

Supporting Information

Photoinduced Copper-Catalyzed Asymmetric Decarboxylative Alkynylation with Terminal Alkynes

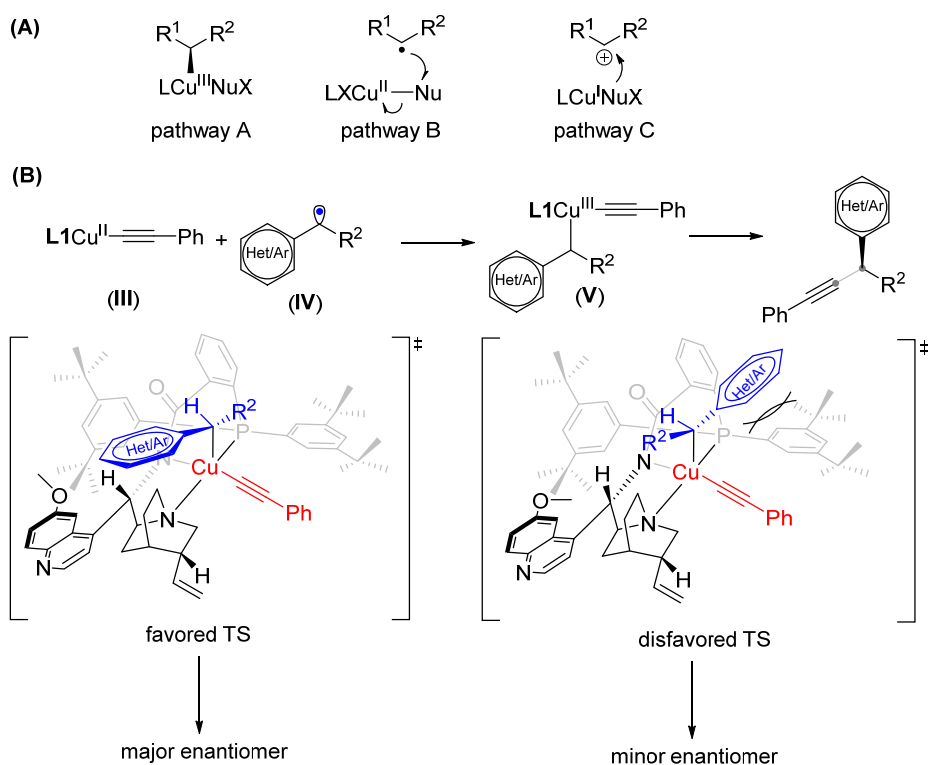
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Scheme S1. Proposed model for asymmetric induction.



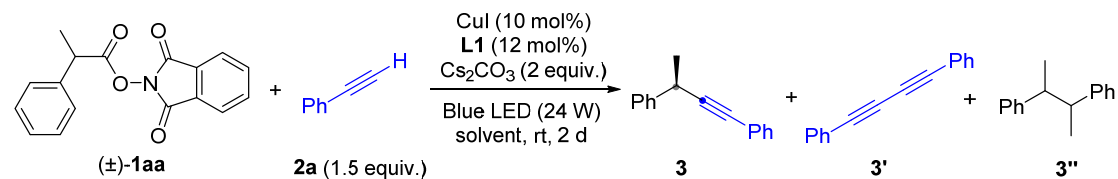
As for the copper-catalyzed radical $C(sp^3)\text{-Nu}$ bond formation, there are mainly three possible pathways between $\text{Cu}^{\text{II}}\text{Nu}$ and the alkyl radical intermediate: (1) the formation of a Cu^{III} species between $\text{Cu}^{\text{II}}\text{Nu}$ and the alkyl radical intermediate, followed by subsequent reductive elimination (pathway A in Scheme S1A); (2) outer-sphere radical substitution (pathway B in Scheme S1A); (3) oxidation of the alkyl radical intermediate to a carbocation intermediate followed by nucleophilic trapping (pathway C in Scheme S1A). At this stage, we have no clear evidence favoring any of these three pathways. Nonetheless, we speculate that the reaction probably proceeds via the formation of Cu^{III} species **V** and subsequent reductive elimination (Scheme S1B). Based on this assumption, we have deduced transition states (TS) that lead to the desired stereochemistry and its mirror image, respectively (Scheme S1B). The unfavored transition state leading to the *S* product involves the clash between the relatively large (hetero)aryl group in substrate and the bulky phosphine moiety in ligand and thus, is unfavored. This result is consistent with the observed stereochemistry (*R* product). The investigation on the proposed formation of Cu^{III} species is still ongoing in our laboratory and the results will be disclosed in due report.

1. General information

Reagents were used as received from commercial suppliers without further purification, unless otherwise stated. CuI was purchased from Sigma-Aldrich. Anhydrous trifluorotoluene (PhCF₃) was purchased from Beijing J&K Scientific Ltd. and was used directly without further purification. NMR spectra were recorded for ¹H NMR (400 MHz), ¹³C NMR (100 MHz) and ¹⁹F NMR (376 MHz) using TMS as an internal standard and Bruker AV 400 as an instrument. The following abbreviations were used to describe peak patterns where appropriate: singlet (s), doublet (d), triplet (t), multiplet (m). High-resolution mass spectroscopy (HRMS) were obtained using Bruker Apex IV RTMS. Enantiomeric excess (ee) was determined by Agilent high-performance liquid chromatography (HPLC) with a Hatachi detector (at appropriate wavelength).

2. Screening of reaction conditions

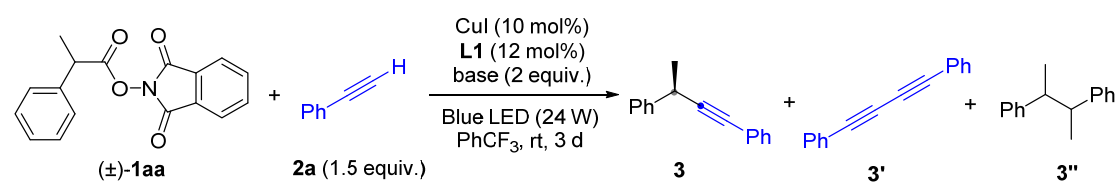
Table S1 Screening of different solvents^[a]



Entry	Solvent	Yield of 3	Yield of 3'	Yield of 3''	ee of 3
1	DMF	< 5%	~30%	9%	NA
2	MeCN	10%	~30%	6%	86%
3	Et ₂ O	ND	~30%	8%	NA
4	THF	9%	~20%	9%	80%
5	fluorobenzene	13%	~30%	16%	92%
6	trifluorotoluene	20%	~30%	13%	92%
7	hexafluorobenzene	5%	~20%	18%	46%
8	EtOAc	ND	~10%	< 5%	NA
9	CH ₂ Cl ₂	14%	~30%	11%	82%

[a] Reaction conditions: (±)-**1aa** (0.050 mmol), **2a** (1.5 equiv.), CuI (10 mol%), **L1** (12 mol%), and Cs₂CO₃ (2.0 equiv.) in anhydrous solvent (0.50 mL) at rt under irradiation of blue LED (24 W) for 2 d under argon. Yields of **3** and **3''** were based on ¹H NMR analysis of the crude product using CH₂Br₂ as an internal standard. Yield of **3'** was the isolated yield. Ee value was determined by HPLC analysis. [b] ND = not detected. [c] NA = not applicable. In most cases, 2-phenylpropanoic acid via the hydrolysis of **1aa** could be detected.

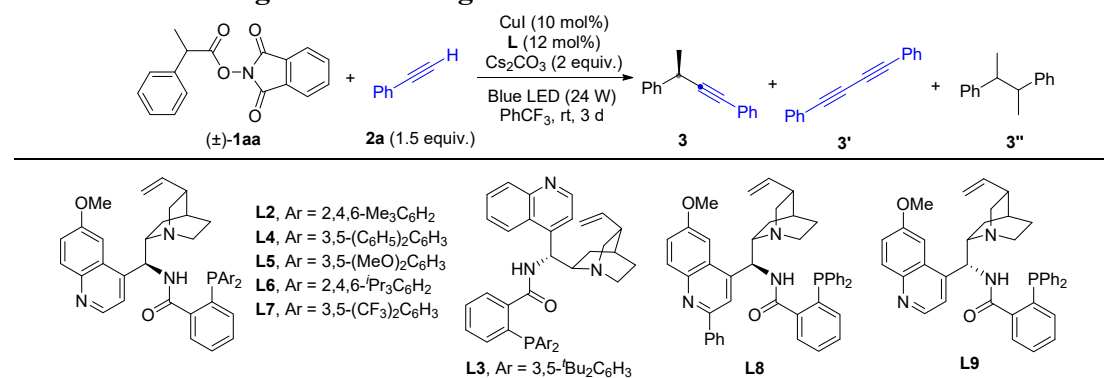
Table S2 Screening of different bases^[a]



Entry	Base	Yield of 3	Yield of 3'	Yield of 3''	ee of 3
1 ^[b]	Cs ₂ CO ₃	20%	~30%	13%	92%
2	Cs₂CO₃	21%	~30%	12%	92%
3	K ₃ PO ₄	13%	~10%	5%	92%
4	K ₂ HPO ₄	ND	< 5%	< 5%	NA
5	CsOAc	ND	< 5%	< 5%	NA
6	Et ₃ N	ND	< 5%	< 5%	NA
7	pyridine	ND	< 5%	< 5%	NA

[a] Reaction conditions: **(±)-1aa** (0.050 mmol), **2a** (1.5 equiv.), CuI (10 mol%), **L1** (12 mol%), and base (2.0 equiv.) in PhCF₃ (0.50 mL) at rt under irradiation of blue LED (24 W) for 3 d under argon. Yields of **3** and **3''** were based on ¹H NMR analysis of the crude product using CH₂Br₂ as an internal standard. Yield of **3'** was the isolated yield. Ee value was determined by HPLC analysis. [b] Reaction time: 2 d.

Table S3 Screening of different ligands^[a]



Entry	L	Yield of 3	Yield of 3'	Yield of 3''	ee of 3
1	L1	21%	~30%	12%	92%
2	L2	< 5%	~10%	< 5%	NA
3	L3	21%	~20%	6%	-72%
4	L4	6%	~20%	7%	70%
5	L5	15%	~30%	16%	76%
6	L6	< 5%	< 5%	< 5%	NA
7	L7	< 5%	~10%	< 5%	NA
8	L8	17%	~30%	14%	58%
9	L9	18%	~10%	< 5%	-48%

[a] Reaction conditions: (±)-**1aa** (0.050 mmol), **2a** (1.5 equiv.), CuI (10 mol%), L (12 mol%), and Cs₂CO₃ (2.0 equiv.) in PhCF₃ (0.50 mL) at rt under irradiation of blue LED (24 W) for 3 d under argon. Yields of **3** and **3''** were based on ¹H NMR analysis of the crude product using CH₂Br₂ as an internal standard. Yield of **3'** was the isolated yield. Ee value was determined by HPLC analysis. In most cases, 2-phenylpropanoic acid via the hydrolysis of **1aa** could be detected.

Table S4. Screening of copper salts^[a]

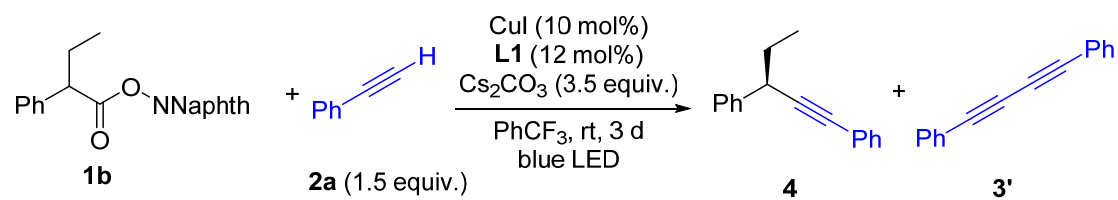
Reaction scheme showing the synthesis of products **3**, **3'**, and **3''** from **(±)-1aa** and **2a** (1.5 equiv.) using various copper salts. The reaction conditions are: [Cu] (10 mol%), **L1** (12 mol%), Cs₂CO₃ (2 equiv.), Blue LED (24 W), PhCF₃, rt, 3 d.

Entry	[Cu] salt	Yield of 3	Yield of 3'	Yield of 3''	ee of 3
1	CuI	21%	~30%	12%	92%
2	CuCl	18%	~20%	5%	92%
3	CuSCN	19%	~20%	7%	92%
4	Cu(MeCN) ₄ PF ₆	17%	~20%	7%	92%
5	CuTc	9%	~30%	14%	72%
6	Cu(PPh ₃)Br	ND	~10%	6%	NA
7	CuCl ₂	ND	~20%	13%	NA

[a] Reaction conditions: **(±)-1aa** (0.050 mmol), **2a** (1.5 equiv.), [Cu] salt (10 mol%), **L1** (12 mol%), and Cs₂CO₃ (2.0 equiv.) in PhCF₃ (0.50 mL) at rt under irradiation of blue LED (24 W) for 3 d under argon. Yield of **3** and **3''** were based on ¹H NMR analysis of the crude product using CH₂Br₂ as an internal standard. Yield of **3'** was the isolated yield. Ee value was determined by HPLC analysis. In most cases, 2-phenylpropanoic acid via the hydrolysis of **1aa** could be detected.

3. Control experiments

Table S5. Variation of reaction parameters^[a]



Entry	Variation	Yield of 4	Yield of 3'	ee of 4
1 ^[b]	No light	11%	0%	97%
2	No CuI	0%	0%	NA
3	No L1	6%	~10%	NA
4	No Cs ₂ CO ₃	0%	0%	NA

[a] Reaction conditions: **1b** (0.050 mmol), **2a** (1.5 equiv.), CuI (10 mol%), **L1** (12 mol%), and Cs₂CO₃ (3.5 equiv.) in PhCF₃ (1 mL) at rt under irradiation of blue LED (24 W) for 3 d under argon. Yield of **4** was based on ¹H NMR analysis of the crude product using CH₂Br₂ as an internal standard. Yield of **3'** was the isolated yield. Ee value was determined by HPLC analysis. [b] The reaction was stirred at rt for 3 d. and then was stirred at reflux for 2 d.

4. Stern-Volmer luminescence quenching experiments

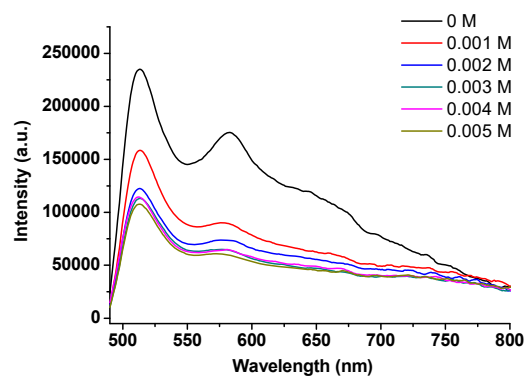


Figure S1a. Cu(I)-phenylacetylide emission quenching by **1b**.

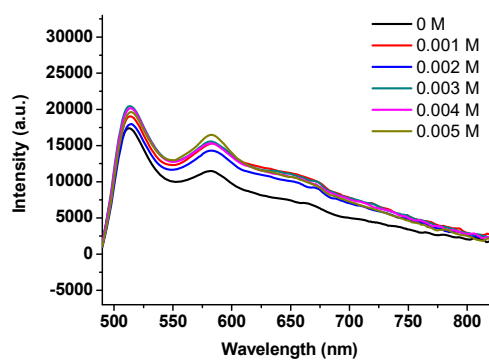


Figure S1b. Cu(I)-phenylacetylide emission quenching by ethynylbenzene (**2a**).

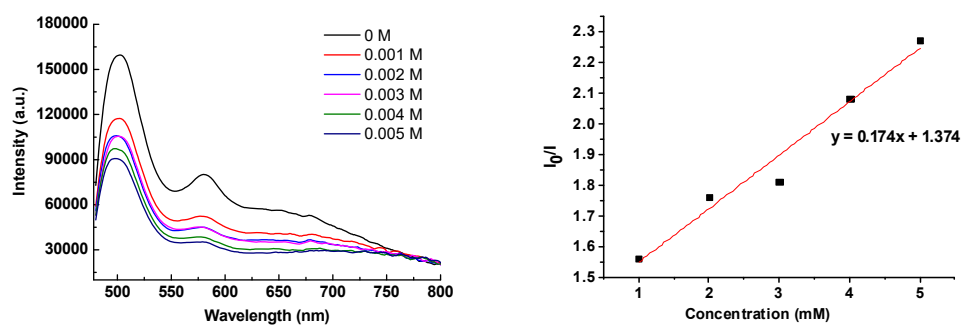


Figure S1c. Cu(I)-phenylacetylide emission quenching by **1aa**.

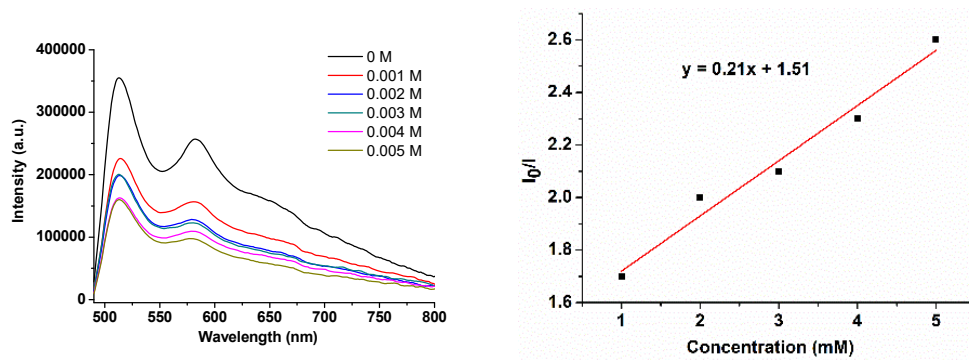


Figure S1d. Cu(I)-phenylacetylide emission quenching by **1ab**.

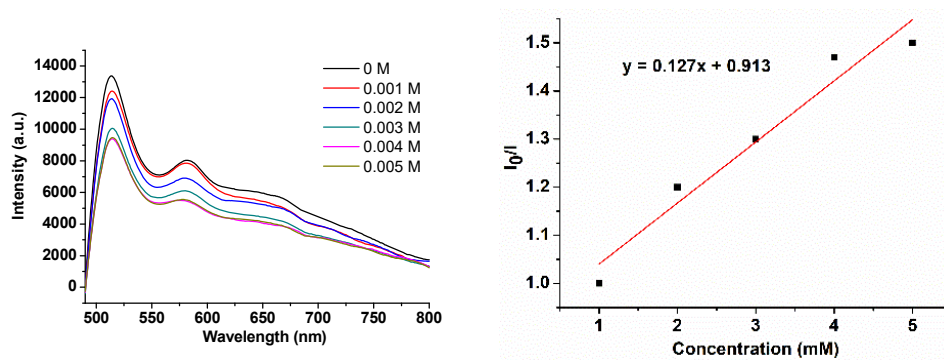


Figure S1e. Cu(I)-phenylacetylide emission quenching by **1ac**.

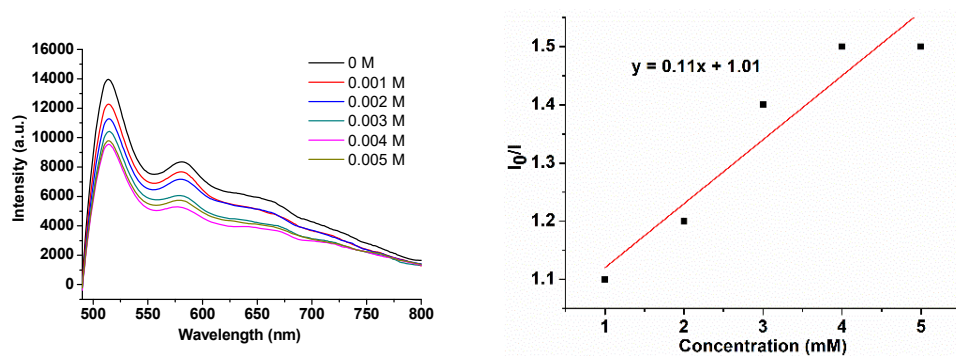


Figure S1f. Cu(I)-phenylacetylide emission quenching by **1ad**.

