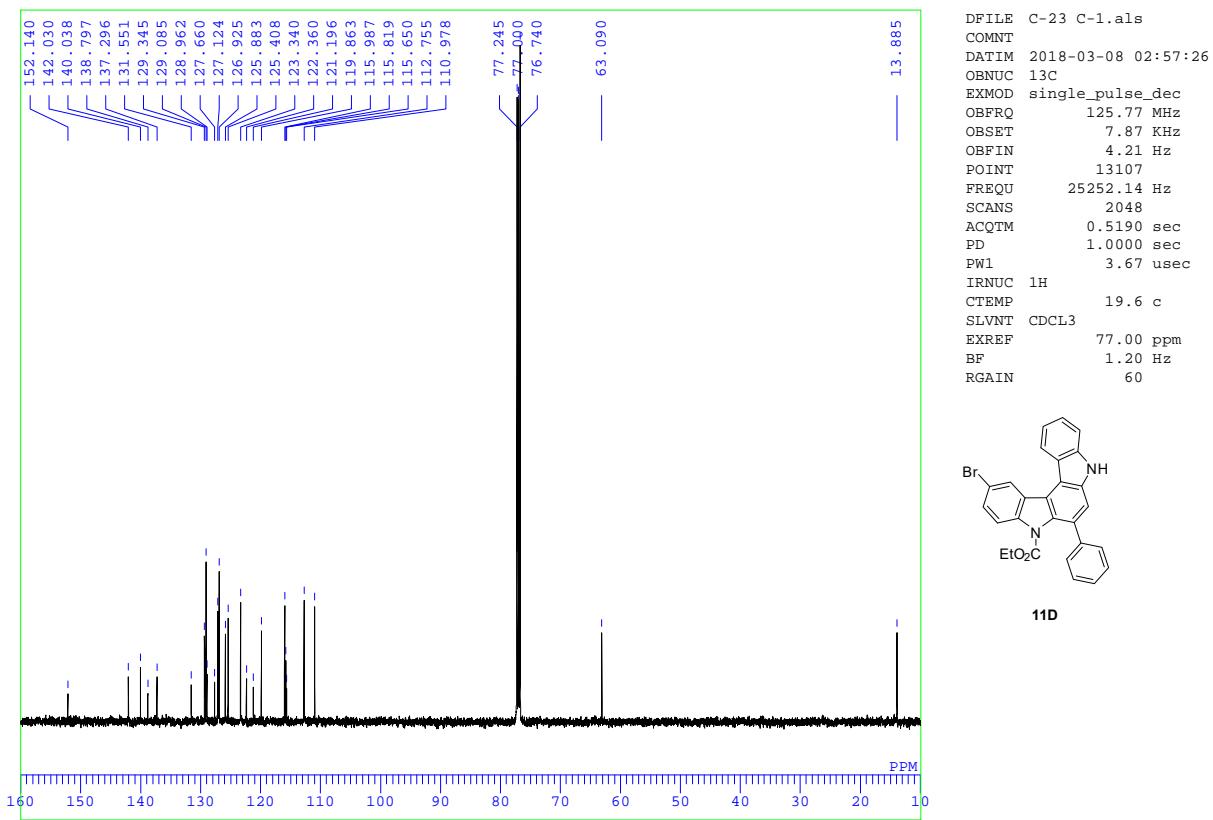
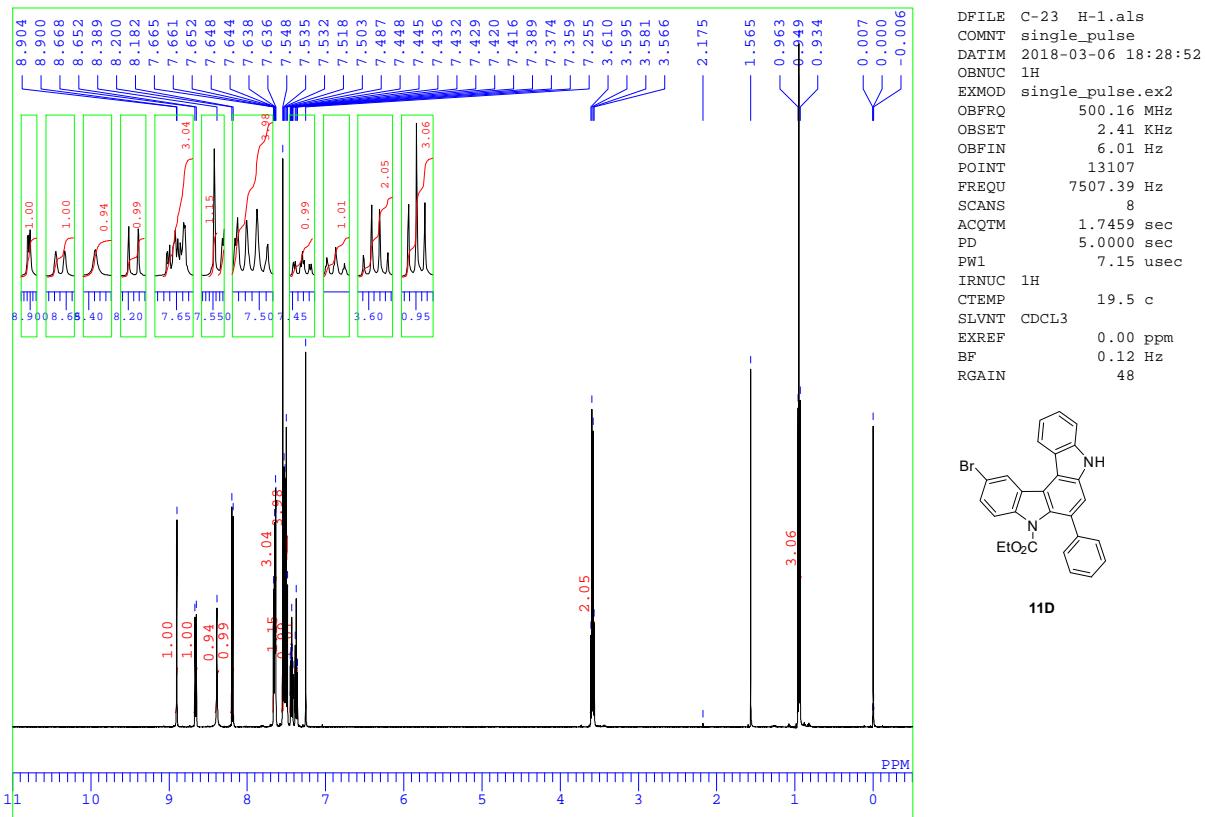