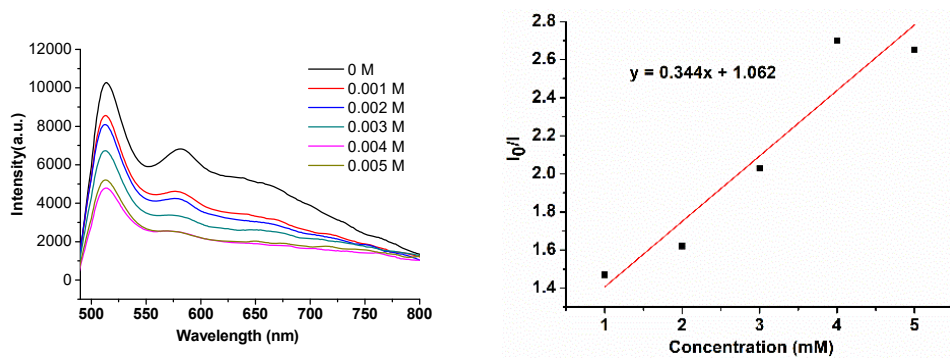


Figure S1g. Cu(I)-phenylacetylide emission quenching by **1ae**.

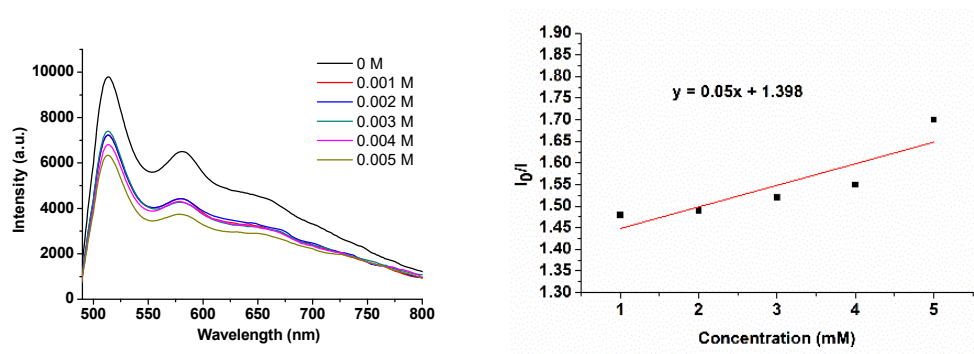


Figure S1h. Cu(I)-phenylacetylide emission quenching by **1af**.

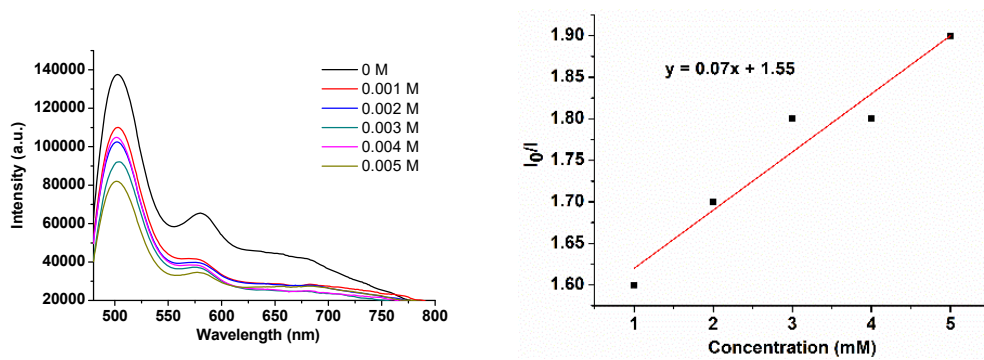


Figure S1i. Cu(I)-phenylacetylide emission quenching by **1ag**.

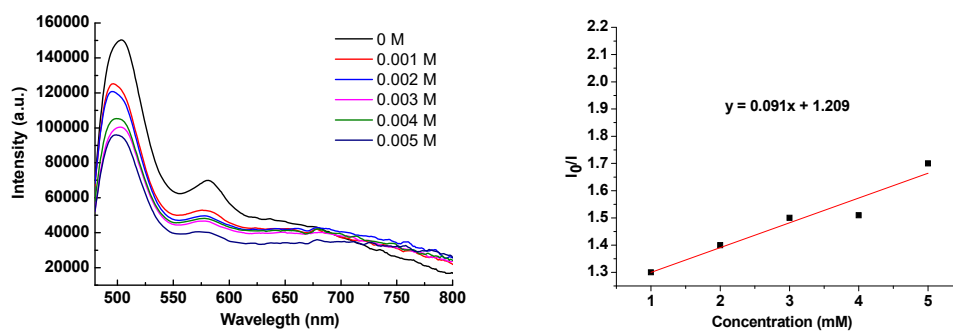


Figure S1j. Cu(I)-phenylacetylide emission quenching by **1ah**.

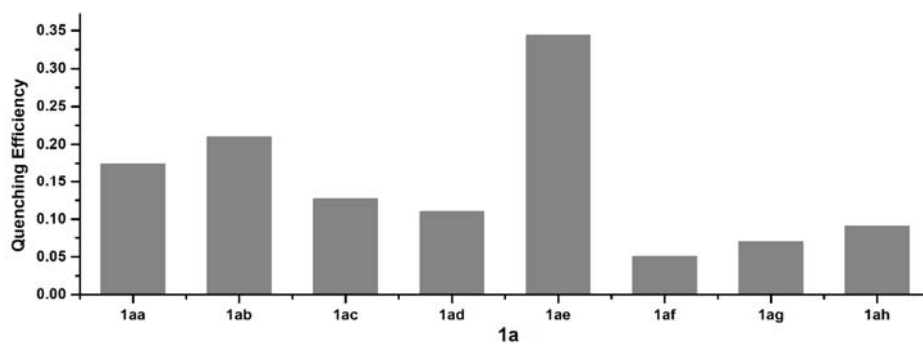


Figure S1k. Comparison of quenching efficiency with **1aa–1ah**.

5. Cyclic voltammetry experiments

Cyclic voltammetry experiment was performed using a CH Instruments Electrochemical Workstation model PalmSens4. The electrochemical cell was equipped with a glassy carbon working electrode, a Pt mesh counter electrode, and a Ag/AgNO₃ (10 mM AgNO₃ in MeCN) reference electrode. A solution of **1b** in MeCN (0.001 M) was tested with 0.1 M Bu₄NPF₆ as the supporting electrolyte and ferrocene was using as an internal standard. Scan rate = 0.1 V/s, 2 sweep segments, a sample interval of 0.001 V.

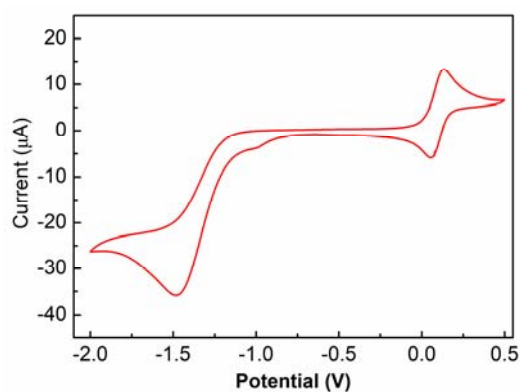
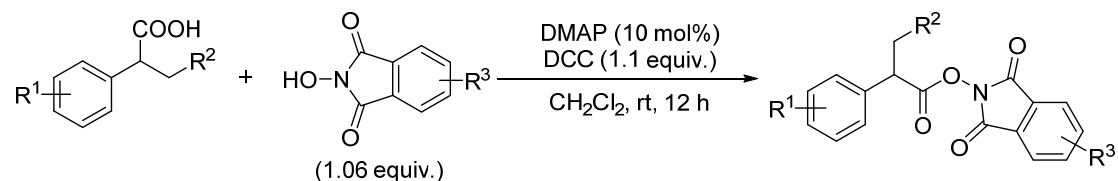


Figure S1. Cyclic voltammogram of **1b** in MeCN. $E_p = -1.567$ V vs. Fc/Fc⁺. E_p (vs. SCE) = -1.567 V + 0.393 V = -1.174 V.

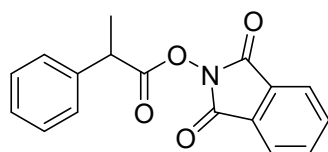
6. The synthesis of *N*-hydroxyphthalimide esters

General procedure 1



To a solution of alkyl carboxylic acid (1.0 equiv.), *N,N*-dimethylpyridin-4-amine (DMAP, 10 mol%) and *N*-hydroxyphthalimide or its analogues (1.06 equiv.) in dry CH_2Cl_2 (0.1 M) was added a solution of *N,N*-dicyclohexylcarbodiimide (DCC, 1.1 equiv.) in CH_2Cl_2 (0.4 M) under an ice-water bath. Then, the reaction mixture was stirred for 12 h at room temperature. After the completion of reaction, the organic solvent was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography on silica gel to afford the pure products.^[1]

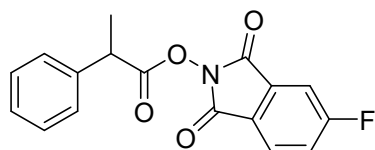
1,3-Dioxoisindolin-2-yl 2-phenylpropanoate (**1aa**)



According to **General procedure 1** with 2-phenylpropanoic acid (300.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxyisoindoline-1,3-dione (345.8 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **1aa** as a colorless solid (442.9 mg, 75% yield).

1H NMR (400 MHz, $CDCl_3$) δ : 7.87-7.85 (m, 2H), 7.79-7.75 (m, 2H), 7.42-7.38 (m, 4H), 7.36-7.31 (m, 1H), 4.13 (q, $J = 7.2$ Hz, 1H), 1.68 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 170.8, 161.8, 138.3, 134.7, 128.9, 128.7, 127.8, 127.5, 123.9, 42.9, 19.0. The NMR spectra were in accord with that reported in literature.^[1]

5-Fluoro-1,3-dioxoisindolin-2-yl 2-phenylpropanoate (**1ab**)

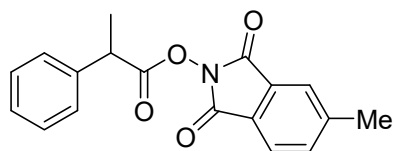


According to **General procedure 1** with 2-phenylpropanoic acid (412.2 mg, 2.7

mmol, 1.0 equiv.) and 5-fluoro-2-hydroxyisoindoline-1,3-dione (522.0 mg, 2.9 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **1ab** as a colorless solid (609.0 mg, 72% yield).

^1H NMR (400 MHz, CDCl_3) δ : 7.89-7.86 (m, 1H), 7.56-7.53 (m, 1H), 7.46-7.43 (m, 1H), 7.42-7.38 (m, 4H), 7.36-7.31 (m, 1H), 4.12 (q, $J = 7.2$ Hz, 1H), 1.67 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.7, 166.6 (d, $J = 257.2$ Hz), 160.6, 138.2, 131.7 (d, $J = 9.5$ Hz), 128.9, 127.8, 127.5, 126.6 (d, $J = 9.4$ Hz), 124.7 (d, $J = 3.2$ Hz), 121.8 (d, $J = 23.4$ Hz), 111.9 (d, $J = 25.0$ Hz), 42.9, 18.9; HRMS (ESI): decomposed to $\text{C}_8\text{H}_4\text{FNO}_3$, m/z calcd for $\text{C}_8\text{H}_3\text{FNO}_3$ $[\text{M}-\text{H}]^-$: 180.0102, found: 180.0095.

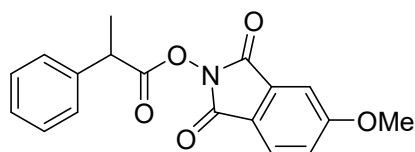
5-Methyl-1,3-dioxisoindolin-2-yl 2-phenylpropanoate (**1ac**)



According to **General procedure 1** with 2-phenylpropanoic acid (300.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-5-methylisoindoline-1,3-dione (375.6 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/4) to yield the product **1ac** as a colorless solid (494.9 mg, 80% yield).

^1H NMR (400 MHz, CDCl_3) δ : 7.73 (d, $J = 7.6$ Hz, 1H), 7.66 (s, 1H), 7.55 (d, $J = 7.6$ Hz, 1H), 7.42-7.37 (m, 4H), 7.36-7.30 (m, 1H), 4.12 (q $J = 7.1$ Hz, 1H), 2.51 (s, 3H), 1.67 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.8, 162.0, 146.2, 138.4, 135.2, 129.2, 128.9, 127.8, 127.6, 126.2, 124.4, 123.9, 42.9, 22.1, 19.0; HRMS (ESI): m/z calcd for $\text{C}_{18}\text{H}_{16}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 310.1074, found: 310.1071.

5-Methoxy-1,3-dioxisoindolin-2-yl 2-phenylpropanoate (**1ad**)

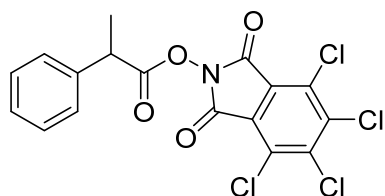


According to **General procedure 1** with 2-phenylpropanoic acid (300.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-5-methoxyisoindoline-1,3-dione (409.5 mg, 2.12

mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **1ad** as a colorless solid (455.4 mg, 70% yield).

^1H NMR (400 MHz, CDCl_3) δ : 7.76 (d, $J = 8.4$ Hz, 1H), 7.42-7.37 (m, 4H), 7.35-7.30 (m, 2H), 7.19 (dd, $J = 8.4, 2.4$ Hz, 1H), 4.11 (q, $J = 7.2$ Hz, 1H), 3.92 (s, 3H), 1.67 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.9, 165.1, 161.8, 138.4, 131.4, 128.9, 127.7, 127.5, 125.8, 120.6, 120.2, 108.9, 56.1, 42.9, 19.0; HRMS (ESI): m/z calcd for $\text{C}_{18}\text{H}_{16}\text{NO}_5$ $[\text{M}+\text{H}]^+$: 326.1023, found: 326.1019.

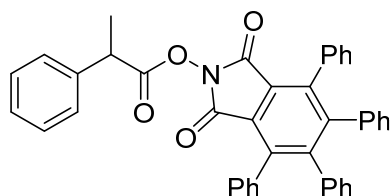
4,5,6,7-Tetrachloro-1,3-dioxoisoindolin-2-yl 2-phenylpropanoate (**1ae**)



According to **General procedure 1** with 2-phenylpropanoic acid (300.4 mg, 2.0 mmol, 1.0 equiv.) and 4,5,6,7-tetrachloro-2-hydroxyisoindoline-1,3-dione (637.9 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **1ae** as a colorless solid (563.0 mg, 65% yield).

^1H NMR (400 MHz, CDCl_3) δ : 7.42-7.37 (m, 4H), 7.35-7.31 (m, 1H), 4.12 (q, $J = 7.2$ Hz, 1H), 1.67 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.3, 157.4, 141.0, 137.9, 130.4, 129.0, 127.9, 127.5, 124.7, 42.9, 18.9; HRMS (ESI): decomposed to $\text{C}_8\text{HCl}_4\text{NO}_3$, m/z calcd for $\text{C}_8\text{HCl}_4\text{NO}_3$ $[\text{M}-\text{H}]^-$: 297.8638, found: 297.8639.

1,3-Dioxo-4,5,6,7-tetraphenylisoindolin-2-yl 2-phenylpropanoate (**1af**)

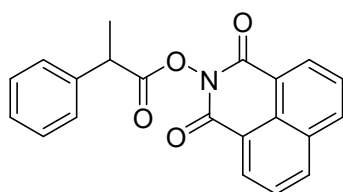


According to **General procedure 1** with 2-phenylpropanoic acid (758.4 mg, 5.1 mmol, 1.0 equiv.) and 2-hydroxy-4,5,6,7-tetraphenylisoindoline-1,3-dione (2.5 g, 5.4 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column

chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **1af** as a colorless solid (2.2 g, 71% yield).

^1H NMR (400 MHz, CDCl_3) δ : 7.32-7.31 (m, 4H), 7.27-7.24 (m, 1H), 7.19-7.18 (m, 6H), 7.11 (s, 4H), 6.90-6.88 (m, 6H), 6.74 (s, 4H), 4.00 (q, $J = 7.2$ Hz, 1H), 1.60 (d, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.6, 148.5, 140.1, 138.2, 137.7, 135.0, 130.6, 129.8, 128.8, 127.6, 127.59, 127.55, 127.4, 127.0, 126.4, 125.1, 42.9, 19.0; HRMS (ESI): m/z calcd for $\text{C}_{41}\text{H}_{30}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 600.2169, found: 600.2164.

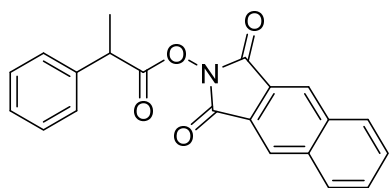
1,3-Dioxo-1*H*-benzo[de]isoquinolin-2(3*H*)-yl 2-phenylpropanoate (**1ag**)



According to **General procedure 1** with 2-phenylpropanoic acid (300.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[de]isoquinoline-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **1ag** as a colorless solid (497.3 mg, 72% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.61-8.55 (m, 2H), 8.25 (d, $J = 8.0$ Hz, 2H), 7.76 (q, $J = 8.0$ Hz, 2H), 7.51 (d, $J = 7.6$ Hz, 2H), 7.43 (t, $J = 7.6$ Hz, 2H), 7.34 (t, $J = 7.2$ Hz, 1H), 4.24 (q, $J = 7.2$ Hz, 1H), 1.76 (d, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.6, 159.6, 159.4, 138.7, 135.0, 134.9, 131.9, 131.8, 128.8, 127.8, 127.7, 127.6, 127.1, 122.3, 43.2, 19.0; HRMS (ESI): m/z calcd for $\text{C}_{21}\text{H}_{16}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 346.1074, found: 346.1070.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylpropanoate (**1ah**)

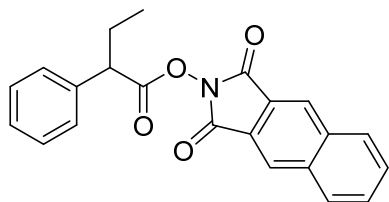


According to **General procedure 1** with 2-phenylpropanoic acid (1.3 g, 8.8 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (2.0 g, 9.3 mmol, 1.06

equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **1ah** as a colorless solid (2.1 g, 70% yield).

¹H NMR (400 MHz, CDCl₃) δ : 8.35 (s, 2H), 8.07-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.46-7.39 (m, 4H), 7.36-7.32 (m, 1H), 4.16 (q, J = 7.1 Hz, 1H), 1.70 (d, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 170.6, 161.4, 138.4, 135.4, 130.4, 129.6, 128.9, 127.8, 127.6, 125.7, 124.4, 43.0, 19.0; HRMS (ESI): m/z calcd for C₂₁H₁₆NO₄ [M+H]⁺: 346.1074, found: 346.1070.

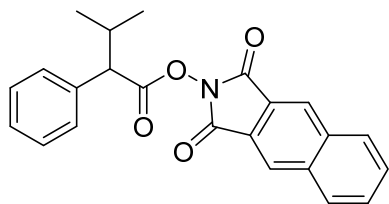
1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-phenylbutanoate (**1b**)



According to **General procedure 1** with 2-phenylbutanoic acid (328.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[*f*]isoindole-1,3(2H)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **1b** as a colorless solid (503.1 mg, 70% yield).