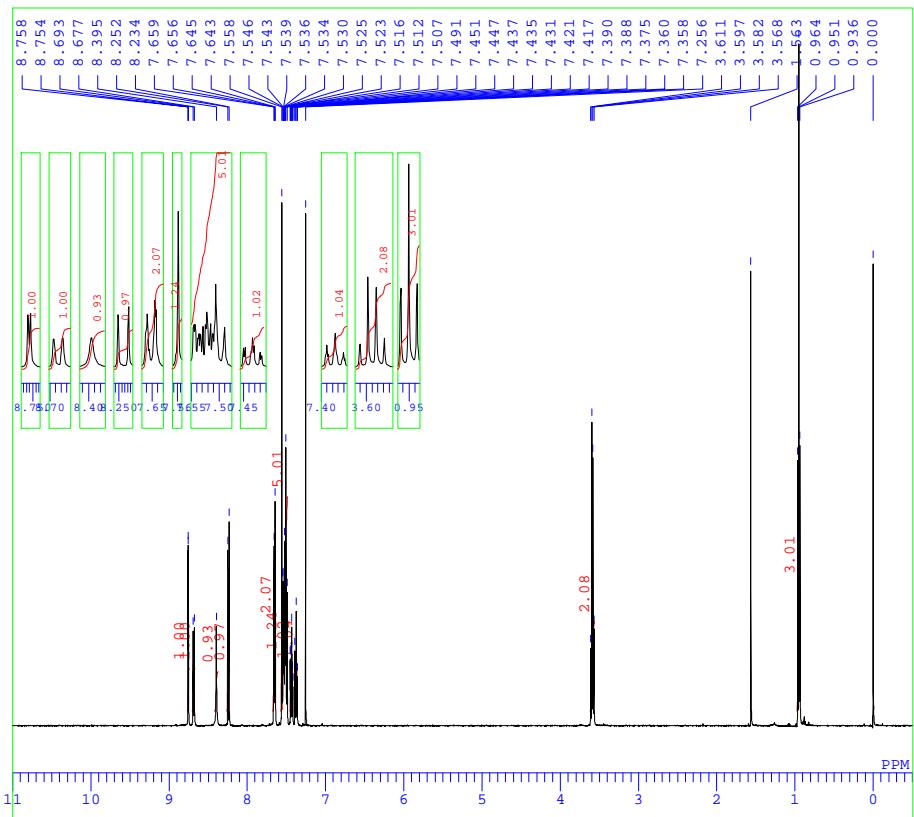
DFILE B-154 H-1.als
 COMNT single_pulse
 DATIM 2018-03-06 18:20:48
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRQ 500.16 MHz
 OBSET 2.41 kHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 7.15 usec
 IRNUC 1H
 CTEMP 19.4 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 40



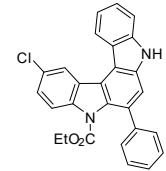
DFILE B-154 C-1.als
 COMNT
 DATIM 2018-03-07 23:55:47
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 kHz
 OBFIN 4.21 Hz
 POINT 13107
 FREQU 25252.14 Hz
 SCANS 2048
 ACQTM 0.5190 sec
 PD 1.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 19.8 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



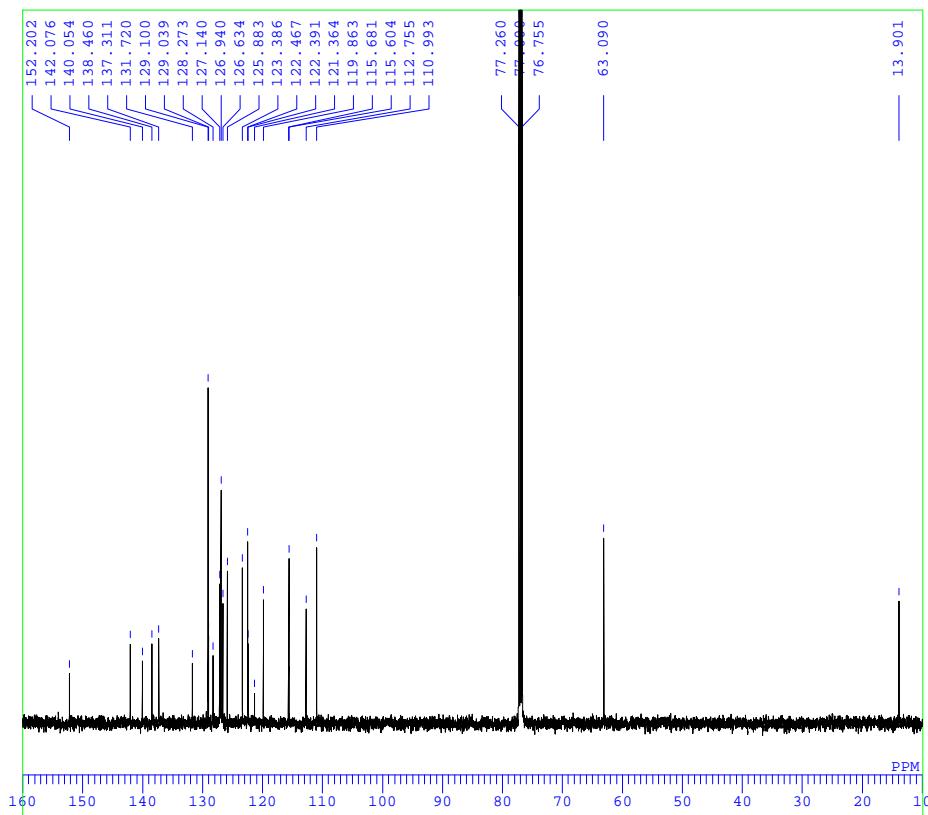




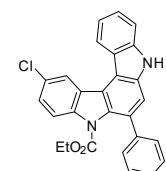
DFILE C-24 H-1.als
 COMNT single_pulse
 DATIM 2018-03-06 18:32:48
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRQ 500.16 MHz
 OBSET 2.41 kHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 7.15 usec
 IRNUC 1H
 CTEMP 19.6 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 48



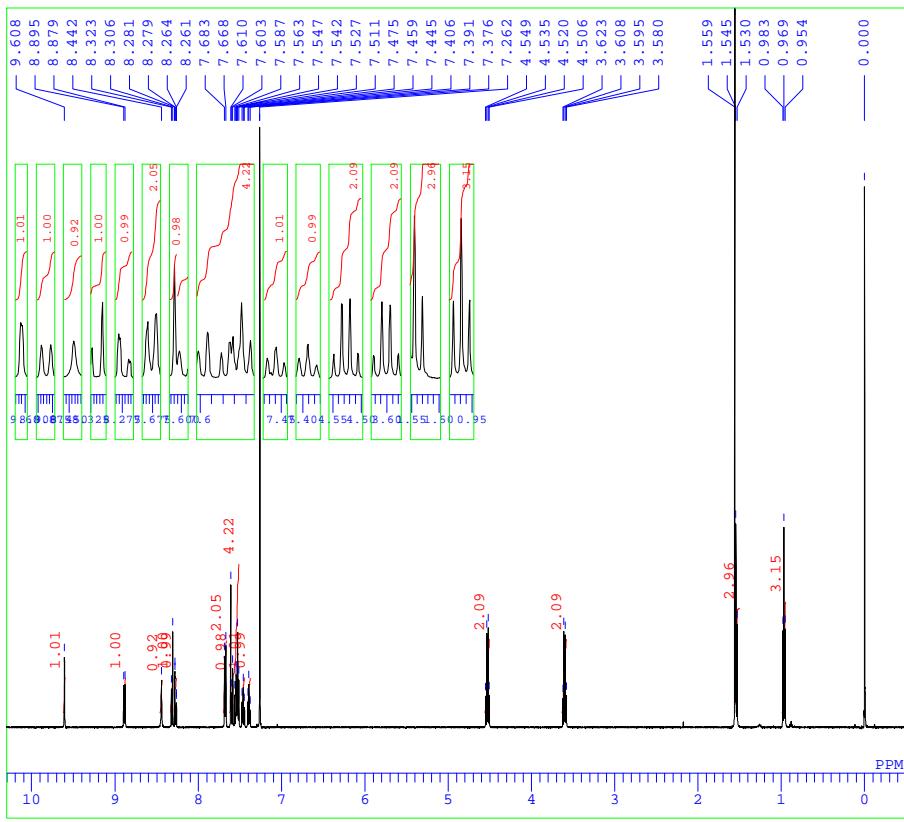
11E



DFILE C-24 C-1.als
 COMNT
 DATIM 2018-03-08 04:06:05
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 kHz
 OBFIN 4.21 Hz
 POINT 13107
 FREQU 25252.14 Hz
 SCANS 2048
 ACQTM 0.5190 sec
 PD 1.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 19.5 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 60



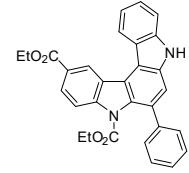
11E



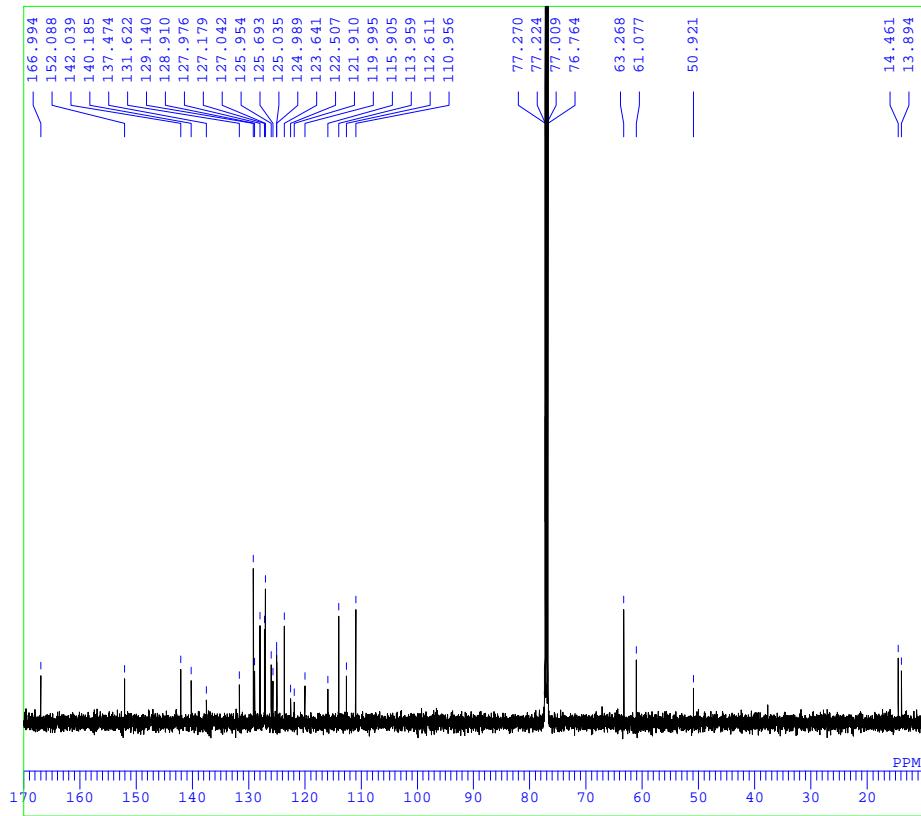
```

DFILE B-194 H-1.als
COMNT single_pulse
DATIM 2018-03-06 18:24:42
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ      500.16 MHz
OBSET       2.41 KHz
OBFIN       6.01 Hz
POINT        13107
FREQU       7507.39 Hz
SCANS         8
ACQTM       1.7459 sec
PD          5.0000 sec
PW1          7.15 usec
IRNUC 1H
CTEMP        19.5 c
SLVNT CDCL3
EXREF       0.00 ppm
BF           0.12 Hz
RGAIN        52