¹H NMR (400 MHz, CDCl₃) δ : 8.35 (s, 2H), 8.07-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.44-7.38 (m, 4H), 7.36-7.31 (m, 1H), 3.89 (t, J = 7.6 Hz, 1H), 2.33-2.22 (m, 1H), 2.04-1.93 (m, 1H), 1.06 (t, J = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 170.1, 161.4, 136.8, 135.4, 130.4, 129.6, 128.8, 128.1, 127.8, 125.7, 124.4, 50.5, 27.2, 11.9; HRMS (ESI): m/z calcd for C₂₂H₁₈NO₄ [M+H]⁺: 360.1230, found: 360.1228.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 3-methyl-2-phenylbutanoate (**S5**)

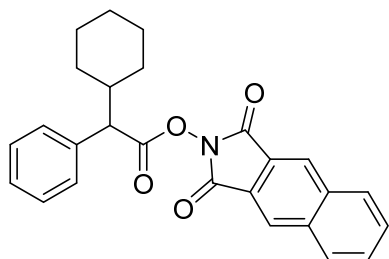


According to **General procedure 1** with 3-methyl-2-phenylbutanoic acid (356.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[*f*]isoindole-1,3(2H)-dione (452.0 mg,

2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S5** as a colorless solid (470.5 mg, 63% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.34 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.43-7.31 (m, 5H), 3.61 (d, $J = 9.6$ Hz, 1H), 2.53-2.40 (m, 1H), 1.25 (d, $J = 6.8$ Hz, 3H), 0.85 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.9, 161.4, 136.0, 135.4, 130.4, 129.6, 128.7, 128.6, 127.9, 125.7, 124.4, 56.6, 32.4, 21.2, 20.2; HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{20}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 374.1387, found: 374.1388.

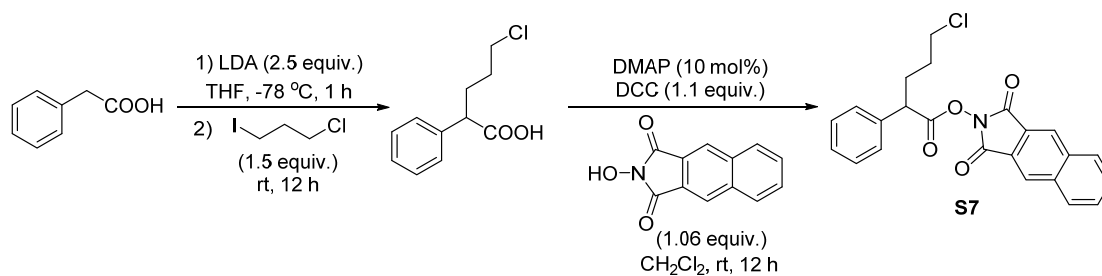
1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-cyclohexyl-2-phenylacetate (S6)



According to **General procedure 1** with 2-cyclohexyl-2-phenylacetic acid (436.6 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S6** as a colorless solid (504.4 mg, 61% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.33 (s, 2H), 8.06-8.04 (m, 2H), 7.73-7.70 (m, 2H), 7.42-7.31 (m, 5H), 3.67 (d, $J = 9.6$ Hz, 1H), 2.16-2.09 (m, 2H), 1.86-1.83 (m, 1H), 1.68-1.66 (m, 2H), 1.47-1.44 (m, 1H), 1.37-1.17 (m, 4H), 0.93-0.85 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.9, 161.4, 135.6, 135.4, 130.4, 129.6, 128.7, 127.8, 125.7, 124.4, 55.6, 41.5, 31.6, 30.4, 26.2, 26.0, 25.9; HRMS (ESI): m/z calcd for $\text{C}_{26}\text{H}_{24}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 414.1700, found: 414.1701.

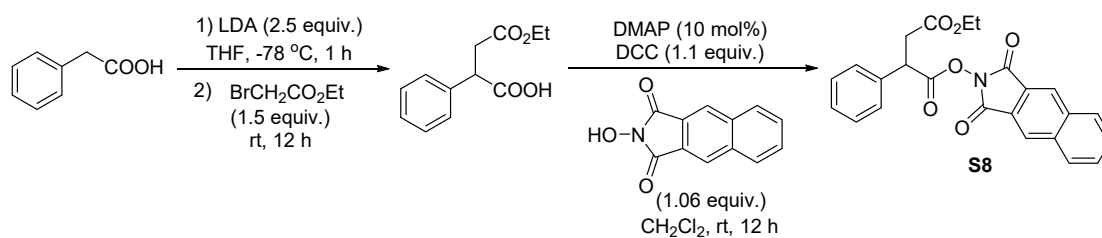
1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 5-chloro-2-phenylpentanoate (S7)



To a stirred solution of $\text{LiN}(i\text{Pr})_2$ (LDA, 7.5 mL, 15 mmol, 2.5 equiv., 2.0 M in THF) in dry THF (20 mL) was added dropwise the solution of 2-phenylacetic acid (6 mmol, 1 equiv.) in dry THF (15 mL) under Ar at $-78\text{ }^\circ\text{C}$. The reaction mixture was stirred at $0\text{ }^\circ\text{C}$ for 1 h and cooled to $-78\text{ }^\circ\text{C}$ again. Then, 1-chloro-3-iodopropane (1.84 g, 9 mmol) was added and the reaction mixture was stirred for 12 h at room temperature. After the completion of reaction, the reaction was quenched with aqueous HCl (1 M). The reaction mixture was extracted with CH_2Cl_2 ($3 \times 10\text{ mL}$). The combined organic layers were washed with saturated brine ($2 \times 30\text{ mL}$) and dried with over anhydrous MgSO_4 . The organic solvent was removed by rotary evaporator under vacuum to yield 5-chloro-2-phenylpentanoic acid which was used directly without further purification. According to **General procedure 1** with 5-chloro-2-phenylpentanoic acid (425.3 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S7** as a colorless solid (571.0 mg, 70% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.34 (s, 2H), 8.06-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.44-7.33 (m, 5H), 4.01 (t, $J = 7.6\text{ Hz}$, 1H), 3.65-3.54 (m, 2H), 2.41-2.32 (m, 1H), 2.20-2.11 (m, 1H), 2.03-1.82 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.8, 161.3, 136.4, 135.4, 130.4, 129.7, 129.0, 128.1, 128.0, 125.8, 124.3, 48.2, 44.3, 31.1, 30.0; HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{19}\text{ClNO}_4$ $[\text{M}+\text{H}]^+$: 408.0997, found: 408.0997.

1-(1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl) 4-ethyl 2-phenylsuccinate (S8)

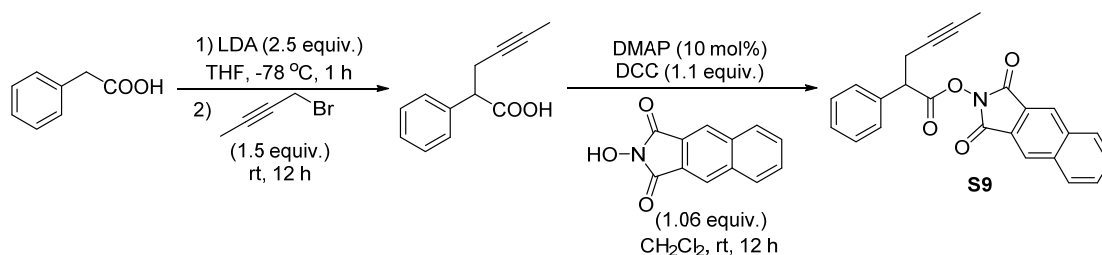


The synthetic procedure of 4-ethoxy-4-oxo-2-phenylbutanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 4-ethoxy-4-oxo-2-phenylbutanoic acid (444.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S8** as a colorless solid (542.6 mg, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.34 (s, 2H), 8.05-8.04 (m, 2H), 7.72-7.70 (m, 2H), 7.44-7.36 (m, 5H), 4.55 (t, *J* = 7.6 Hz, 1H), 4.20 (q, *J* = 6.8 Hz, 2H), 3.31-3.25 (m, 1H), 2.90-2.85 (m, 1H), 1.26 (t, *J* = 7.0 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.2, 169.4, 161.1, 135.6, 135.4, 130.4, 129.6, 129.1, 128.3, 128.0, 125.7, 124.3, 61.2, 45.0, 38.2, 14.1; HRMS (ESI): *m/z* calcd for C₂₄H₂₀NO₆ [M+H]⁺: 418.1285, found: 418.1285.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylhex-4-ynoate (**S9**)

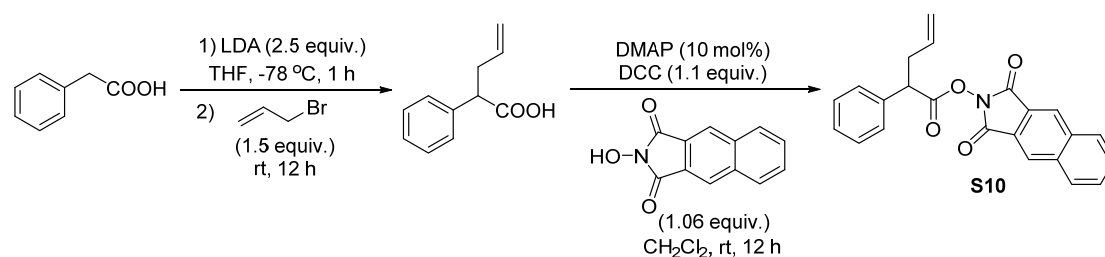


The synthetic procedure of 2-phenylhex-4-ynoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-phenylhex-4-ynoic acid (376.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S9** as a colorless solid (460.1 mg, 60% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.35 (s, 2H), 8.07-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.46-7.34 (m, 5H), 4.20 (t, $J = 7.6$ Hz, 1H), 3.05-2.97 (m, 1H), 2.77-2.69 (m, 1H), 1.81 (t, $J = 2.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.1, 161.2, 135.8, 135.4, 130.4, 129.6, 128.9, 128.2, 127.9, 125.7, 124.3, 78.8, 74.8, 48.8, 24.0, 3.5; HRMS (ESI): m/z calcd for $\text{C}_{24}\text{H}_{18}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 384.1230, found: 384.1230.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylpent-4-enoate (**S10**)

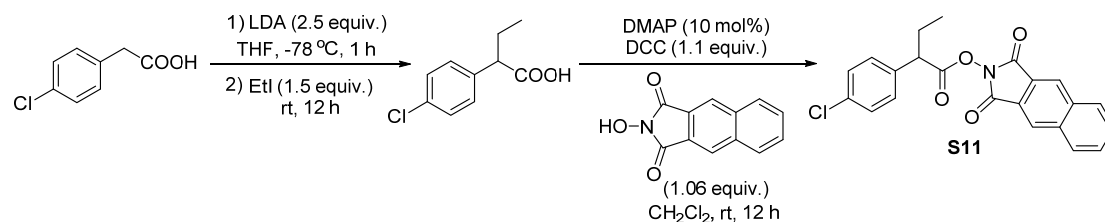


The synthetic procedure of 2-phenylpent-4-enoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-phenylpent-4-enoic acid (352.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S10** as a colorless solid (453.1 mg, 61% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.34 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.44-7.32 (m, 5H), 5.89-5.78 (m, 1H), 5.20-5.12 (m, 2H), 4.07 (t, $J = 7.6$ Hz, 1H), 3.02-2.95 (m, 1H), 2.74-2.67 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.6, 161.3, 136.3, 135.4, 133.9, 130.4, 129.6, 128.9, 128.1, 128.0, 125.7, 124.4, 118.1, 48.7, 37.8; HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{18}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 372.1230, found: 372.1231.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-chlorophenyl)butanoate (**S11**)

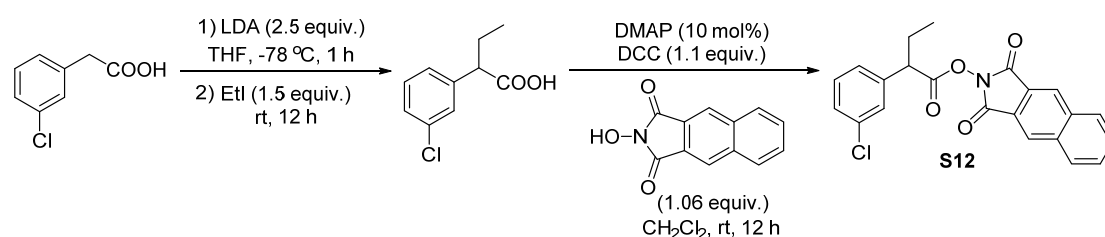


The synthetic procedure of 2-(4-chlorophenyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(4-chlorophenyl)butanoic acid (397.2 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S11** as a colorless solid (590.7 mg, 75% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.35 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.71 (m, 2H), 7.39-7.34 (m, 4H), 3.86 (t, *J* = 7.6 Hz, 1H), 2.31-2.20 (m, 1H), 2.01-1.90 (m, 1H), 1.05 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 169.8, 161.3, 135.4, 135.3, 133.8, 130.4, 129.7, 129.5, 129.0, 125.8, 124.3, 49.9, 27.2, 11.8; HRMS (ESI): *m/z* calcd for C₂₂H₁₇ClNO₄ [M+H]⁺: 394.0841, found: 394.0839.

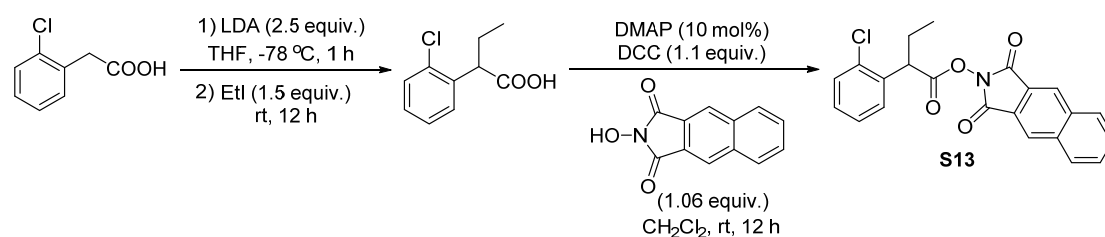
1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(3-chlorophenyl)butanoate (**S12**)



The synthetic procedure of 2-(3-chlorophenyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(3-chlorophenyl)butanoic acid (397.2 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S12** as a colorless solid (551.4 mg, 70% yield).

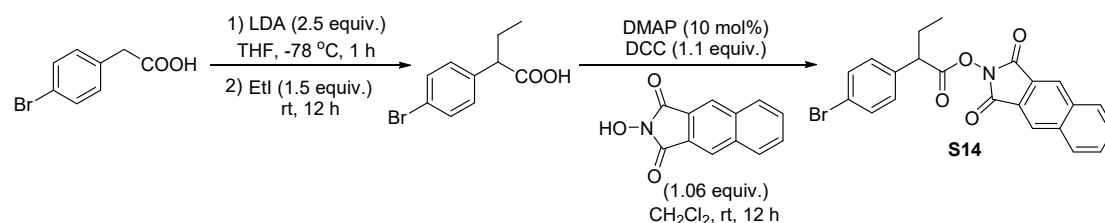
¹H NMR (400 MHz, CDCl₃) δ: 8.35 (s, 2H), 8.07-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.43 (s, 1H), 7.36-7.30 (m, 3H), 3.86 (t, *J* = 7.6 Hz, 1H), 2.32-2.21 (m, 1H), 2.03-1.92 (m, 1H), 1.07 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 169.6, 161.3, 138.7, 135.5, 134.7, 130.4, 130.1, 129.7, 128.4, 128.1, 126.3, 125.8, 124.4, 50.2, 27.2, 11.8; HRMS (ESI): *m/z* calcd for C₂₂H₁₇ClNO₄ [M+H]⁺: 394.0841, found: 394.0841.

1,3-Dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl 2-(2-chlorophenyl)butanoate**(S13)**

The synthetic procedure of 2-(2-chlorophenyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound S7).

According to **General procedure 1** with 2-(2-chlorophenyl)butanoic acid (397.2 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[f]isoindole-1,3(2H)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S13** as a colorless solid (496.2 mg, 63% yield).

¹H NMR (400 MHz, CDCl₃) δ : 8.35 (s, 2H), 8.06-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.52 (dd, $J = 7.6, 1.6$ Hz, 1H), 7.45 (dd, $J = 8.0, 1.6$ Hz, 1H), 7.34 (td, $J = 7.6, 1.2$ Hz, 1H), 7.29-7.25 (m, 1H), 4.54 (t, $J = 7.4$ Hz, 1H), 2.30-2.19 (m, 1H), 2.05-1.94 (m, 1H), 1.08 (t, $J = 7.4$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 169.7, 161.3, 135.5, 134.8, 134.1, 130.4, 129.8, 129.7, 129.0, 128.8, 127.3, 125.7, 124.4, 46.4, 26.5, 11.7; HRMS (ESI): m/z calcd for C₂₂H₁₇ClNO₄ [M+H]⁺: 394.0841, found: 394.0840.

1,3-Dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl 2-(4-bromophenyl)butanoate**(S14)**

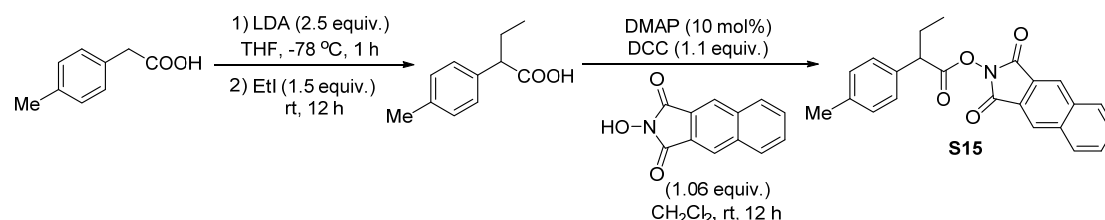
The synthetic procedure of 2-(4-bromophenyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound S7).

According to **General procedure 1** with 2-(4-bromophenyl)butanoic acid (486.1 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[f]isoindole-1,3(2H)-dione (452.0 mg,

2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S14** as a colorless solid (622.4 mg, 71% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.35 (s, 2H), 8.07-8.04 (m, 2H), 7.73-7.71 (m, 2H), 7.53 (d, $J = 8.4$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 2H), 3.85 (t, $J = 7.6$ Hz, 1H), 2.31-2.20 (m, 1H), 2.01-1.90 (m, 1H), 1.05 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.7, 161.3, 135.9, 135.5, 132.0, 130.4, 129.8, 129.7, 125.8, 124.3, 121.9, 50.0, 27.2, 11.8; HRMS (ESI): m/z calcd for $\text{C}_{22}\text{H}_{17}\text{BrNO}_4$ $[\text{M}+\text{H}]^+$: 438.0335, found: 438.0335.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-(*p*-tolyl)butanoate (**S15**)

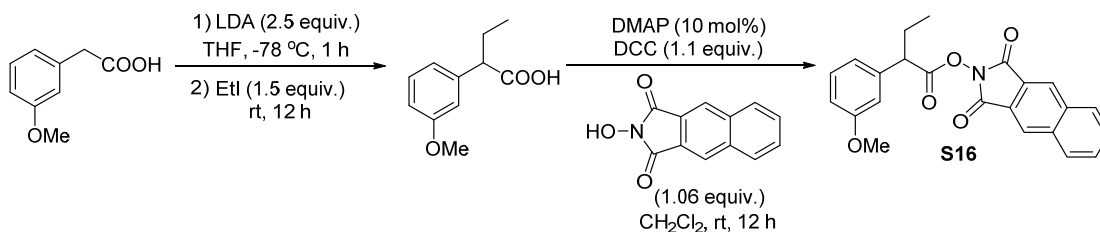


The synthetic procedure of 2-(*p*-tolyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(*p*-tolyl)butanoic acid (356.3 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[*f*]isoindole-1,3(2H)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S15** as a colorless solid (560.1 mg, 75% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.34 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.21 (d, $J = 8.0$ Hz, 2H), 3.85 (t, $J = 7.6$ Hz, 1H), 2.36 (s, 3H), 2.31-2.20 (m, 1H), 2.02-1.91 (m, 1H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.3, 161.4, 137.5, 135.4, 133.9, 130.4, 129.6, 129.5, 127.9, 125.7, 124.4, 50.1, 27.2, 21.1, 11.9; HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{20}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 374.1387, found: 374.1386.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-(3-methoxyphenyl)butanoate (**S16**)

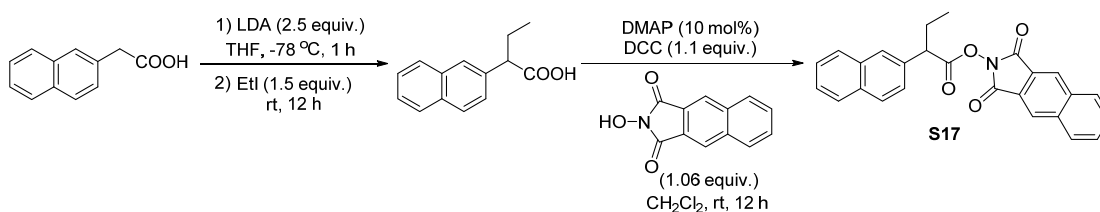


The synthetic procedure of 2-(3-methoxyphenyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(3-methoxyphenyl)butanoic acid (388.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S16** as a colorless solid (599.7 mg, 77% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.35 (s, 2H), 8.07-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.31 (t, *J* = 7.8 Hz, 1H), 7.01-6.99 (m, 2H), 6.89-6.86 (m, 1H), 3.89-3.85 (m, 4H), 2.31-2.21 (m, 1H), 2.04-1.93 (m, 1H), 1.07 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.0, 161.4, 159.9, 138.3, 135.4, 130.4, 129.7, 129.6, 125.7, 124.4, 120.5, 113.5, 55.3, 50.5, 27.2, 11.9; HRMS (ESI): *m/z* calcd for C₂₃H₂₀NO₅ [M+H]⁺: 390.1336, found: 390.1332.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(naphthalen-2-yl)butanoate (**S17**)

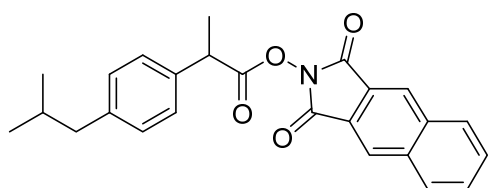


The synthetic procedure of 2-(naphthalen-2-yl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(naphthalen-2-yl)butanoic acid (428.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S17** as a colorless solid (573.2 mg, 70% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.34 (s, 2H), 8.05-8.03 (m, 2H), 7.91-7.83 (m, 4H), 7.73-7.69 (m, 2H), 7.57-7.47 (m, 3H), 4.07 (t, $J = 7.6$ Hz, 1H), 2.43-2.32 (m, 1H), 2.15-2.04 (m, 1H), 1.10 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.1, 161.4, 135.4, 134.3, 133.4, 132.9, 130.4, 129.6, 128.6, 128.0, 127.7, 127.3, 126.3, 126.1, 125.71, 125.69, 124.4, 50.7, 27.2, 11.9; HRMS (ESI): m/z calcd for $\text{C}_{26}\text{H}_{20}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 410.1387, found: 410.1388.