```



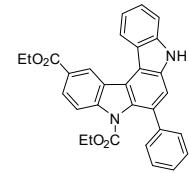
11F



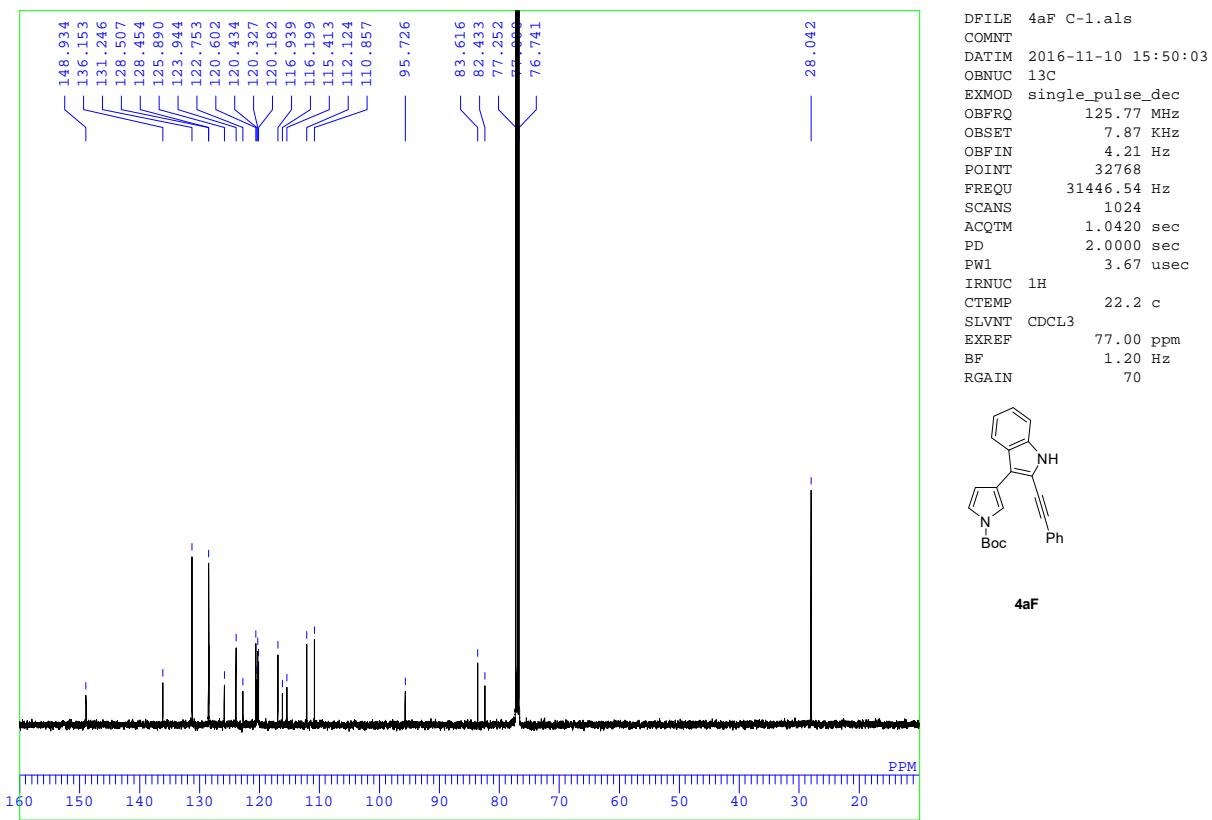
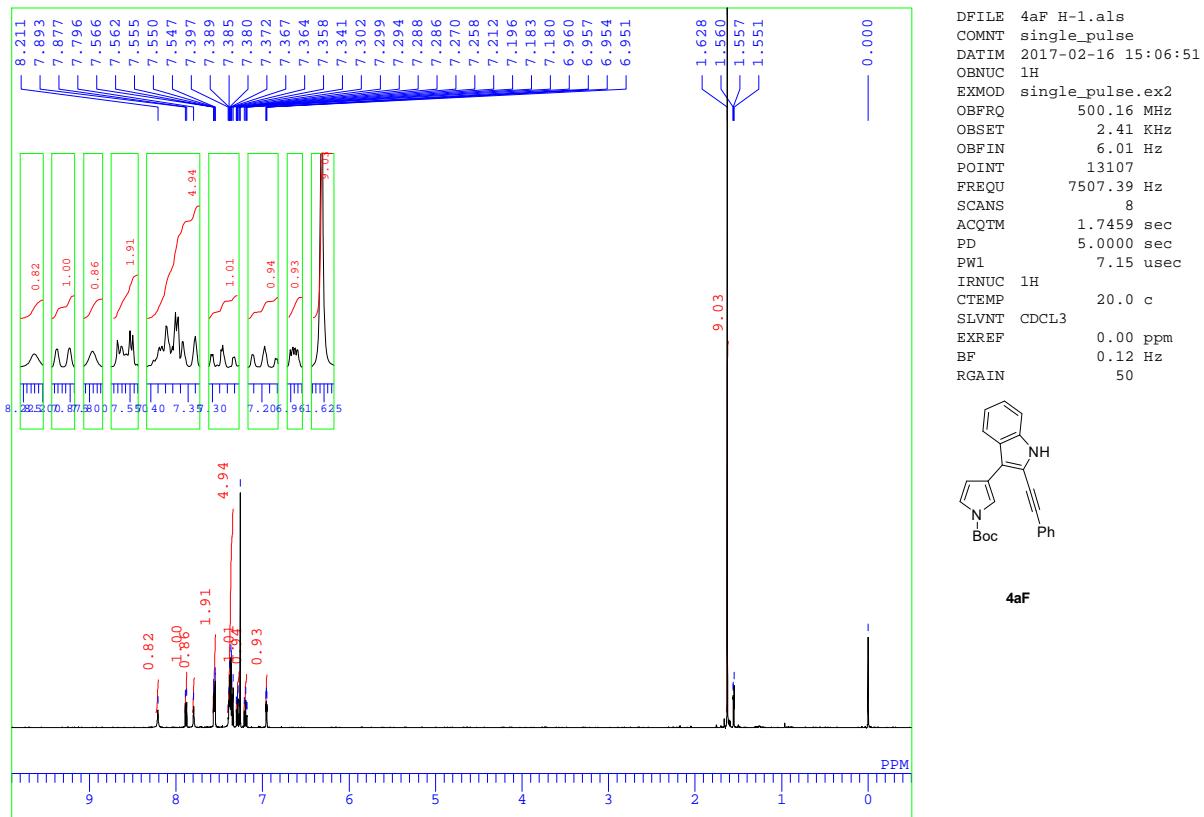
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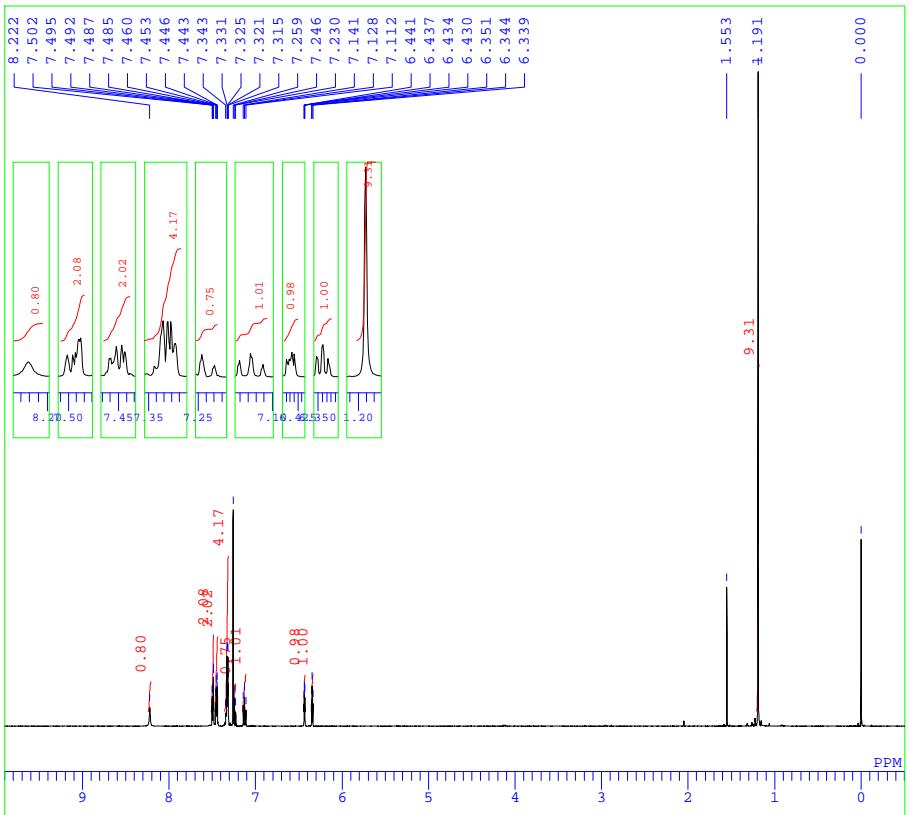
DFILE A194 C.als
COMNT
DATIM 2018-04-29 13:25:59
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 16384
FREQU 31565.66 Hz
SCANS 18969
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 60

```