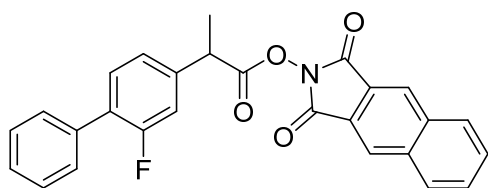
1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-(4-isobutylphenyl)propanoate (S18)



According to **General procedure 1** with ibuprofen (309.4 mg, 1.5 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (339.0 mg, 1.59 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S18** as a colorless solid (439.6 mg, 73% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.35 (s, 2H), 8.06-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.34 (d, $J = 8.4$ Hz, 2H), 7.18 (d, $J = 8.0$ Hz, 2H), 4.13 (q, $J = 7.1$ Hz, 1H), 2.49 (d, $J = 7.2$ Hz, 2H), 1.93-1.83 (m, 1H), 1.69 (d, $J = 7.2$ Hz, 3H), 0.92 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.8, 161.4, 141.2, 135.6, 135.4, 130.4, 129.62, 129.60, 127.3, 125.7, 124.4, 45.1, 42.7, 30.1, 22.4, 19.0; HRMS (ESI): m/z calcd for $\text{C}_{25}\text{H}_{24}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 402.1700, found: 402.1701.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (S19)



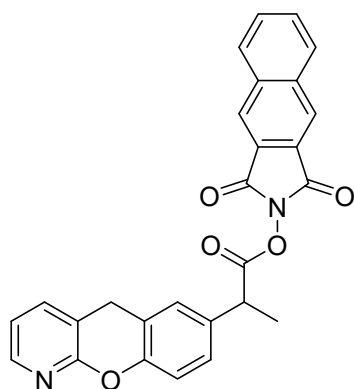
According to **General procedure 1** with flurbiprofen acid (488.5 mg, 2.0 mmol, 1.0

equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S19** as a colorless solid (536.1 mg, 61% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.37 (s, 2H), 8.08-8.05 (m, 2H), 7.75-7.71 (m, 2H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.51-7.44 (m, 3H), 7.40-7.36 (m, 1H), 7.32-7.29 (m, 2H), 4.20 (q, *J* = 7.2 Hz, 1H), 1.75 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.2, 161.3, 159.8 (d, *J* = 247.4 Hz), 139.5 (d, *J* = 7.7 Hz), 135.4, 135.3, 131.2 (d, *J* = 3.9 Hz), 130.4, 129.7, 129.0 (d, *J* = 2.9 Hz), 128.6, 128.4, 127.7, 125.8, 124.3, 123.6 (d, *J* = 3.4 Hz), 115.5 (d, *J* = 24.0 Hz), 42.5, 19.0; ¹⁹F NMR (376 MHz, CDCl₃) δ: -116.9; HRMS (ESI): *m/z* calcd for C₂₇H₁₉FNO₄ [M+H]⁺: 440.1293, found: 440.1288.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

2-(5*H*-chromeno[2,3-*b*]pyridin-7-yl)propanoate (**S20**)



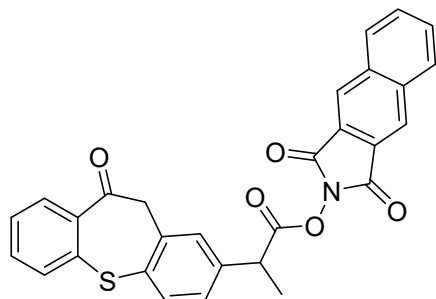
According to **General procedure 1** with pranoprofen (510.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/dichloromethane = 1/30) to yield the product **S20** as a colorless solid (504.5 mg, 56% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.36 (s, 2H), 8.18 (d, *J* = 4.0 Hz, 1H), 8.07-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.55 (d, *J* = 6.4 Hz, 1H), 7.29-7.27 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.06-7.03 (m, 1H), 4.16-4.10 (m, 3H), 1.69 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.6, 161.4, 158.3, 151.2, 146.7, 138.4, 135.4, 133.7, 130.4,

129.7, 127.7, 127.4, 125.8, 124.3, 119.9, 119.86, 117.6, 115.3, 42.3, 28.1, 19.1;
HRMS (ESI): m/z calcd for $C_{27}H_{19}N_2O_5$ $[M+H]^+$: 451.1288, found: 451.1285.

1,3-Dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl

2-((10-oxo-10,11-dihydrodibenzo[b,f]thiepin-2-yl)propanoate (S21)

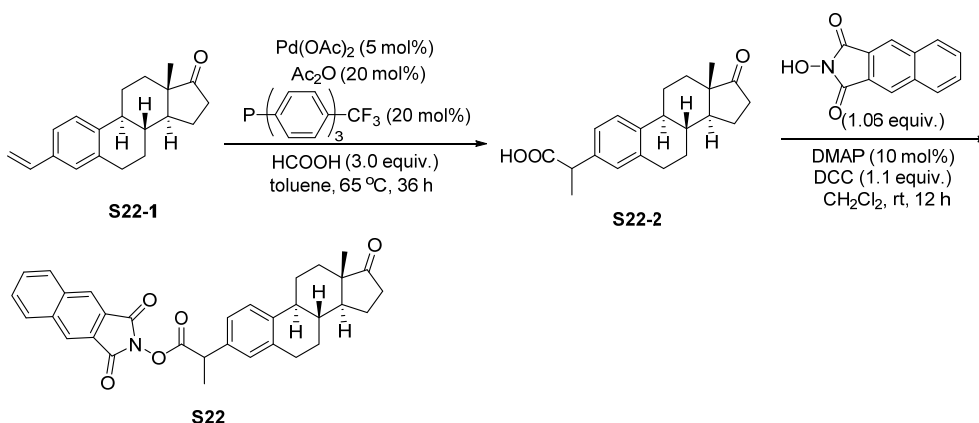


According to **General procedure 1** with zaltoprofen (596.7 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[f]isoindole-1,3(2H)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/dichloromethane = 1/50) to yield the product **S21** as a colorless solid (572.5 mg, 58% yield).

1H NMR (400 MHz, $CDCl_3$) δ : 8.25 (s, 2H), 8.17 (d, $J = 7.6$ Hz, 1H), 7.97-7.95 (m, 2H), 7.67-7.63 (m, 3H), 7.57-7.53 (m, 2H), 7.39 (td, $J = 7.4, 0.8$ Hz, 1H), 7.31-7.26 (m, 2H), 4.40-4.34 (m, 2H), 4.16 (q, $J = 7.2$ Hz, 1H), 1.67 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 191.0, 170.0, 161.1, 140.4, 139.8, 138.1, 135.9, 135.2, 133.9, 132.4, 131.6, 131.4, 130.7, 130.2, 129.5, 128.6, 126.7, 126.3, 125.6, 124.0, 50.9, 42.5, 18.9; HRMS (ESI): m/z calcd for $C_{29}H_{20}NO_5S$ $[M+H]^+$: 494.1057, found: 494.1057.

1,3-Dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl

2-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[α]phenanthren-3-yl)propanoate (S22)



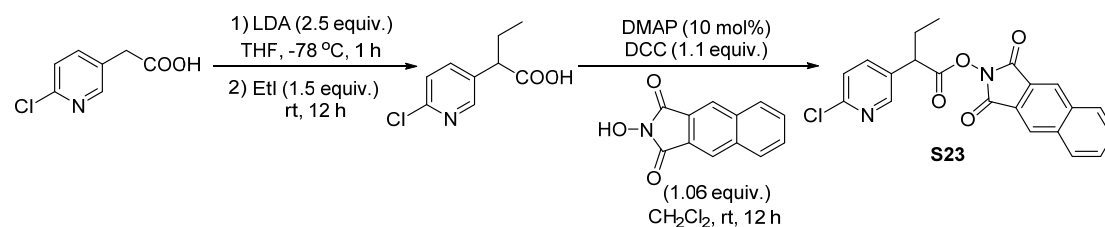
To a mixture of Pd(OAc)₂ (56 mg, 0.25 mmol, 5 mol%), tri[4-(trifluoromethyl)phenyl]phosphane (466 mg, 1.0 mmol, 0.2 equiv.), and toluene (5.0 mL) in a vial (50.0 mL) were added (8*R*,9*S*,13*S*,14*S*)-13-methyl-3-vinyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[α]phenanthren-17-one **S22-1** (1.40 g, 5.0 mmol, 1.0 equiv.) which was synthesized according to the literature^[2], HCOOH (0.69 g, 15 mmol, 3.0 equiv.), and Ac₂O (0.102 g, 1.0 mmol, 0.2 equiv.). The vial was purged with argon to remove the air and tightly sealed with a septum cap. The reaction mixture was stirred at 65 °C for 48 h, and then cooled to room temperature. The reaction mixture was diluted with CH₂Cl₂ (30 mL), and transferred into a separatory funnel, followed by the addition of 1 M NaOH (30 mL). The mixture was washed with CH₂Cl₂ (3 × 20 mL) with vigorous shaking. The aqueous layer was acidified with 3 M HCl (15 mL) and extracted with CH₂Cl₂ (3 × 30 mL). The combined organic layers were dried over Na₂SO₄ and the organic solvent was removed by rotary evaporator under vacuum. The residue was purified by flash column chromatography on silica gel to give the product **S22-2** (0.65 g, 40% yield).^[3]

According to **General procedure 1** with 2-((8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[α]phenanthren-3-yl)propanoic acid **S22-2** (650.0 mg, 2.0 mmol, 1.2 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (360.0 mg, 1.7 mmol, 1.0 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/10) to yield the product **S22** as a pale yellow colorless solid (720.0 mg, 69% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.36 (s, 2H), 8.12-7.99 (m, 2H), 7.78-7.68 (m, 2H), 7.33 (d, $J = 8.1$ Hz, 1H), 7.21 (d, $J = 8.4$ Hz, 1H), 7.17 (s, 1H), 4.10 (q, $J = 7.4$ Hz, 1H), 3.04-2.89 (m, 3H), 2.51 (dd, $J = 18.7, 8.6$ Hz, 1H), 2.48-2.39 (m, 1H), 2.37-2.26 (m, 1H), 2.23-1.91 (m, 5H), 1.68 (d, $J = 7.3$ Hz, 4H), 1.57-1.40 (m, 3H), 0.92 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 220.9, 170.7, 161.4, 139.3, 137.1, 135.8, 135.4, 130.4, 129.7, 128.2, 128.1, 125.9, 125.7, 125.0, 124.8, 124.4, 50.5, 48.0, 44.3, 42.6, 38.0, 35.8, 31.6, 29.4, 26.5, 25.6, 21.6, 19.1, 13.8; HRMS (ESI) m/z calcd. for $\text{C}_{33}\text{H}_{32}\text{NO}_5$ $[\text{M} + \text{H}]^+$ 522.2275, found 522.2272.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

2-(6-chloropyridin-3-yl)butanoate (**S23**)

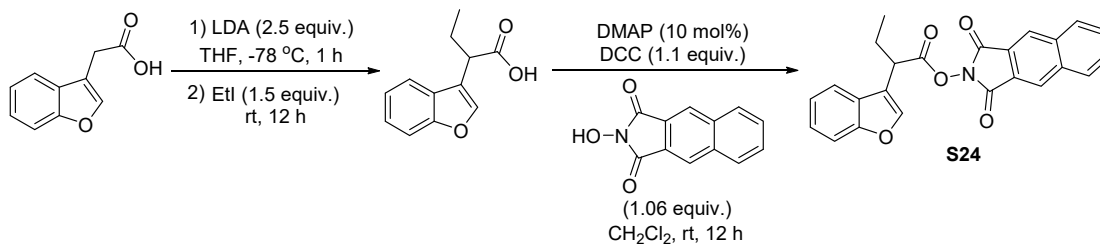


The synthetic procedure of 2-(6-chloropyridin-3-yl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(6-chloropyridin-3-yl)butanoic acid (399.2 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 2/1) to yield the product **S23** as a colorless solid (497.5 mg, 63% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.42 (d, $J = 2.4$ Hz, 1H), 8.36 (s, 2H), 8.07-8.04 (m, 2H), 7.78 (dd, $J = 8.4, 2.8$ Hz, 1H), 7.75-7.71 (m, 2H), 7.39 (d, $J = 8.4$ Hz, 1H), 3.91 (t, $J = 7.6$ Hz, 1H), 2.35-2.24 (m, 1H), 2.03-1.92 (m, 1H), 1.07 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.3, 161.2, 151.2, 149.5, 138.1, 135.4, 131.6, 130.4, 129.8, 125.9, 124.6, 124.2, 47.3, 27.3, 11.7; HRMS (ESI): m/z calcd for $\text{C}_{21}\text{H}_{16}\text{ClN}_2\text{O}_4$ $[\text{M} + \text{H}]^+$: 395.0793, found: 395.0791.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(benzofuran-3-yl)butanoate (**S24**)



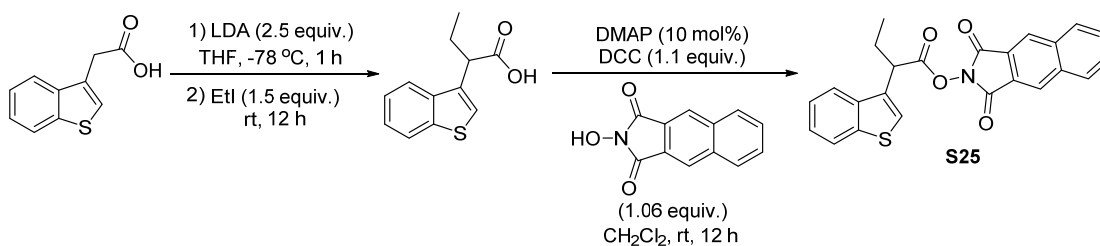
The synthetic procedure of 2-(benzofuran-3-yl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(benzofuran-3-yl)butanoic acid (408.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S24** as a colorless solid (519.2 mg, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.36 (s, 2H), 8.07-8.05 (m, 2H), 7.77-7.71 (m, 4H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.36-7.28 (m, 2H), 4.16 (t, *J* = 7.2 Hz, 1H), 2.38-2.18 (m, 2H), 1.17 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 169.6, 161.4, 155.4, 142.8, 135.4, 130.4, 129.7, 126.5, 125.8, 124.7, 124.3, 122.8, 120.0, 116.3, 111.7, 41.0, 25.9, 11.8; HRMS (ESI): *m/z* calcd for C₂₄H₁₈NO₅ [M+H]⁺: 400.1179, found: 400.1178.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

2-(benzo[*b*]thiophen-3-yl)butanoate (**S25**)



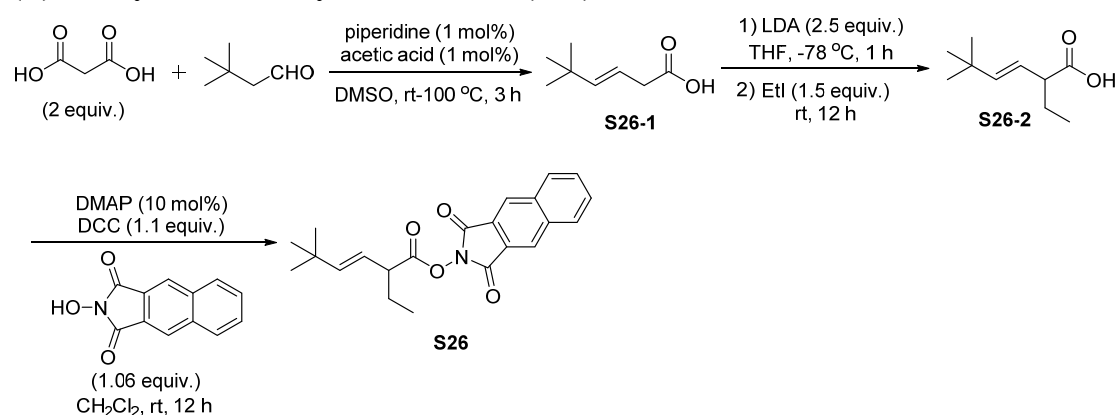
The synthetic procedure of 2-(benzo[*b*]thiophen-3-yl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(benzo[*b*]thiophen-3-yl)butanoic acid (440.6 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S25** as a colorless solid (523.5 mg, 63% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.35 (s, 2H), 8.06-8.03 (m, 2H), 7.92-7.89 (m, 2H), 7.74-7.70 (m, 2H), 7.58 (s, 1H), 7.49-7.45 (m, 1H), 7.43-7.38 (m, 1H), 4.39-4.35 (m, 1H), 2.40-2.29 (m, 1H), 2.25-2.15 (m, 1H), 1.18 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.6, 161.4, 140.3, 137.8, 135.4, 131.0, 130.4, 129.7, 125.8, 124.6, 124.34, 124.3, 124.2, 123.0, 121.5, 44.2, 26.4, 12.0; HRMS (ESI): m/z calcd for $\text{C}_{24}\text{H}_{18}\text{NO}_4\text{S}$ $[\text{M}+\text{H}]^+$: 416.0951, found: 416.0950.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl

(*E*)-2-ethyl-5,5-dimethylhex-3-enoate (**S26**)



Piperidine (25 μL , 0.25 mmol, 1 mol%) and acetic acid (15 μL , 0.25 mmol, 1 mol%) were added to a stirred solution of malonic acid (5 g, 50 mmol, 2 equiv.) and 3,3-dimethylbutanal (2.5 g, 25 mmol, 1 equiv.) in DMSO (20 mL) at room temperature. The reaction mixture was stirred for 20 min at room temperature and then was heated for 2 h at 100 °C. The reaction mixture was cooled to room temperature and water (60 mL) was added. Subsequently, the reaction mixture was extracted with diethyl ether (3 \times 30 mL). The combined organic layers were washed with saturated brine (2 \times 30 mL) and dried with over anhydrous MgSO_4 . The organic solvent was removed by rotary evaporator under vacuum to afford the crude product **S26-1** without further purification.