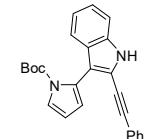


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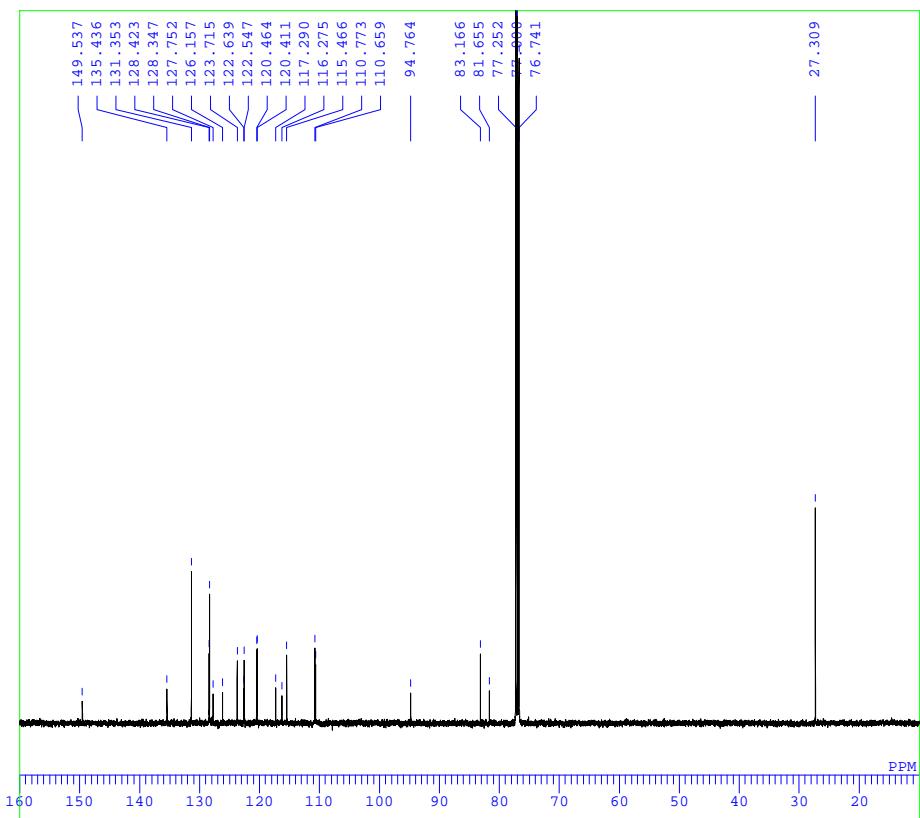




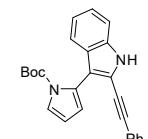
DFILE 5aF H-1.als
 COMNT single_pulse
 DATIM 2017-02-16 15:10:30
 OBNUC 1H
 EXMOD single_pulse.ex2
 OBFRQ 500.16 MHz
 OBSET 2.41 kHz
 OBFIN 6.01 Hz
 POINT 13107
 FREQU 7507.39 Hz
 SCANS 8
 ACQTM 1.7459 sec
 PD 5.0000 sec
 PW1 7.15 usec
 IRNUC 1H
 CTEMP 20.2 c
 SLVNT CDCL₃
 EXREF 0.00 ppm
 BF 0.12 Hz
 RGAIN 50



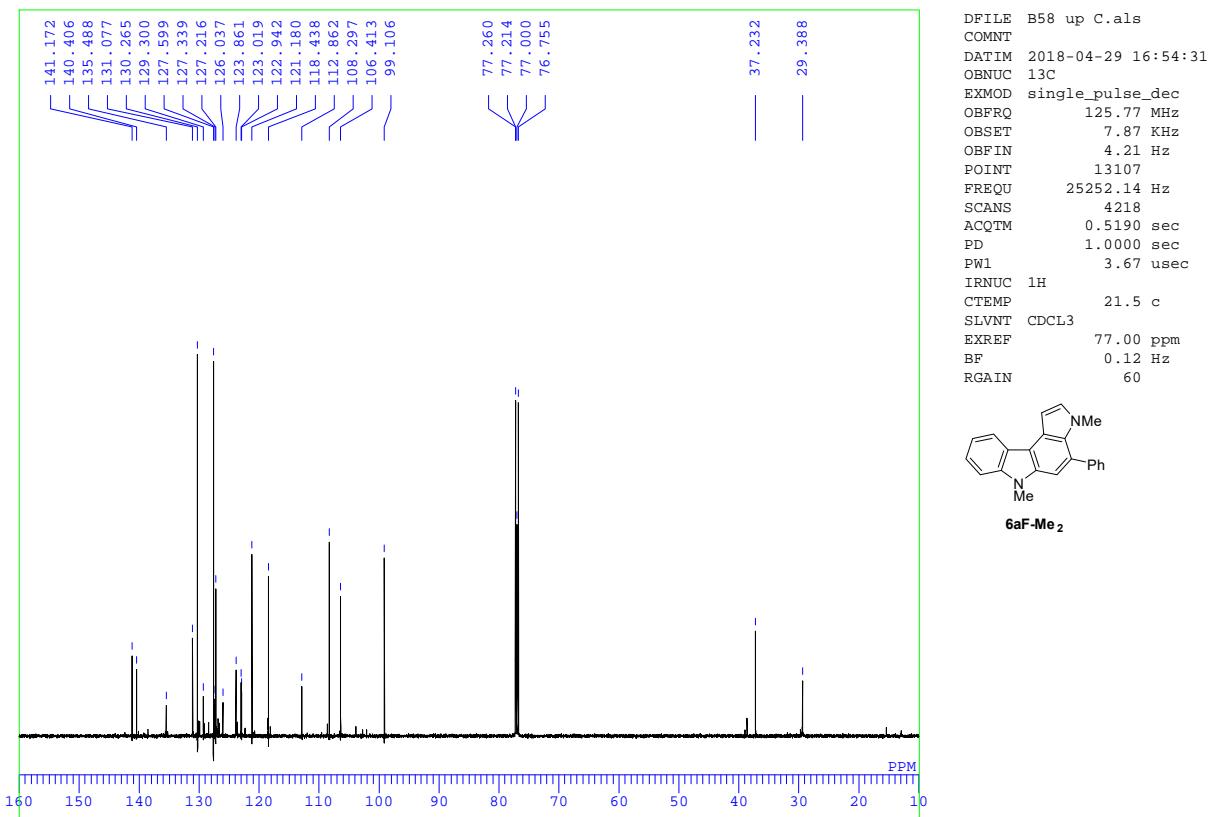
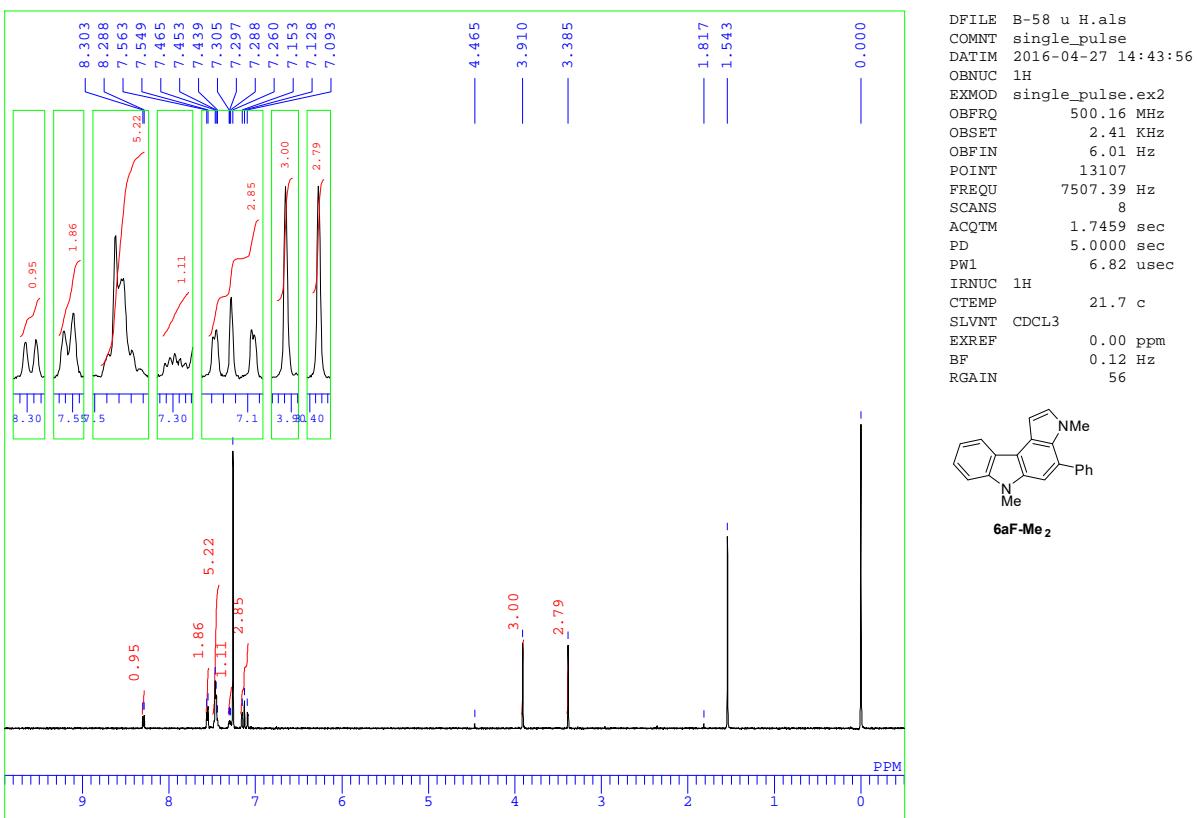
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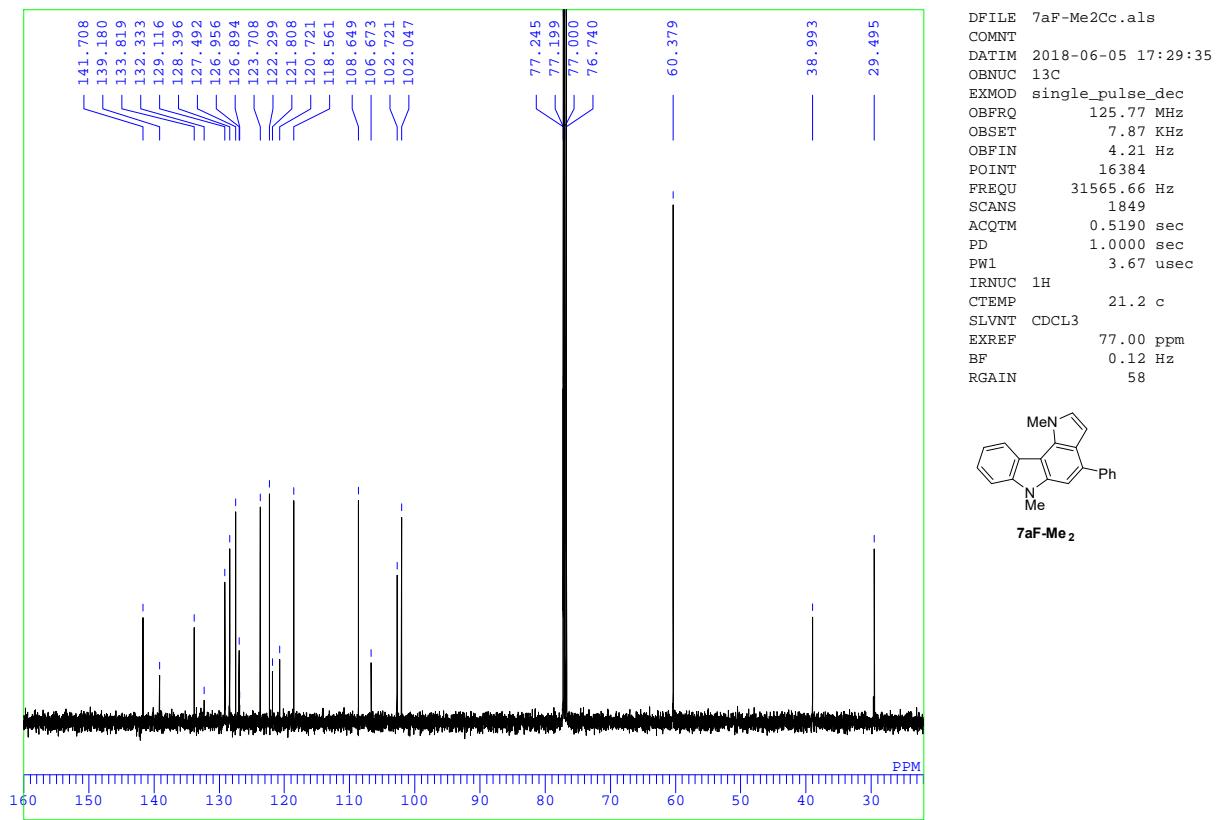
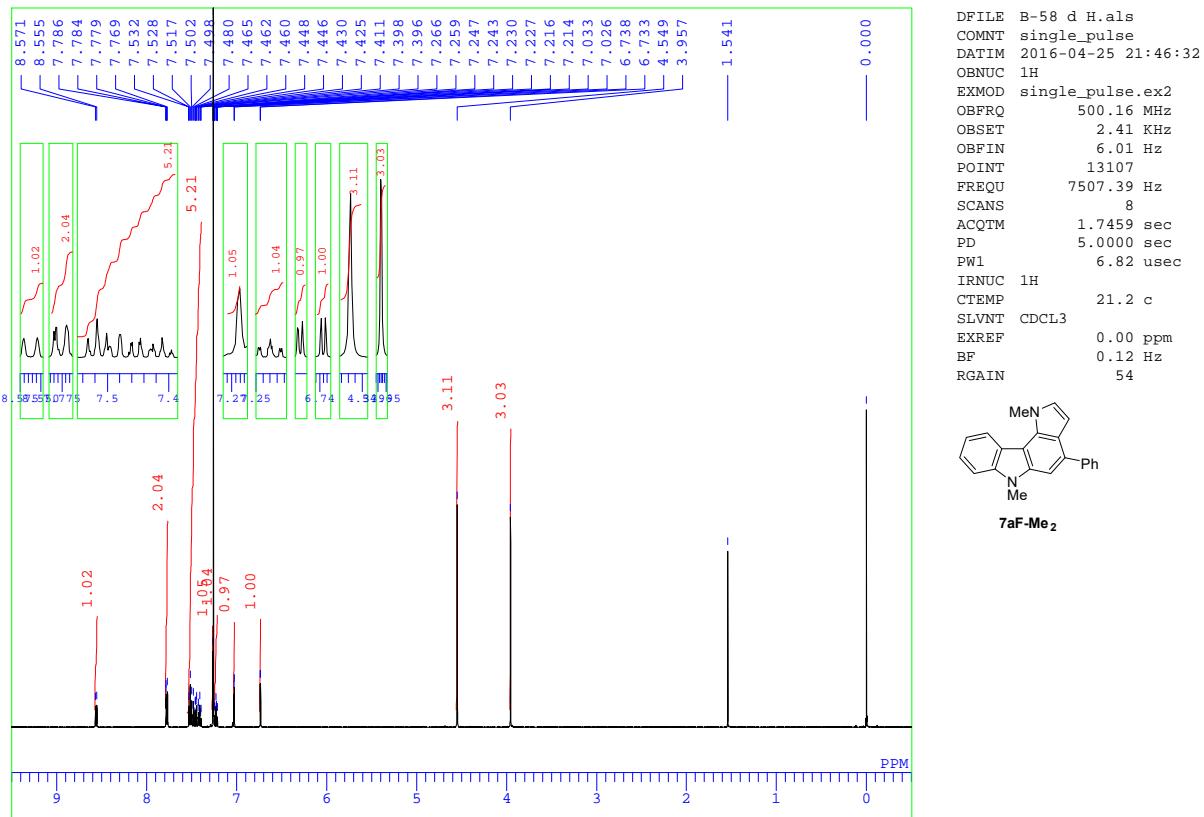


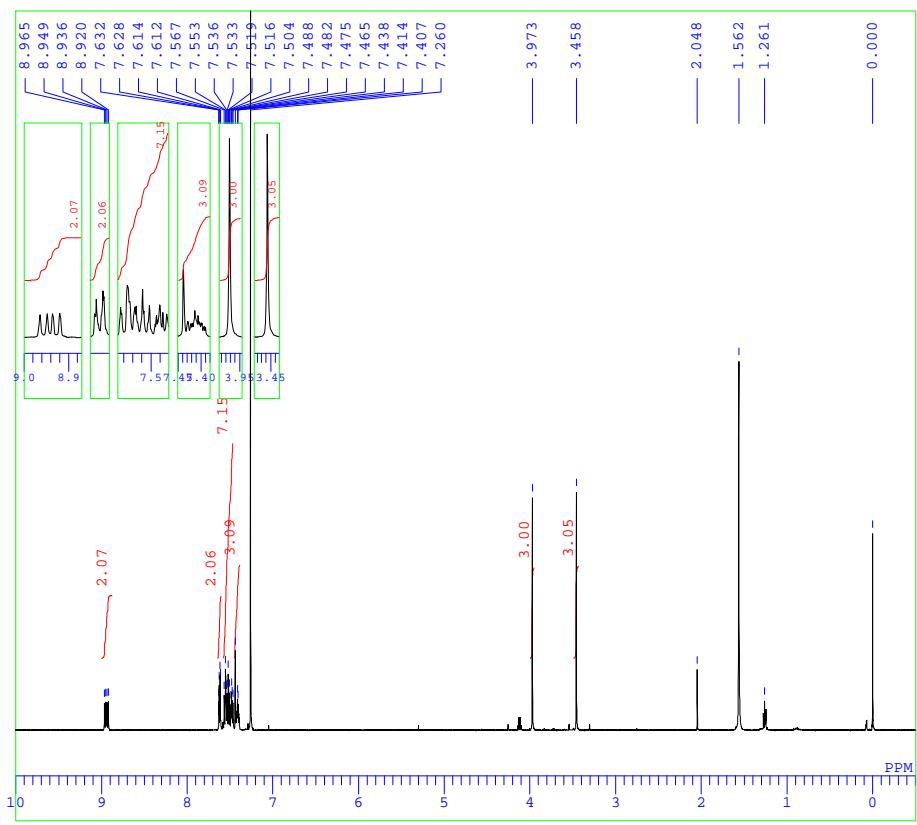
DFILE 5aF C-1.als
 COMNT
 DATIM 2016-11-10 19:17:52
 OBNUC 13C
 EXMOD single_pulse_dec
 OBFRQ 125.77 MHz
 OBSET 7.87 kHz
 OBFIN 4.21 Hz
 POINT 32768
 FREQU 31446.54 Hz