The synthetic procedure of (*E*)-2-ethyl-5,5-dimethylhex-3-enoic acid **S26-2** is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

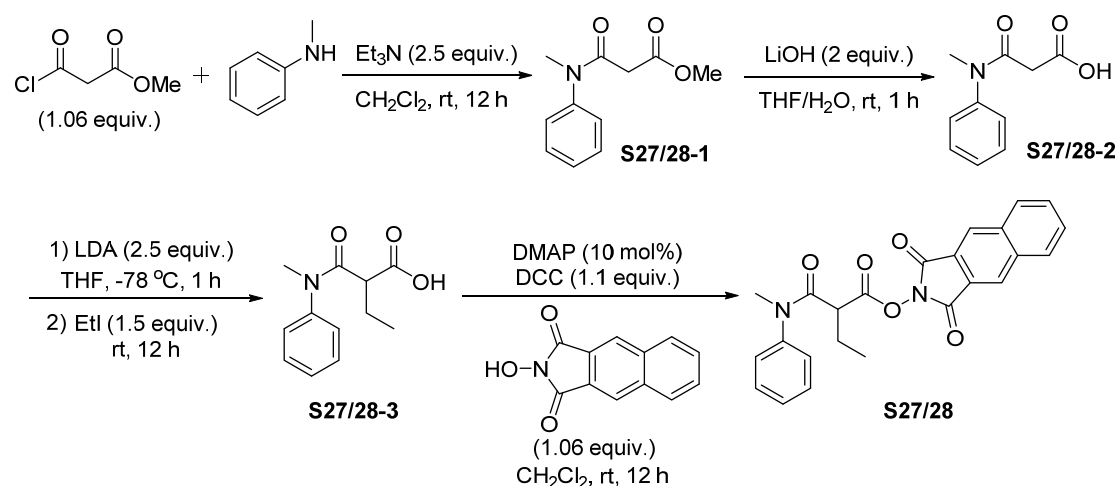
According to **General procedure 1** with (*E*)-2-ethyl-5,5-dimethylhex-3-enoic acid **S26-2** (510.6 mg, 3.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[*f*]isoindole-1,3(2H)-dione (678.0 mg, 3.18 mmol, 1.06 equiv.)

for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S26** as a colorless solid (745.5 mg, 68% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.36 (s, 2H), 8.07-8.04 (m, 2H), 7.74-7.70 (m, 2H), 5.78 (d, $J = 15.6$ Hz, 1H), 5.43 (dd, $J = 15.6, 8.4$ Hz, 1H), 3.27 (q, $J = 7.6$ Hz, 1H), 2.04-1.93 (m, 1H), 1.80-1.69 (m, 1H), 1.07-1.04 (m, 12H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.6, 161.5, 146.3, 135.4, 130.4, 129.6, 125.7, 124.5, 120.2, 47.9, 33.3, 29.4, 26.2, 11.4; HRMS (ESI): m/z calcd for $\text{C}_{22}\text{H}_{24}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 366.1700, found: 366.1697.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

2-(methyl(phenyl)carbamoyl)butanoate (**S27/28**)



To a stirred solution of *N*-methylaniline (2.14 g, 20 mmol, 1 equiv.) and Et_3N (5.06 g, 50 mmol, 2.5 equiv.) in CH_2Cl_2 (80 mL) was added dropwise methyl 3-chloro-3-oxopropanoate (2.9 g, 21.2 mmol, 1.06 equiv.) at 0°C . The reaction mixture was stirred for 12 h at room temperature and then was extracted with ethyl acetate (3×50 mL). The combined organic layers were washed with saturated brine (2×30 mL) and dried with over anhydrous MgSO_4 . The organic solvent was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 1/2) to afford **S27/28-1** (3.15 g, 76% yield).

To a stirred solution of methyl 3-(methyl(phenyl)amino)-3-oxopropanoate **S27/28-1** (1.04 g, 5 mmol, 1 equiv.) in $\text{THF}/\text{H}_2\text{O}$ (1/1 mL) was added LiOH (240 mg, 10 mmol,

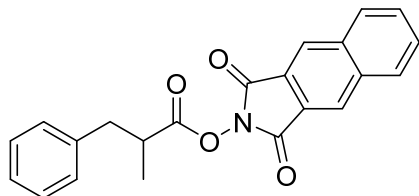
2 equiv.) at room temperature. The reaction mixture was stirred for 1 h at room temperature and then was extracted with ethyl acetate (3 × 20 mL). The combined organic layers were washed with saturated brine (2 × 10 mL) and dried with over anhydrous MgSO₄. The organic solvent was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography on silica gel (ethyl acetate/dichloromethane = 1/2) to afford **S27/28-2** (656.9 mg, 68% yield).

The synthetic procedure of 2-(methyl(phenyl)carbamoyl)butanoic acid **S27/28-3** is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(methyl(phenyl)carbamoyl)butanoic acid **S27/28-3** (553.0 mg, 2.5 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (565.0 mg, 2.65 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/dichloromethane = 1/50) to yield the product **S27/28** as a pale yellow solid (739.2 mg, 71% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.35 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.42 (t, *J* = 7.4 Hz, 1H), 7.36 (d, *J* = 7.2 Hz, 2H), 3.61-3.58 (m, 1H), 3.39 (s, 3H), 2.22-2.13 (m, 1H), 2.10-2.01 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 166.3, 161.1, 142.9, 135.4, 130.4, 130.3, 129.7, 128.5, 127.7, 125.7, 124.3, 48.4, 38.0, 23.7, 11.8; HRMS (ESI): *m/z* calcd for C₂₄H₂₁N₂O₅ [M+H]⁺: 417.1445, found: 417.1441.

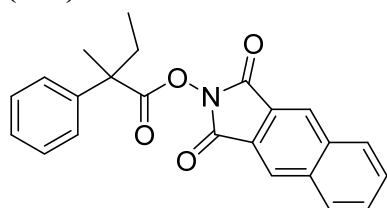
1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-methyl-3-phenylpropanoate (**S29**)



According to **General procedure 1** with 2-methyl-3-phenylpropanoic acid (328.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S29** as a colorless solid (524.7 mg, 73% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.37 (s, 2H), 8.08-8.04 (m, 2H), 7.75-7.71 (m, 2H), 7.37-7.33 (m, 2H), 7.29-7.25 (m, 3H), 3.30 (dd, $J = 13.6, 6.0$ Hz, 1H), 3.19-3.11 (m, 1H), 2.85 (dd, $J = 13.6, 8.4$ Hz, 1H), 1.36 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 172.0, 161.5, 138.0, 135.4, 130.4, 129.6, 129.2, 128.6, 126.7, 125.7, 124.4, 39.2, 38.9, 16.3; HRMS (ESI): m/z calcd for $\text{C}_{22}\text{H}_{18}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 360.1230, found: 360.1228.

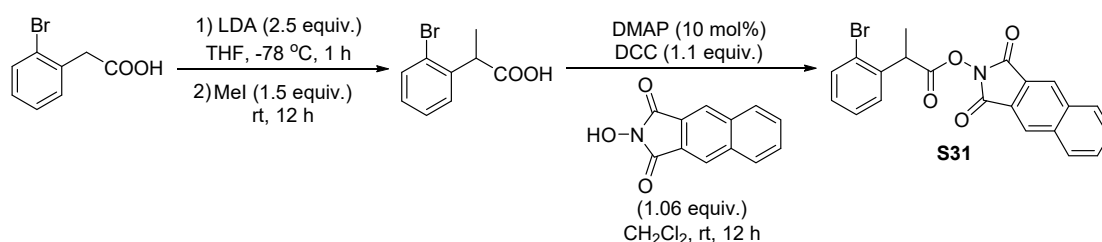
1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-methyl-2-phenylbutanoate (S30)



According to **General procedure 1** with 2-methyl-2-phenylbutanoic acid (445.6 mg, 2.5 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[*f*]isoindole-1,3(2*H*)-dione (565.0 mg, 2.65 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S30** as a colorless solid (653.5 mg, 70% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.35 (s, 2H), 8.08-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.51 (d, $J = 7.6$ Hz, 2H), 7.44 (t, $J = 7.6$ Hz, 2H), 7.33 (t, $J = 7.2$ Hz, 1H), 2.36-2.27 (m, 1H), 2.18-2.09 (m, 1H), 1.76 (s, 3H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 172.6, 161.5, 141.9, 135.4, 130.4, 129.6, 128.6, 127.3, 126.3, 125.6, 124.5, 50.7, 32.2, 23.3, 9.0; HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{20}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 374.1387, found: 374.1387.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-(2-bromophenyl)propanoate (S31)

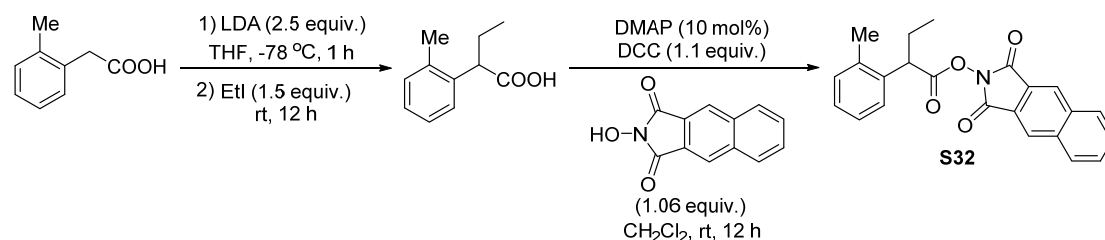


The synthetic procedure of 2-(2-bromophenyl)propanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(2-bromophenyl)propanoic acid (458.0 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S31** as a colorless solid (568.5 mg, 67% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.36 (s, 2H), 8.07-8.05 (m, 2H), 7.74-7.71 (m, 2H), 7.63 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.52 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.41 (td, *J* = 7.6, 1.2 Hz, 1H), 7.20 (td, *J* = 7.6, 1.6 Hz, 1H), 4.69 (q, *J* = 7.2 Hz, 1H), 1.67 (d, *J* = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.2, 161.3, 138.1, 135.4, 133.1, 130.4, 129.7, 129.3, 128.6, 128.1, 125.8, 124.3, 124.1, 42.6, 18.0; HRMS (ESI): *m/z* calcd for C₂₁H₁₅BrNO₄ [M+H]⁺: 424.0179, found: 424.0178.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(*o*-tolyl)butanoate (**S32**)



The synthetic procedure of 2-(*o*-tolyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

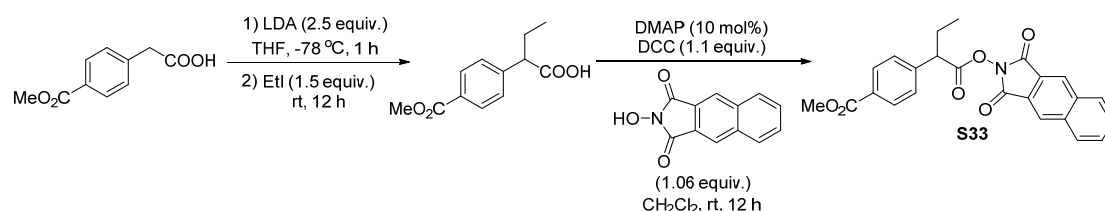
According to **General procedure 1** with 2-(*o*-tolyl)butanoic acid (356.3 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S32** as a colorless solid (522.8 mg, 70% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.33 (s, 2H), 8.05-8.02 (m, 2H), 7.73-7.69 (m, 2H), 7.41 (d, *J* = 7.2 Hz, 1H), 7.29-7.22 (m, 3H), 4.15 (t, *J* = 7.4 Hz, 1H), 2.48 (s, 3H), 2.34-2.23 (m, 1H), 2.02-1.91 (m, 1H), 1.08 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.4, 161.4, 136.2, 135.4, 135.3, 130.7, 130.4, 129.6, 127.6, 127.2, 126.6, 125.7, 124.4, 46.2, 26.5, 19.8, 12.0; HRMS (ESI): *m/z* calcd for C₂₃H₂₀NO₄ [M+H]⁺:

374.1387, found: 374.1385.

Methyl

4-(1-((1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl)oxy)-1-oxobutan-2-yl)benzoate (S33)

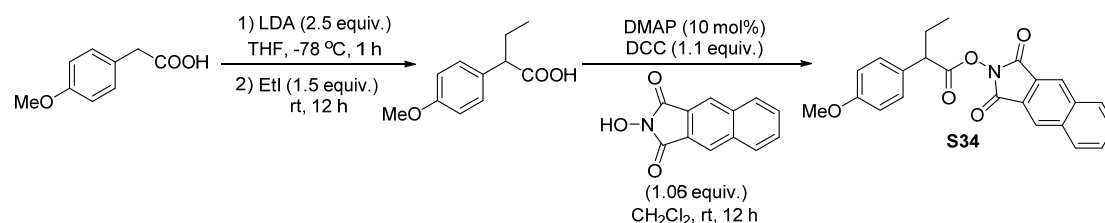


The synthetic procedure of 2-(4-(methoxycarbonyl)phenyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound S7).

According to **General procedure 1** with 2-(4-(methoxycarbonyl)phenyl)butanoic acid (444.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product S33 as a colorless solid (542.6 mg, 65% yield).

¹H NMR (400 MHz, CDCl₃) δ : 8.35 (s, 2H), 8.10-8.03 (m, 4H), 7.74-7.70 (m, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 3.97-3.92 (m, 4H), 2.35-2.24 (m, 1H), 2.05-1.94 (m, 1H), 1.06 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 169.6, 166.7, 161.3, 141.9, 135.4, 130.4, 130.1, 129.74, 129.69, 128.2, 125.8, 124.3, 52.1, 50.4, 27.2, 11.8; HRMS (ESI): *m/z* calcd for C₂₄H₂₀NO₆ [M+H]⁺: 418.1285, found: 418.1284.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-methoxyphenyl)butanoate (S34)



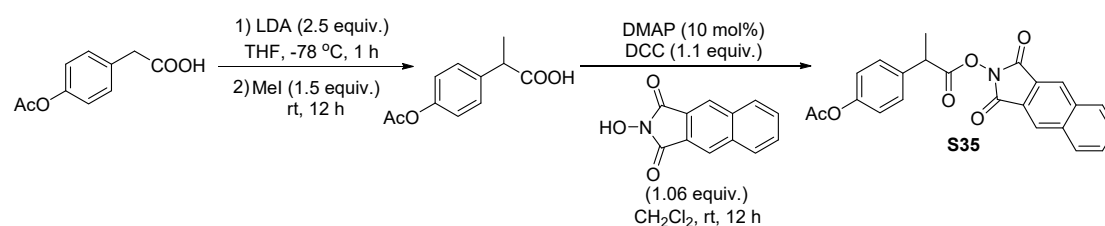
The synthetic procedure of 2-(4-methoxyphenyl)butanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound S7).

According to **General procedure 1** with 2-(4-methoxyphenyl)butanoic acid (388.5

mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S34** as a colorless solid (584.1 mg, 75% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.34 (s, 2H), 8.07-8.03 (m, 2H), 7.74-7.70 (m, 2H), 7.35-7.32 (m, 2H), 6.95-6.91 (m, 2H), 3.86-3.82 (m, 4H), 2.30-2.19 (m, 1H), 2.01-1.90 (m, 1H), 1.05 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.3, 161.4, 159.2, 135.4, 130.4, 129.6, 129.1, 128.9, 125.7, 124.4, 114.2, 55.2, 49.7, 27.2, 11.9; HRMS (ESI): *m/z* calcd for C₂₃H₂₀NO₅ [M+H]⁺: 390.1336, found: 390.1333.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-acetoxyphenyl)propanoate (**S35**)



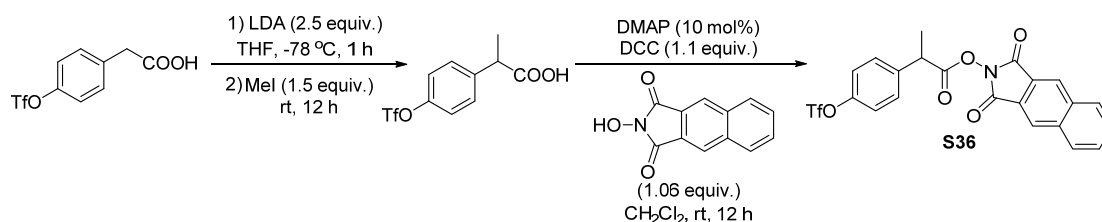
The synthetic procedure of 2-(4-acetoxyphenyl)propanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 2-(4-acetoxyphenyl)propanoic acid (416.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S35** as a colorless solid (492.1 mg, 61% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.34 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.71 (m, 2H), 7.45 (d, *J* = 8.8 Hz, 2H), 7.14 (d, *J* = 8.8 Hz, 2H), 4.16 (q, *J* = 7.2 Hz, 1H), 2.31 (s, 3H), 1.70 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.4, 169.3, 161.3, 150.2, 135.8, 135.4, 130.4, 129.7, 128.7, 125.7, 124.3, 122.0, 42.5, 21.1, 19.1; HRMS (ESI): *m/z* calcd for C₂₃H₁₈NO₆ [M+H]⁺: 404.1129, found: 404.1125.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

2-(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)propanoate (S36)

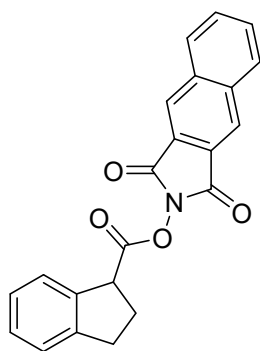


The synthetic procedure of 2-(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)propanoic acid is the same as that of 5-chloro-2-phenylpentanoic acid (see compound S7).

According to **General procedure 1** with 2-(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)propanoic acid (596.5 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **S36** as a colorless solid (542.8 mg, 55% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.36 (s, 2H), 8.07-8.05 (m, 2H), 7.75-7.72 (m, 2H), 7.54 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 8.8 Hz, 2H), 4.20 (q, *J* = 7.2 Hz, 1H), 1.71 (d, *J* = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 170.0, 161.3, 149.0, 138.9, 135.4, 130.4, 129.7, 129.6, 125.8, 124.2, 121.8, 118.7 (q, *J* = 318.8 Hz), 42.4, 19.0; ¹⁹F NMR (376 MHz, CDCl₃) δ: -72.8; HRMS (ESI): *m/z* calcd for C₂₂H₁₅F₃NO₇S [M+H]⁺: 494.0516, found: 494.0516.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2,3-dihydro-1*H*-indene-1-carboxylate (S37)



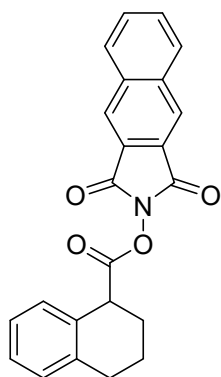
According to **General procedure 1** with 2,3-dihydro-1*H*-indene-1-carboxylic acid (324.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione

(452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S37** as a colorless solid (464.6 mg, 65% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.37 (s, 2H), 8.07-8.05 (m, 2H), 7.74-7.72 (m, 2H), 7.68-7.66 (m, 1H), 7.32-7.29 (m, 3H), 4.46 (t, $J = 7.8$ Hz, 1H), 3.22-3.15 (m, 1H), 3.07-2.99 (m, 1H), 2.65-2.58 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.2, 161.5, 143.9, 138.7, 135.4, 130.4, 129.7, 128.2, 126.9, 125.8, 125.1, 124.7, 124.4, 47.5, 31.7, 29.3; HRMS (ESI): m/z calcd for $\text{C}_{22}\text{H}_{16}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 358.1074, found: 358.1072.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl

1,2,3,4-tetrahydronaphthalene-1-carboxylate (**S38**)

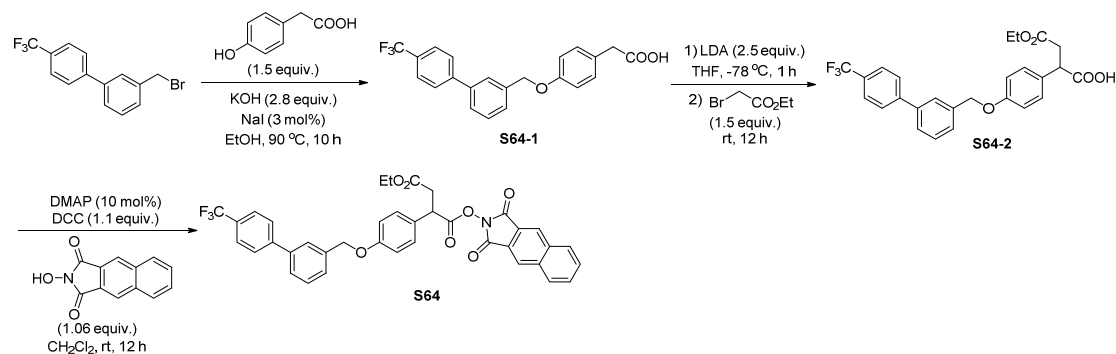


According to **General procedure 1** with 1,2,3,4-tetrahydronaphthalene-1-carboxylic acid (352.4 mg, 2.0 mmol, 1.0 equiv.) and 2-hydroxy-1H-benzo[*f*]isoindole-1,3(2H)-dione (452.0 mg, 2.12 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **S38** as a colorless solid (445.7 mg, 60% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.35 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.70 (m, 2H), 7.44-7.41 (m, 1H), 7.25-7.20 (m, 2H), 7.15-7.13 (m, 1H), 4.25 (t, $J = 6.4$ Hz, 1H), 2.95-2.78 (m, 2H), 2.41-2.25 (m, 2H), 2.15-2.06 (m, 1H), 1.93-1.84 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 171.0, 161.5, 137.2, 135.4, 131.1, 130.4, 129.6, 129.5, 127.5, 126.2, 125.7, 124.4, 42.6, 28.9, 27.1, 20.5; HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{18}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 372.1230, found: 372.1228.

1-(1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl)

4-ethyl

2-(4-((4'-(trifluoromethyl)-[1,1'-biphenyl]-3-yl)methoxy)phenyl)succinate (**S64**)

To a stirred solution of 2-(4-hydroxyphenyl)acetic acid (228.2 mg, 1.5 mmol, 1.5 equiv.), KOH (160 mg, 2.8 mmol, 2.8 equiv.) and NaI (4.5 mg, 0.03 mmol, 3 mol%) in EtOH (10 mL) was added dropwise the solution of 3-(bromomethyl)-4'-(trifluoromethyl)-1,1'-biphenyl (315 mg, 1 mmol, 1 equiv.) in EtOH (10 mL). The reaction mixture was stirred at 90 °C for 10 h and then the pH of the reaction mixture was adjusted to 1~2 with 3.0 M HCl. The aqueous phase was extracted with CH₂Cl₂ (3 × 10 mL). The organic solvent was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography (ethyl acetate/dichloromethane = 1/2) on silica gel to afford the desired product **S64-1** (355.5 mg, 92% yield).

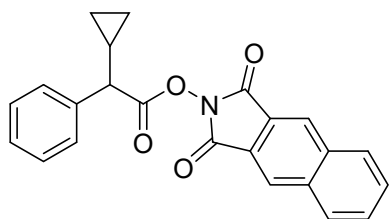
The synthetic procedure of 2-(4-((4'-(trifluoromethyl)-[1,1'-biphenyl]-3-yl)methoxy)phenyl)acetic acid **S64-2** (colorless oil, 84 mg, 46% yield) is the same as that of 5-chloro-2-phenylpentanoic acid (see compound **S7**).

According to **General procedure 1** with 4-ethoxy-4-oxo-2-(4-((4'-(trifluoromethyl)-[1,1'-biphenyl]-3-yl)methoxy)phenyl)butanoic acid **S64-2** (68.4 mg, 0.15 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (33 mg, 0.16 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 4/1) to yield the product **S64** as a colorless solid (49.3 mg, 51% yield).

¹H NMR (400 MHz, CDCl₃) δ: 8.34 (s, 2H), 8.06-8.04 (m, 2H), 7.74-7.68 (m, 7H), 7.58-7.56 (m, 1H), 7.53-7.45 (m, 2H), 7.38 (d, *J* = 8.4 Hz, 2H), 7.05 (d, *J* = 8.4 Hz,

2H), 5.15 (s, 2H), 4.53-4.49 (m, 1H), 4.23-4.16 (m, 2H), 3.29-3.23 (m, 1H), 2.89-2.83 (m, 1H), 1.26 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.3, 169.5, 161.2, 158.6, 144.4, 140.1, 137.7, 135.4, 130.4, 129.7, 129.3, 129.2, 128.1, 127.5, 127.2, 126.9, 126.3, 125.7 (q, $J = 4.6$ Hz), 124.3, 122.9 (q, $J = 271.3$ Hz), 115.4, 69.8, 61.2, 44.2, 38.3, 14.1; ^{19}F NMR (376 MHz, CDCl_3) δ : -62.4; HRMS (ESI): m/z calcd for $\text{C}_{38}\text{H}_{28}\text{F}_3\text{NO}_7\text{Na}$ $[\text{M}+\text{Na}]^+$: 690.1710, found: 690.1707.

1,3-Dioxo-1,3-dihydro-2H-benzo[*f*]isoindol-2-yl 2-cyclopropyl-2-phenylacetate (1c)

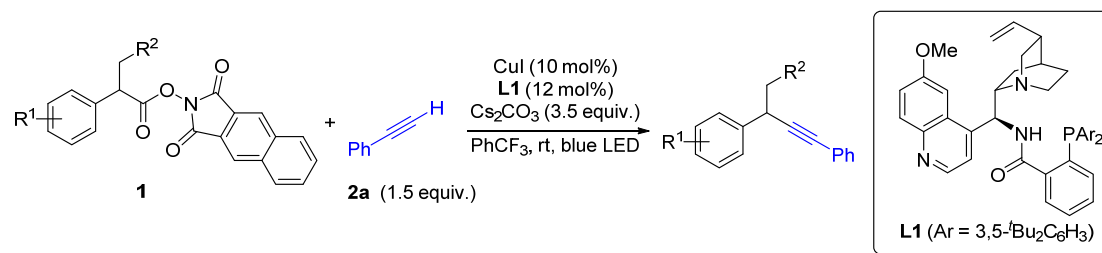


According to **General procedure 1** with 2-cyclopropyl-2-phenylacetic acid which was synthesized according to reference^[4] (529.0 mg, 3.0 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (678.0 mg, 3.18 mmol, 1.06 equiv.) for 12 h, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1) to yield the product **1c** as a colorless solid (791.1 mg, 71% yield).

^1H NMR (400 MHz, CDCl_3) δ : 8.36 (s, 2H), 8.07-8.05 (m, 2H), 7.74-7.71 (m, 2H), 7.50 (d, $J = 7.2$ Hz, 2H), 7.42 (t, $J = 7.4$ Hz, 2H), 7.35 (t, $J = 7.2$ Hz, 1H), 3.33 (d, $J = 9.6$ Hz, 1H), 1.58-1.51 (m, 1H), 0.88-0.81 (m, 1H), 0.74-0.59 (m, 2H), 0.43-0.37 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.7, 161.4, 136.7, 135.4, 130.4, 129.6, 128.8, 128.0, 127.9, 125.7, 124.4, 53.3, 14.5, 4.8; HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{18}\text{NO}_4$ $[\text{M}+\text{H}]^+$: 372.1230, found: 372.1227.

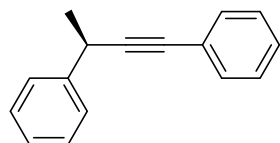
7. Asymmetric decarboxylative alkylation: Scope of NHP-type esters

General procedure 2:



CuI (3.8 mg, 0.02 mmol, 10 mol%), L1 (20.1 mg, 0.024 mmol, 12 mol%), Cs₂CO₃ (228.1 mg, 0.70 mmol, 3.5 equiv.), *N*-hydroxyphthalimide esters **1** (0.20 mmol, 1.0 equiv.) and anhydrous PhCF₃ (4.0 mL) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere. Then, ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) was added to the mixture. The reaction mixture was stirred under the irradiation of a 24 W blue LED for 3 to 4 d at room temperature. After the completion of reaction, the reaction mixture was filtered and the precipitate was washed by EtOAc. The filtrate was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography on silica gel to afford the alkylation product.

(*R*)-But-1-yne-1,3-diylidibenzene (**3**)



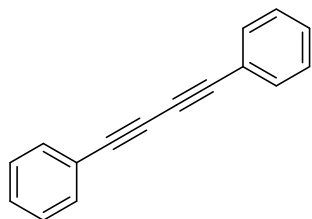
According to General procedure 2 with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylpropanoate **1ah** (69.1 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **3** as a colorless oil (27.6 mg, 67% yield, 93% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 97/3, flow rate 0.8 mL/min, λ = 254 nm), t_R (minor) = 11.49 min, t_R (major) = 17.02 min.

¹H NMR (400 MHz, CDCl₃) δ : 7.46-7.43 (m, 4H), 7.36-7.32 (m, 2H), 7.30-7.22 (m, 4H), 3.98 (q, J = 7.2 Hz, 1H), 1.58 (d, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃)

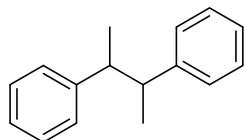
δ : 143.3, 131.6, 128.5, 128.2, 127.7, 126.9, 126.6, 123.7, 92.6, 82.4, 32.5, 24.5. The NMR spectra were in accord with that reported in literature.^[5]

1,4-Diphenylbuta-1,3-diyne (3')



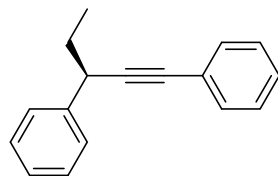
Colorless solid. ¹H NMR (400 MHz, CDCl₃) δ : 7.54-7.52 (m, 4H), 7.40-7.32 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ : 132.5, 129.2, 128.4, 121.8, 81.5, 73.9. The NMR spectra were in accord with that reported in literature.^[6]

Butane-2,3-diylidibenzene (3'')



Colorless solid. ¹H NMR (400 MHz, CDCl₃) δ : 7.32-7.29 (m, 2H), 7.21 (d, $J = 7.2$ Hz, 3H), 7.17-7.14 (m, 2H), 7.10-7.06 (m, 1H), 7.01-6.99 (m, 2H), 2.96-2.91 (m, 1H), 2.81-2.78 (m, 1H), 1.27 (d, $J = 6.8$ Hz, 3H), 1.02 (d, $J = 6.0$ Hz, 3H). The NMR spectra were in accord with that reported in literature.^[7]

(R)-Pent-1-yne-1,3-diylidibenzene (4)



According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (71.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **4** as a colorless oil (33.9 mg, 77% yield, 97% *ee*).

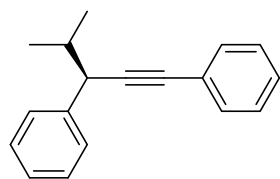
HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 1.0 mL/min, $\lambda =$

254 nm), t_R (minor) = 11.58 min, t_R (major) = 17.07 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.46-7.41 (m, 4H), 7.35-7.22 (m, 6H), 3.79 (t, J = 7.0 Hz, 1H), 1.90-1.82 (m, 2H), 1.06 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 142.0, 131.6, 128.4, 128.2, 127.7, 127.5, 126.6, 123.8, 91.4, 83.4, 39.9, 31.7, 11.8.

The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-(4-Methylpent-1-yne-1,3-diyl)dibenzene (**5**)

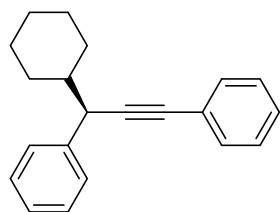


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 3-methyl-2-phenylbutanoate **S5** (74.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3.5 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **5** as a colorless oil (29.1 mg, 62% yield, 99% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 8.39 min, t_R (major) = 9.02 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.47-7.45 (m, 2H), 7.40 (d, J = 7.2 Hz, 2H), 7.34-7.22 (m, 6H), 3.73 (d, J = 6.0 Hz, 1H), 2.09-2.01 (m, 1H), 1.04 (d, J = 6.4 Hz, 3H), 0.99 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.0, 131.6, 128.19, 128.16, 127.7, 126.6, 123.9, 90.2, 84.2, 45.7, 35.2, 21.3, 18.7. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-(3-Cyclohexylprop-1-yne-1,3-diyl)dibenzene (**6**)



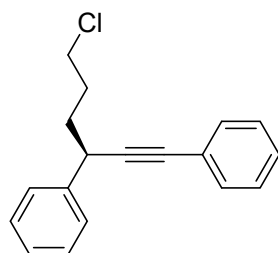
According to **General procedure 2** with

1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-cyclohexyl-2-phenylacetate **S6** (82.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3.5 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **6** as a colorless oil (35.7 mg, 65% yield, 99% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 9.88 min, t_R (major) = 11.73 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.46-7.44 (m, 2H), 7.38 (d, J = 7.2 Hz, 2H), 7.34-7.22 (m, 6H), 3.69 (d, J = 6.4 Hz, 1H), 1.88-1.86 (m, 1H), 1.73-1.65 (m, 5H), 1.21-1.14 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.8, 131.6, 128.3, 128.2, 128.1, 127.6, 126.5, 124.0, 90.8, 84.0, 45.1, 44.6, 31.7, 29.5, 26.4, 26.3, 26.2. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-(6-Chlorohex-1-yne-1,3-diyl)dibenzene (7)

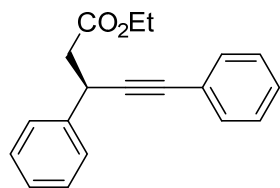


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 5-chloro-2-phenylpentanoate **S7** (81.6 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **7** as a colorless oil (33.3 mg, 62% yield, 96% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99/1, flow rate 0.7 mL/min, λ = 254 nm), t_R (major) = 15.48 min, t_R (minor) = 20.72 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.46-7.42 (m, 4H), 7.37-7.24 (m, 6H), 3.89 (s, 1H), 3.58 (s, 2H), 2.03-1.96 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.5, 131.6, 128.6, 128.2, 127.9, 127.4, 126.9, 123.5, 90.6, 83.8, 44.7, 37.7, 35.7, 30.3; HRMS (ESI): m/z calcd for $\text{C}_{18}\text{H}_{18}\text{Cl}$ [$\text{M}+\text{H}$] $^+$: 269.1092, found: 269.1091.

Ethyl (*R*)-3,5-diphenylpent-4-ynoate (**8**)

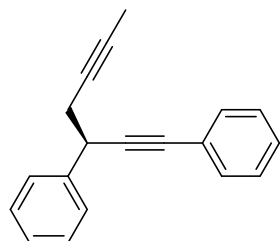


According to **General procedure 2** with 1-(1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl) 4-ethyl 2-phenylsuccinate **S8** (83.5 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **8** as a colorless oil (17.8 mg, 32% yield, 93% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 97/3, flow rate 0.7 mL/min, λ = 240 nm), t_R (major) = 8.01 min, t_R (minor) = 12.96 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.47-7.41 (m, 4H), 7.35 (t, J = 7.6 Hz, 2H), 7.29-7.25 (m, 4H), 4.38 (t, J = 7.6 Hz, 1H), 4.18-4.13 (m, 2H), 2.90 (dd, J = 15.2, 8.4 Hz, 1H), 2.80 (dd, J = 15.2, 7.2 Hz, 1H), 1.23 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.9, 140.5, 131.7, 128.7, 128.2, 128.0, 127.5, 127.2, 123.3, 89.8, 83.5, 60.7, 43.4, 34.8, 14.2. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-Hepta-1,5-diyne-1,3-diylidibenzene (**9**)

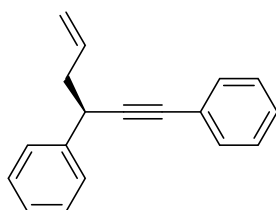


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylhex-4-ynoate **S9** (76.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **9** as a colorless oil (24.9 mg, 51% yield, 90% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99/1, flow rate 0.7 mL/min, λ = 254 nm), t_R (major) = 6.53 min, t_R (minor) = 7.43 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.47-7.46 (m, 4H), 7.35 (t, J = 7.4 Hz, 2H), 7.30-7.25 (m, 4H), 4.01 (t, J = 7.0 Hz, 1H), 2.75-2.61 (m, 2H), 1.78 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.7, 131.7, 128.4, 128.2, 127.9, 127.6, 127.1, 123.5, 90.5, 83.6, 77.9, 76.3, 38.4, 28.8, 3.5; HRMS (ESI): m/z calcd for $\text{C}_{19}\text{H}_{17}$ $[\text{M}+\text{H}]^+$: 245.1325, found: 245.1323.

(*R*)-Hex-5-en-1-yne-1,3-diylidibenzene (10)

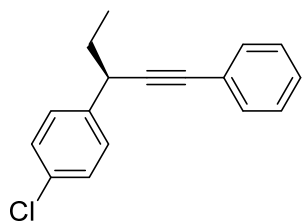


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylpent-4-enoate **S10** (74.3 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **10** as a colorless oil (24.6 mg, 53% yield, 96% *ee*).

HPLC analysis: Chiralcel ADH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 7.16 min, t_R (major) = 7.89 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.45-7.42 (m, 4H), 7.34 (t, J = 7.4 Hz, 2H), 7.30-7.23 (m, 4H), 5.97-5.87 (m, 1H), 5.13-5.06 (m, 2H), 3.92 (t, J = 7.0 Hz, 1H), 2.59 (t, J = 7.0 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.4, 135.4, 131.6, 128.5, 128.2, 127.8, 127.6, 126.8, 123.7, 117.1, 90.9, 83.8, 42.8, 38.5. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-1-Chloro-4-(1-phenylpent-1-yn-3-yl)benzene (11)

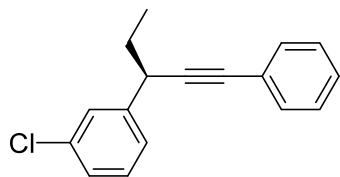


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-chlorophenyl)butanoate **S11** (78.8 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **11** as a colorless oil (32.6 mg, 64% yield, 96% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 230 nm), t_R (minor) = 7.79 min, t_R (major) = 11.23 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.47-7.43 (m, 2H), 7.37-7.35 (m, 2H), 7.32-7.29 (m, 5H), 3.78 (t, J = 6.8 Hz, 1H), 1.91-1.80 (m, 2H), 1.05 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.5, 132.4, 131.6, 128.9, 128.5, 128.2, 127.9, 123.6, 90.8, 83.7, 39.4, 31.5, 11.7; HRMS (ESI): m/z calcd for $\text{C}_{17}\text{H}_{16}\text{Cl}$ [$\text{M}+\text{H}$] $^+$: 255.0935, found: 255.0935.

(*R*)-1-Chloro-3-(1-phenylpent-1-yn-3-yl)benzene (12)

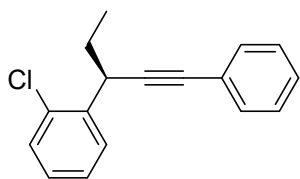


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(3-chlorophenyl)butanoate **S12** (78.8 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **12** as a colorless oil (33.1 mg, 65% yield, 95% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 8.10 min, t_R (major) = 9.62 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.46-7.41 (m, 3H), 7.31-7.28 (m, 4H), 7.26-7.20 (m, 2H), 3.77 (t, $J = 7.0$ Hz, 1H), 1.91-1.80 (m, 2H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 144.0, 134.2, 131.7, 129.6, 128.2, 127.9, 127.7, 126.9, 125.8, 123.5, 90.5, 83.8, 39.6, 31.5, 11.7; HRMS (ESI): m/z calcd for $\text{C}_{17}\text{H}_{16}\text{Cl}$ $[\text{M}+\text{H}]^+$: 255.0935, found: 255.0933.