 SCANS 1024
 ACQTM 1.0420 sec
 PD 2.0000 sec
 PW1 3.67 usec
 IRNUC 1H
 CTEMP 22.1 c
 SLVNT CDCL₃
 EXREF 77.00 ppm
 BF 1.20 Hz
 RGAIN 70



5aF



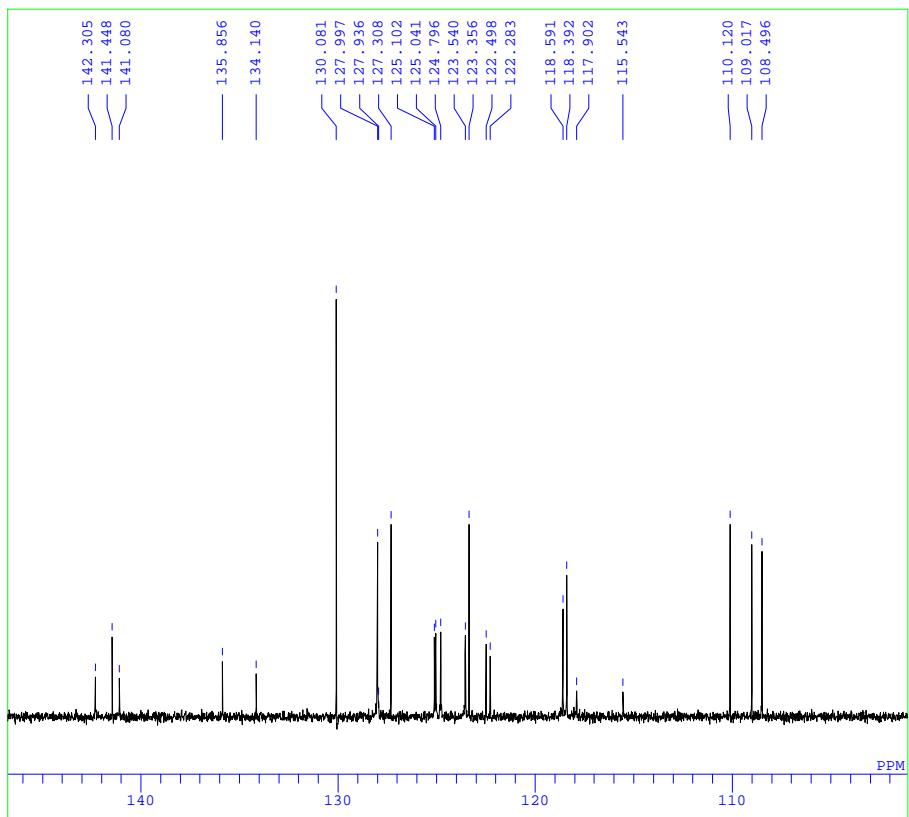
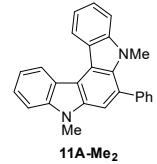




```

FILE B190.als
COMNT single_pulse
DATIM 2018-05-23 18:36:49
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ      500.16 MHz
OBSET       2.41 KHz
OBFIN       6.01 Hz
POINT        13107
FREQU       7507.39 Hz
SCANS         8
ACQTM      1.7459 sec
PD          5.0000 sec
PW1          7.15 usec
IRNUC      1H
CTEMP       20.9 c
SLVNT      CDCL3
EXREF       0.00 ppm
BF          0.12 Hz
RGAIN        50

```



```

DFILE B190 C.als
COMNT
DATIM 2018-05-23 11:22:42
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 16384
FREQU 31565.66 Hz
SCANS 1747
ACQTM 0.5190 sec
PD 1.0000 sec
PW1 3.67 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 60

```