(S)-1-Chloro-2-(1-phenylpent-1-yn-3-yl)benzene (13)

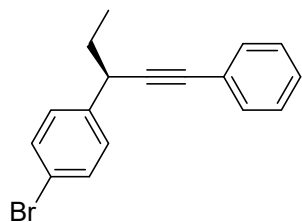


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(2-chlorophenyl)butanoate **S13** (78.8 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **13** as a colorless oil (31.1 mg, 61% yield, 98% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, $\lambda = 254$ nm), t_R (major) = 14.63 min, t_R (minor) = 17.31 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.70 (dd, $J = 7.6, 1.6$ Hz, 1H), 7.47-7.44 (m, 2H), 7.36-7.25 (m, 5H), 7.18 (td, $J = 7.6, 1.6$ Hz, 1H), 4.33-4.29 (m, 1H), 1.97-1.87 (m, 1H), 1.82-1.71 (m, 1H), 1.10 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 139.5, 132.8, 131.7, 129.5, 129.4, 128.2, 127.9, 127.8, 127.0, 123.6, 90.7, 83.4, 36.7, 29.8, 11.8; HRMS (ESI): m/z calcd for $\text{C}_{17}\text{H}_{16}\text{Cl}$ $[\text{M}+\text{H}]^+$: 255.0935, found: 255.0933.

(R)-1-Bromo-4-(1-phenylpent-1-yn-3-yl)benzene (14)



According to **General procedure 2** with

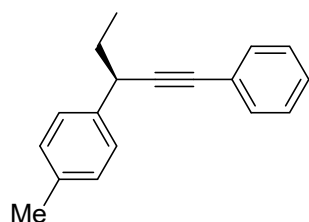
1,3-dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl 2-(4-bromophenyl)butanoate **S14** (87.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **14** as a colorless oil (37.7 mg, 63% yield, 96% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 8.71 min, t_R (major) = 13.70 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.49-7.43 (m, 4H), 7.34-7.28 (m, 5H), 3.76 (t, J = 7.0 Hz, 1H), 1.91-1.80 (m, 2H), 1.06 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.0, 131.6, 131.5, 129.3, 128.2, 127.9, 123.5, 120.4, 90.7, 83.7, 39.4, 31.5, 11.7.

The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-1-Methyl-4-(1-phenylpent-1-yn-3-yl)benzene (**15**)

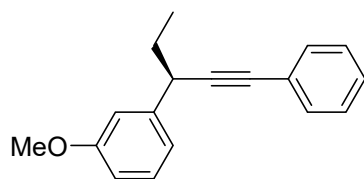


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl 2-(*p*-tolyl)butanoate **S15** (74.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **15** as a colorless oil (38.0 mg, 81% yield, 94% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 6.69 min, t_R (major) = 8.58 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.48-7.45 (m, 2H), 7.34-7.28 (m, 5H), 7.17 (d, J = 8.0 Hz, 2H), 3.77 (t, J = 7.0 Hz, 1H), 2.36 (s, 3H), 1.93-1.82 (m, 2H), 1.08 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 139.0, 136.2, 131.6, 129.1, 128.2, 127.6, 127.4, 123.9, 91.7, 83.2, 39.6, 31.7, 21.0, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(R)-1-Methoxy-3-(1-phenylpent-1-yn-3-yl)benzene (16)

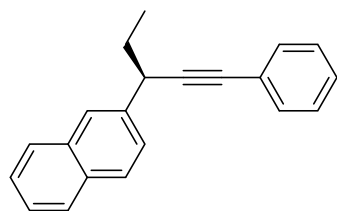


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(3-methoxyphenyl)butanoate **S16** (77.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **16** as a colorless oil (35.0 mg, 70% yield, 96% *ee*).

HPLC analysis: Chiralcel AD (hexane/*i*-PrOH = 97/3, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 10.55 min, t_R (major) = 11.78 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.45-7.43 (m, 2H), 7.30-7.23 (m, 4H), 7.00 (d, J = 6.8 Hz, 2H), 6.80-6.77 (m, 1H), 3.81 (s, 3H), 3.76 (t, J = 6.8 Hz, 1H), 1.92-1.81 (m, 2H), 1.06 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 159.7, 143.6, 131.6, 129.3, 128.2, 127.7, 123.8, 120.0, 113.5, 111.9, 91.3, 83.4, 55.2, 40.0, 31.5, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(R)-2-(1-Phenylpent-1-yn-3-yl)naphthalene (17)



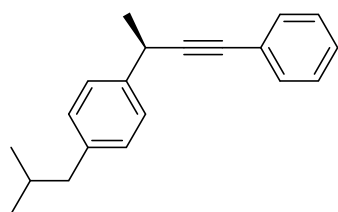
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(naphthalen-2-yl)butanoate **S17** (81.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3.5 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **17** as a colorless oil (32.4 mg, 60% yield, 95% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99/1, flow rate 0.5 mL/min, λ =

254 nm), t_R (minor) = 11.11 min, t_R (major) = 12.40 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.89-7.84 (m, 4H), 7.58-7.45 (m, 5H), 7.36-7.31 (m, 3H), 3.98 (t, J = 7.0 Hz, 1H), 2.04-1.93 (m, 2H), 1.11 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 139.4, 133.5, 132.5, 131.7, 128.2, 128.1, 127.8, 127.7, 127.6, 126.0, 125.97, 125.5, 123.8, 91.4, 83.6, 40.1, 31.4, 11.9. HRMS (ESI): m/z calcd for $\text{C}_{21}\text{H}_{19}$ $[\text{M}+\text{H}]^+$: 271.1481, found: 271.1478.

(*R*)-1-Isobutyl-4-(4-phenylbut-3-yn-2-yl)benzene (**18**)

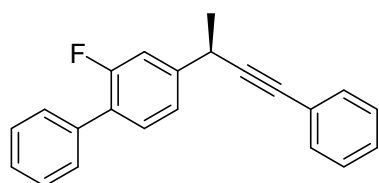


According to General procedure 2 with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-isobutylphenyl)propanoate **S18** (80.3 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.3 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **18** as a colorless oil (36.2 mg, 69% yield, 91% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 8.05 min, t_R (major) = 10.63 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.47-7.45 (m, 2H), 7.37 (d, J = 8.0 Hz, 2H), 7.33-7.29 (m, 3H), 7.13 (d, J = 7.6 Hz, 2H), 3.98 (q, J = 7.1 Hz, 1H), 2.48 (d, J = 7.2 Hz, 2H), 1.93-1.83 (m, 1H), 1.59 (d, J = 7.2 Hz, 3H), 0.93 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.5, 140.0, 131.6, 129.3, 128.2, 127.7, 126.6, 123.8, 92.9, 82.2, 45.0, 32.1, 30.2, 24.5, 22.4; HRMS (ESI): m/z calcd for $\text{C}_{20}\text{H}_{23}$ $[\text{M}+\text{H}]^+$: 263.1794, found: 263.1792.

(*R*)-2-Fluoro-4-(4-phenylbut-3-yn-2-yl)-1,1'-biphenyl (**19**)

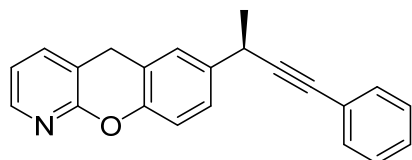


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate **S19** (87.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.3 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **19** as a colorless oil (30.6 mg, 51% yield, 90% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 6.85 min, t_R (major) = 7.42 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.55 (d, J = 7.6 Hz, 2H), 7.48-7.41 (m, 5H), 7.39-7.27 (m, 6H), 4.02 (q, J = 7.1 Hz, 1H), 1.62 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 159.7 (d, J = 246.5 Hz), 144.8 (d, J = 7.5 Hz), 135.7, 131.6, 130.8 (d, J = 3.9 Hz), 129.0 (d, J = 2.7 Hz), 128.4, 128.2, 127.9, 127.6, 123.4, 122.9 (d, J = 3.1 Hz), 114.8, 114.6, 91.7, 82.9, 32.0, 24.2; ^{19}F NMR (376 MHz, CDCl_3) δ : -117.8; HRMS (ESI): m/z calcd for $\text{C}_{22}\text{H}_{18}\text{F}$ [$\text{M}+\text{H}$] $^+$: 301.1387, found: 301.1385.

(*R*)-7-(4-Phenylbut-3-yn-2-yl)-5*H*-chromeno[2,3-*b*]pyridine (20)



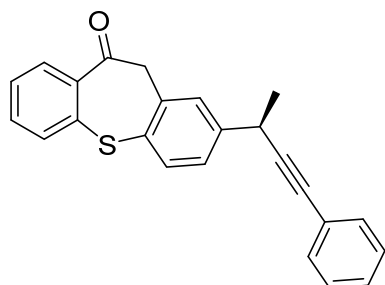
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(5*H*-chromeno[2,3-*b*]pyridin-7-yl)propanoate **S20** (90.1 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.3 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 4/1) to yield the product **20** as a colorless oil (39.2 mg, 63% yield, 98% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R (minor) = 7.13 min, t_R (major) = 8.24 min.

^1H NMR (400 MHz, CDCl_3) δ : 8.16 (d, J = 4.0 Hz, 1H), 7.53 (d, J = 7.2 Hz, 1H), 7.46-7.43 (m, 2H), 7.31-7.27 (m, 4H), 7.25 (s, 1H), 7.13 (d, J = 8.4 Hz, 1H), 7.03-7.00 (m, 1H), 4.11 (s, 2H), 3.95 (q, J = 7.2 Hz, 1H), 1.57 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 158.4, 150.3, 146.6, 138.7, 138.4, 131.6, 128.2, 127.8,

126.8, 126.6, 123.6, 119.7, 119.4, 117.2, 115.4, 92.4, 82.5, 31.8, 28.2, 24.5; HRMS (ESI): m/z calcd for $C_{22}H_{18}NO$ $[M+H]^+$: 312.1383, found: 312.1381.

(R)-2-(4-Phenylbut-3-yn-2-yl)dibenzo[*b,f*]thiepin-10(11*H*)-one (21)



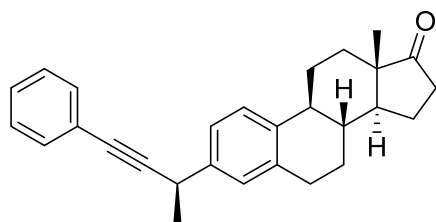
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

2-(10-oxo-10,11-dihydrodibenzo[*b,f*]thiepin-2-yl)propanoate **S21** (98.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.3 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/50) to yield the product **21** as a colorless oil (28.4 mg, 40% yield, 88% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99/1, flow rate 0.7 mL/min, λ = 240 nm), t_R (major) = 16.74 min, t_R (minor) = 19.45 min.

1H NMR (400 MHz, $CDCl_3$) δ : 8.21 (d, J = 8.0 Hz, 1H), 7.63-7.60 (m, 2H), 7.52 (s, 1H), 7.44-7.41 (m, 3H), 7.33-7.29 (m, 5H), 4.43-4.35 (m, 2H), 3.98 (q, J = 7.2 Hz, 1H), 1.56 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 191.5, 145.6, 140.4, 137.8, 136.2, 132.7, 132.5, 131.6, 131.5, 131.45, 130.8, 128.2, 128.0, 127.9, 126.8, 125.9, 123.4, 91.7, 82.9, 51.1, 32.2, 24.3; HRMS (ESI): m/z calcd for $C_{24}H_{19}OS$ $[M+H]^+$: 355.1151, found: 355.1149.

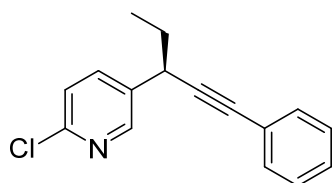
(8*R*,9*S*,13*S*,14*S*)-13-Methyl-3-((*R*)-4-phenylbut-3-yn-2-yl)-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[α]phenanthren-17-one (22)



According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl (*S*)-2-((8*R*,9*S*,13*S*,14*S*)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6*H*-cyclopenta[α]phenanthren-3-yl)propanoate **S22** (78.2 mg, 0.15 mmol, 1.0 equiv.), ethynylbenzene **2a** (23.0 mg, 0.225 mmol, 1.5 equiv.), CuI (5.7 mg, 0.03 mmol, 20 mol%) and **L1** (30.1 mg, 0.036 mmol, 24 mol%) for 3 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/2) to yield the product **22** as a colorless oil (24.7 mg, 43% yield, dr > 20:1). The diastereomeric ratio was determined by crude ¹H NMR spectroscopy.

¹H NMR (400 MHz, CDCl₃) δ : 7.44-7.42 (m, 2H), 7.30-7.22 (m, 6H), 7.18 (s, 1H), 3.92 (q, *J* = 7.1 Hz, 1H), 2.95-2.92 (m, 2H), 2.54-2.41 (m, 2H), 2.32-2.27 (m, 1H), 2.17-1.94 (m, 4H), 1.66-1.45 (m, 9H), 0.91 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 220.9, 140.8, 138.1, 136.6, 131.6, 128.2, 127.7, 127.5, 125.6, 124.4, 123.8, 92.8, 82.1, 50.5, 48.0, 44.3, 38.1, 35.8, 32.0, 31.6, 29.5, 26.5, 25.7, 24.4, 21.6, 13.8. The NMR spectra were in accord with that reported in literature.^[5]

(*S*)-2-Chloro-5-(1-phenylpent-1-yn-3-yl)pyridine (**23**)



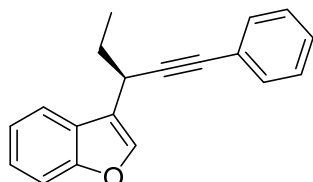
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(6-chloropyridin-3-yl)butanoate **S23** (79.0 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **23** as a colorless oil (29.7 mg, 58% yield, 96% *ee*).

HPLC analysis: Chiralcel ASH (hexane/*i*-PrOH = 97/3, flow rate 0.7 mL/min, λ = 240 nm), *t*_R (minor) = 9.53 min, *t*_R (major) = 12.47 min.

¹H NMR (400 MHz, CDCl₃) δ : 8.41 (d, *J* = 2.4 Hz, 1H), 7.74 (dd, *J* = 8.4, 2.8 Hz, 1H), 7.45-7.41 (m, 2H), 7.32-7.30 (m, 4H), 3.82 (t, *J* = 7.0 Hz, 1H), 1.90-1.82 (m, 2H), 1.07 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 149.8, 148.9, 138.0, 136.5,

131.6, 128.3, 128.1, 124.0, 123.1, 89.3, 84.3, 36.8, 31.3, 11.6; HRMS (ESI): m/z calcd for $C_{16}H_{15}ClN$ $[M+H]^+$: 256.0888, found: 256.0886.

(S)-3-(1-Phenylpent-1-yn-3-yl)benzofuran (24)

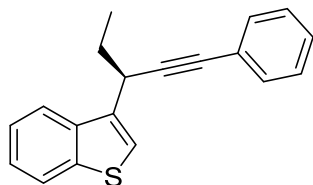


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(benzofuran-3-yl)butanoate **S24** (79.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **24** as a colorless oil (35.4 mg, 68% yield, 97% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 10.86 min, t_R (major) = 12.59 min.

1H NMR (400 MHz, $CDCl_3$) δ : 7.73 (d, J = 7.2 Hz, 1H), 7.61 (s, 1H), 7.50-7.43 (m, 3H), 7.32-7.23 (m, 5H), 4.00 (t, J = 6.8 Hz, 1H), 2.10-1.92 (m, 2H), 1.12 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 155.7, 141.8, 131.6, 128.2, 127.9, 126.7, 124.2, 123.5, 122.4, 120.8, 120.1, 111.6, 90.0, 82.5, 30.0, 28.6, 11.7; HRMS (ESI): m/z calcd for $C_{19}H_{17}O$ $[M+H]^+$: 261.1274, found: 261.1272.

(S)-3-(1-Phenylpent-1-yn-3-yl)benzo[*b*]thiophene (25)



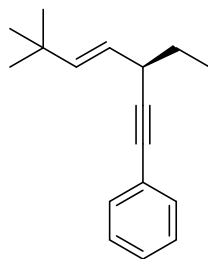
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(benzo[*b*]thiophen-3-yl)butanoate **S25** (83.1 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **25** as a colorless oil (35.9 mg, 65%

yield, 97% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99/1, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 18.24 min, t_R (major) = 20.50 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.92-7.86 (m, 2H), 7.44-7.24 (m, 8H), 4.19 (t, J = 6.6 Hz, 1H), 2.12-1.92 (m, 2H), 1.13 (td, J = 7.2, 1.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.9, 137.5, 136.0, 131.6, 128.2, 127.8, 124.2, 123.9, 123.6, 123.0, 122.7, 121.9, 90.5, 83.0, 34.1, 28.9, 11.9; HRMS (ESI): m/z calcd for $\text{C}_{19}\text{H}_{17}\text{S}$ $[\text{M}+\text{H}]^+$: 277.1045, found: 277.1046.

(*R,E*)-(3-Ethyl-6,6-dimethylhept-4-en-1-yn-1-yl)benzene (26)



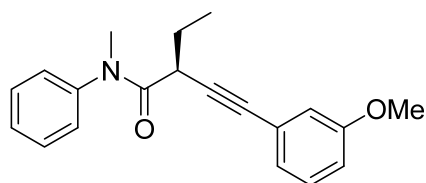
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

(*E*)-2-ethyl-5,5-dimethylhex-3-enoate **S26** (36.5 mg, 0.10 mmol, 1.0 equiv.), ethynylbenzene **2a** (15.3 mg, 0.15 mmol, 1.5 equiv.), CuI (3.8 mg, 0.02 mmol, 20 mol%) and **L1** (20.1 mg, 0.024 mmol, 24 mol%) in Et_2O (2 mL) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **26** as a colorless oil (9.5 mg, 42% yield, 77% *ee*).

HPLC analysis: Chiralcel OD3 (hexane/*i*-PrOH = 100/0, flow rate 0.5 mL/min, λ = 254 nm), t_R (minor) = 10.90 min, t_R (major) = 12.01 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.43-7.42 (m, 2H), 7.31-7.26 (m, 3H), 5.73 (d, J = 15.6 Hz, 1H), 5.32 (dd, J = 15.6, 6.8 Hz, 1H), 3.14 (q, J = 6.7 Hz, 1H), 1.66-1.60 (m, 2H), 1.04-1.00 (m, 12H); ^{13}C NMR (100 MHz, CDCl_3) δ : 142.4, 131.6, 128.1, 127.5, 124.3, 124.1, 91.7, 83.0, 36.6, 32.8, 29.7, 29.2, 11.4; HRMS (ESI): m/z calcd for $\text{C}_{17}\text{H}_{23}$ $[\text{M}+\text{H}]^+$: 227.1794, found: 227.1796.

(*S*)-2-Ethyl-4-(3-methoxyphenyl)-*N*-methyl-*N*-phenylbut-3-ynamide (27)

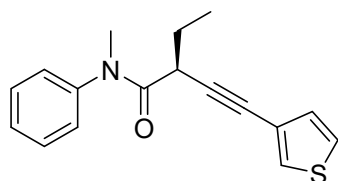


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(methyl(phenyl)carbamoyl)butanoate **S27/28** (41.6 mg, 0.10 mmol, 1.0 equiv.), 1-ethynyl-3-methoxybenzene **2b** (19.8 mg, 0.15 mmol, 1.5 equiv.), CuI (2.9 mg, 0.015 mmol, 15 mol%) and **L1** (15.1 mg, 0.018 mmol, 18 mol%) for 3 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1:10) to yield the product **27** as a colorless oil (11.4 mg, 37% yield, 70% *ee*).

HPLC analysis: Chiralcel IG (hexane/*i*-PrOH = 95/5, flow rate 0.9 mL/min, λ = 254 nm), t_R (major) = 39.44 min, t_R (minor) = 46.02 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.45 (t, J = 7.4 Hz, 2H), 7.37 (t, J = 7.4 Hz, 1H), 7.31 (d, J = 7.6 Hz, 2H), 7.18 (t, J = 8.0 Hz, 1H), 6.98 (d, J = 7.6 Hz, 1H), 6.92 (s, 1H), 6.84 (dd, J = 8.4, 2.4 Hz, 1H), 3.79 (s, 3H), 3.32-3.26 (m, 4H), 1.95-1.78 (m, 2H), 0.93 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.1, 159.2, 143.5, 129.8, 129.1, 128.0, 127.5, 124.4, 124.3, 116.5, 114.5, 87.6, 82.3, 55.3, 37.9, 37.8, 26.1, 11.9; HRMS (ESI): m/z calcd for $\text{C}_{20}\text{H}_{22}\text{NO}_2$ [$\text{M}+\text{H}$] $^+$: 308.1645, found: 308.1641.

(*S*)-2-Ethyl-*N*-methyl-*N*-phenyl-4-(thiophen-3-yl)but-3-ynamide (**28**)



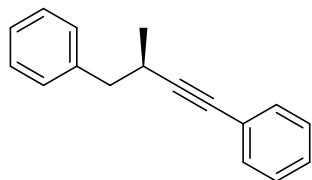
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(methyl(phenyl)carbamoyl)butanoate **S27/28** (41.6 mg, 0.10 mmol, 1.0 equiv.), 3-ethynylthiophene **2c** (16.2 mg, 0.15 mmol, 1.5 equiv.), CuI (2.9 mg, 0.015 mmol, 15 mol%) and **L1** (15.1 mg, 0.018 mmol, 18 mol%) for 3 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1:10)

to yield the product **28** as a colorless oil (8.8 mg, 31% yield, 76% *ee*).

HPLC analysis: Chiralcel IG (hexane/*i*-PrOH = 97/3, flow rate 0.9 mL/min, λ = 240 nm), t_R (minor) = 43.68 min, t_R (major) = 48.32 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.45 (t, J = 7.4 Hz, 2H), 7.39-7.37 (m, 2H), 7.30 (d, J = 7.6 Hz, 2H), 7.23-7.21 (m, 1H), 7.06 (d, J = 5.2 Hz, 1H), 3.32-3.26 (m, 4H), 1.93-1.77 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.1, 143.6, 130.0, 129.8, 128.3, 128.0, 127.5, 124.9, 122.3, 87.3, 77.5, 37.9, 37.8, 26.1, 11.9; HRMS (ESI): m/z calcd for $\text{C}_{17}\text{H}_{18}\text{NOS}$ $[\text{M}+\text{H}]^+$: 284.1104, found: 284.1101.

(*R*)-(3-Methylbut-1-yne-1,4-diyl)dibenzene (**29**)

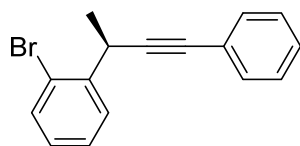


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-methyl-3-phenylpropanoate **S29** (71.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **29** as a colorless oil (22.5 mg, 51% yield, <5% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 11.18 min, t_R (major) = 11.95 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.36-7.20 (m, 10H), 2.95-2.87 (m, 2H), 2.81-2.74 (m, 1H), 1.26 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 139.6, 131.5, 129.3, 128.1, 127.5, 126.3, 123.9, 94.1, 81.6, 43.2, 28.6, 20.6. The NMR spectra were in accord with that reported in literature.^[8]

(*S*)-1-Bromo-2-(4-phenylbut-3-yn-2-yl)benzene (**31**)



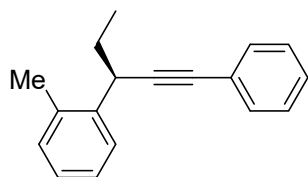
According to **General procedure 2** with

1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(2-bromophenyl)propanoate **S31** (84.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **31** as a colorless oil (29.7 mg, 52% yield, 97% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 240 nm), t_R (major) = 8.66 min, t_R (minor) = 9.32 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.76 (dd, J = 7.6, 1.6 Hz, 1H), 7.55 (dd, J = 8.0, 1.2 Hz, 1H), 7.47-7.44 (m, 2H), 7.36-7.29 (m, 4H), 7.12 (td, J = 7.6, 1.6 Hz, 1H), 4.43 (q, J = 6.9 Hz, 1H), 1.56 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 142.4, 132.8, 131.6, 128.8, 128.3, 128.2, 127.9, 127.9, 123.5, 123.0, 91.9, 82.5, 32.4, 23.1. The NMR spectra were in accord with that reported in literature.^[5]

(*S*)-1-Methyl-2-(1-phenylpent-1-yn-3-yl)benzene (**32**)

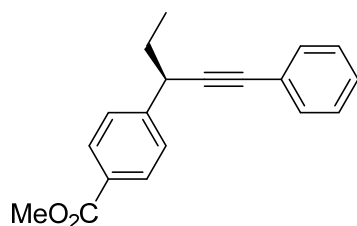


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(*o*-tolyl)butanoate **S32** (74.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **32** as a colorless oil (30.5 mg, 65% yield, 96% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (major) = 8.12 min, t_R (minor) = 8.74 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.58 (d, J = 7.2 Hz, 1H), 7.46-7.43 (m, 2H), 7.32-7.28 (m, 3H), 7.24-7.20 (m, 1H), 7.16-7.15 (m, 2H), 3.99-3.96 (m, 1H), 2.40 (s, 3H), 1.89-1.77 (m, 2H), 1.12 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.2, 134.9, 131.6, 130.4, 128.2, 127.6, 126.6, 126.2, 123.9, 91.9, 82.7, 36.5, 30.1, 19.2, 12.2. The NMR spectra were in accord with that reported in literature.^[5]

Methyl (*R*)-4-(1-phenylpent-1-yn-3-yl)benzoate (**33**)

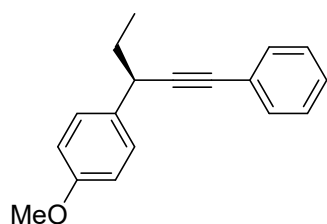


According to **General procedure 2** with methyl 4-(1-((1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl)oxy)-1-oxobutan-2-yl)benzoate **S33** (83.5 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **33** as a colorless oil (26.2 mg, 47% yield, 87% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 97/3, flow rate 0.7 mL/min, λ = 240 nm), t_R (minor) = 7.64 min, t_R (major) = 9.22 min.

^1H NMR (400 MHz, CDCl_3) δ : 8.01 (d, J = 8.4 Hz, 2H), 7.49 (d, J = 8.4 Hz, 2H), 7.47-7.43 (m, 2H), 7.34-7.29 (m, 3H), 3.92 (s, 3H), 3.85 (t, J = 7.0 Hz, 1H), 1.93-1.82 (m, 2H), 1.05 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 167.0, 147.3, 131.6, 129.8, 128.6, 128.2, 127.9, 127.6, 123.5, 90.4, 83.9, 52.0, 39.9, 31.4, 11.7; HRMS (ESI): m/z calcd for $\text{C}_{19}\text{H}_{19}\text{O}_2$ [$\text{M}+\text{H}$] $^+$: 279.1380, found: 279.1380.

(*R*)-1-Methoxy-4-(1-phenylpent-1-yn-3-yl)benzene (**34**)



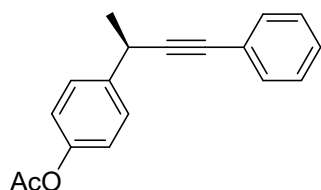
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-methoxyphenyl)butanoate **S34** (77.9 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **34** as a colorless oil (35.5 mg, 71% yield, 88% *ee*).

HPLC analysis: Chiralcel AD (hexane/*i*-PrOH = 97/3, flow rate 0.7 mL/min, λ = 254

nm), t_R (minor) = 11.02 min, t_R (major) = 12.23 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.45-7.43 (m, 2H), 7.35-7.27 (m, 5H), 6.89-6.86 (m, 2H), 3.80 (s, 3H), 3.74 (t, J = 7.0 Hz, 1H), 1.87-1.79 (m, 2H), 1.04 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 158.3, 134.1, 131.6, 128.5, 128.2, 127.6, 123.9, 113.8, 91.81, 83.2, 55.3, 39.1, 31.7, 11.8; HRMS (ESI): m/z calcd for $\text{C}_{18}\text{H}_{19}\text{O}$ $[\text{M}+\text{H}]^+$: 251.1430, found: 251.1429.

(*R*)-4-(4-Phenylbut-3-yn-2-yl)phenyl acetate (35)

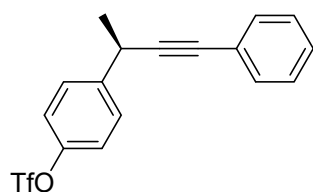


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-acetoxyphenyl)propanoate **S35** (80.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/2) to yield the product **35** as a colorless oil (18.5 mg, 35% yield, 93% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 97/3, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 9.43 min, t_R (major) = 10.12 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.47-7.41 (m, 4H), 7.31-7.27 (m, 3H), 7.07-7.04 (m, 2H), 3.98 (q, J = 7.1 Hz, 1H), 2.30 (s, 3H), 1.57 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 169.6, 149.3, 140.8, 131.6, 128.2, 128.0, 127.8, 123.6, 121.5, 92.3, 82.6, 32.0, 24.4, 21.1; HRMS (ESI): m/z calcd for $\text{C}_{18}\text{H}_{16}\text{NaO}_2$ $[\text{M}+\text{Na}]^+$: 287.1043, found: 287.1041.

(*R*)-4-(4-Phenylbut-3-yn-2-yl)phenyl trifluoromethanesulfonate (36)



According to **General procedure 2** with

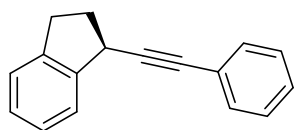
1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl

2-(4-(((trifluoromethyl)sulfonyl)oxy)phenyl)propanoate **S36** (98.7 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **36** as a colorless oil (21.3 mg, 30% yield, 93% *ee*).

HPLC analysis: Chiralcel IA (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 38.88 min, t_R (major) = 46.45 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.55-7.52 (m, 2H), 7.45-7.43 (m, 2H), 7.31-7.30 (m, 3H), 7.26-7.24 (m, 2H), 4.02 (q, J = 7.1 Hz, 1H), 1.59 (d, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 148.2, 143.8, 131.6, 128.8, 128.3, 128.0, 123.2, 121.4, 118.7 (q, J = 318.8 Hz), 91.3, 83.2, 32.0, 24.4; ^{19}F NMR (376 MHz, CDCl_3) δ : -72.8; HRMS (ESI): m/z calcd for $\text{C}_{17}\text{H}_{14}\text{F}_3\text{O}_3\text{S}$ $[\text{M}+\text{H}]^+$: 355.0610, found: 355.0609.

(*S*)-1-(Phenylethynyl)-2,3-dihydro-1*H*-indene (**37**)

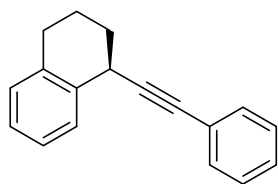


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2,3-dihydro-1*H*-indene-1-carboxylate **S37** (71.5 mg, 0.20 mmol, 1.0 equiv.), ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) and **L2** (16.7 mg, 0.024 mmol, 12 mol%) in Et_2O (4 mL) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **37** as a colorless oil (17.5 mg, 40% yield, 82% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 20.24 min, t_R (major) = 30.71 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.48-7.41 (m, 3H), 7.30-7.19 (m, 7H), 4.20 (t, J = 8.4 Hz, 1H), 3.06-2.99 (m, 1H), 2.95-2.87 (m, 1H), 2.62-2.55 (m, 1H), 2.25-2.15 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 143.6, 143.0, 131.7, 128.2, 127.7, 127.1, 126.6, 124.5, 124.3, 123.7, 91.5, 81.6, 36.8, 34.4, 31.5; HRMS (ESI): m/z calcd for $\text{C}_{17}\text{H}_{15}$ $[\text{M}+\text{H}]^+$: 219.1168, found: 219.1168.

(S)-1-(Phenylethynyl)-1,2,3,4-tetrahydronaphthalene (38)



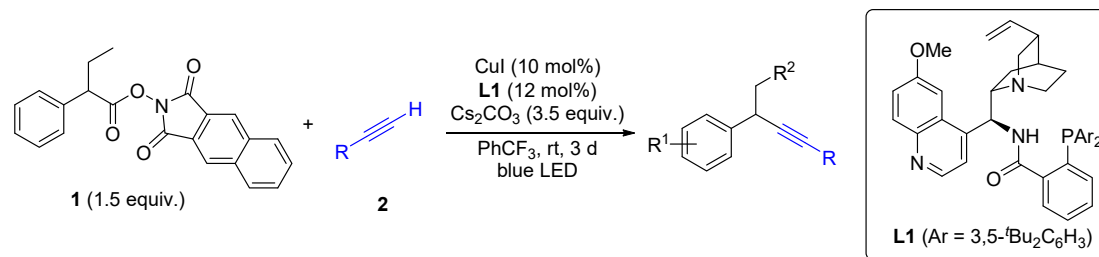
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 1,2,3,4-tetrahydronaphthalene-1-carboxylate **S38** (74.3 mg, 0.20 mmol, 1.0 equiv.), ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) and **L2** (16.7 mg, 0.024 mmol, 12 mol%) in Et₂O (4 mL) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **38** as a colorless oil (19.1 mg, 41% yield, 67% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 15.28 min, t_R (major) = 18.84 min.

¹H NMR (400 MHz, CDCl₃) δ : 7.55-7.53 (m, 1H), 7.43-7.39 (m, 2H), 7.29-7.25 (m, 3H), 7.20-7.13 (m, 2H), 7.10-7.08 (m, 1H), 4.01 (t, J = 6.6 Hz, 1H), 2.91-2.75 (m, 2H), 2.22-2.16 (m, 1H), 2.11-1.96 (m, 2H), 1.86-1.77 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ : 136.3, 136.2, 131.6, 129.2, 129.1, 128.1, 127.6, 126.5, 126.0, 123.8, 93.0, 81.4, 32.1, 30.3, 29.1, 21.3; HRMS (ESI): m/z calcd for C₁₈H₁₇ [M+H]⁺: 233.1325, found: 233.1323.

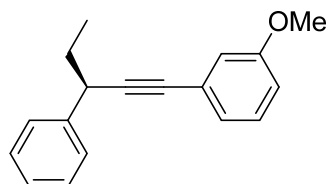
8. Asymmetric decarboxylative alkylation: Scope of alkynes

General procedure 3:



CuI (3.8 mg, 0.02 mmol, 10 mol%), **L1** (20.1 mg, 0.024 mmol, 12 mol%), Cs₂CO₃ (228.1 mg, 0.70 mmol, 3.5 equiv.), *N*-hydroxyphthalimide esters **1** (0.30 mmol, 1.5 equiv.) and anhydrous PhCF₃ (4.0 mL) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere. Then, alkyne **2** (0.20 mmol, 1.0 equiv.) was added to the mixture. The reaction mixture was stirred under the irradiation of a 24 W blue LED for 3 to 4 d at room temperature. After the completion of reaction, the reaction mixture was filtered and the precipitate was washed by EtOAc. The filtrate was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography on silica gel to afford the alkylation product.

(*R*)-1-Methoxy-3-(3-phenylpent-1-yn-1-yl)benzene (**39**)



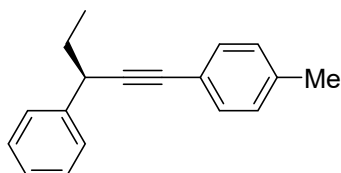
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 1-ethynyl-3-methoxybenzene **2b** (26.4 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **39** as a colorless oil (36.1 mg, 72% yield, 97% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), *t_R* (minor) = 26.49 min, *t_R* (major) = 33.84 min.

¹H NMR (400 MHz, CDCl₃) δ : 7.43-7.41 (m, 2H), 7.36-7.32 (m, 2H), 7.27-7.18 (m,

2H), 7.06-7.04 (m, 1H), 6.98-6.97 (m, 1H), 6.86-6.83 (m, 1H), 3.79-3.77 (m, 4H), 1.92-1.81 (m, 2H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 159.3, 141.9, 129.2, 128.4, 127.5, 126.6, 124.8, 124.2, 116.5, 114.3, 91.3, 83.2, 55.2, 39.9, 31.6, 11.9. The NMR spectra were in accord with that reported in literature.^[5]

(R)-1-Methyl-4-(3-phenylpent-1-yn-1-yl)benzene (40)



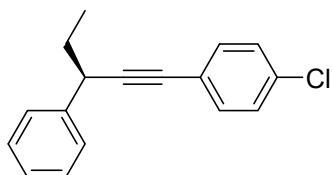
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 1-ethynyl-4-methylbenzene **2d** (23.2 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **40** as a colorless oil (35.2 mg, 75% yield, 97% *ee*).

$[\alpha]_{\text{D}}^{25} = -10.9$ (c 0.73, CH_2Cl_2).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, $\lambda = 254$ nm), t_{R} (minor) = 14.83 min, t_{R} (major) = 18.13 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.43-7.41 (m, 2H), 7.35-7.31 (m, 4H), 7.26-7.21 (m, 1H), 7.10 (d, $J = 7.6$ Hz, 2H), 3.78 (t, $J = 7.0$ Hz, 1H), 2.33 (s, 3H), 1.91-1.80 (m, 2H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 142.1, 137.7, 131.5, 128.9, 128.4, 127.5, 126.6, 120.7, 90.6, 83.4, 39.9, 31.7, 21.4, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(R)-1-Chloro-4-(3-phenylpent-1-yn-1-yl)benzene (41)



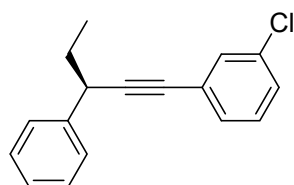
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg,

0.30 mmol, 1.5 equiv.) and 1-chloro-4-ethynylbenzene **2e** (27.3 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **41** as a colorless oil (35.7 mg, 70% yield, 93% *ee*).

HPLC analysis: Chiralcel AD (hexane/*i*-PrOH = 100/0, flow rate 0.5 mL/min, λ = 254 nm), t_R (minor) = 12.33 min, t_R (major) = 13.79 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.41-7.32 (m, 6H), 7.29-7.23 (m, 3H), 3.77 (t, J = 7.0 Hz, 1H), 1.90-1.82 (m, 2H), 1.04 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.7, 133.6, 132.9, 128.49, 128.45, 127.5, 126.7, 122.3, 92.6, 82.2, 40.0, 31.5, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-1-Chloro-3-(3-phenylpent-1-yn-1-yl)benzene (**42**)

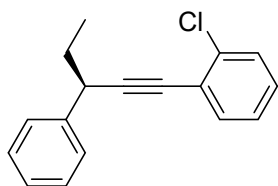


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 1-chloro-3-ethynylbenzene **2f** (27.3 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **42** as a colorless oil (38.7 mg, 76% yield, 95% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 12.21 min, t_R (major) = 13.06 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.44-7.38 (m, 3H), 7.36-7.30 (m, 3H), 7.28-7.20 (m, 3H), 3.78 (t, J = 7.0 Hz, 1H), 1.92-1.81 (m, 2H), 1.05 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.6, 134.0, 131.5, 129.8, 129.4, 128.5, 128.0, 127.5, 126.8, 125.5, 92.9, 82.0, 39.9, 31.5, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-1-Chloro-2-(3-phenylpent-1-yn-1-yl)benzene (**43**)

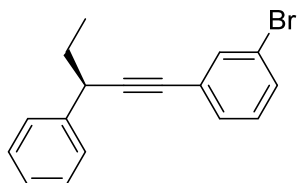


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 1-chloro-2-ethynylbenzene **2g** (27.3 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **43** as a colorless oil (36.7 mg, 72% yield, 95% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 16.70 min, t_R (major) = 18.58 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.48-7.45 (m, 3H), 7.40-7.32 (m, 3H), 7.26-7.16 (m, 3H), 3.88-3.84 (m, 1H), 1.95-1.84 (m, 2H), 1.09 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.6, 136.0, 133.3, 129.1, 128.7, 128.4, 127.6, 126.7, 126.3, 123.7, 97.0, 80.3, 40.2, 31.7, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-1-Bromo-3-(3-phenylpent-1-yn-1-yl)benzene (**44**)



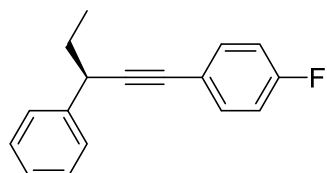
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 1-bromo-3-ethynylbenzene **2h** (36.2 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **44** as a colorless oil (41.9 mg, 70% yield, 93% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 12.76 min, t_R (major) = 13.61 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.60-7.59 (m, 1H), 7.43-7.32 (m, 6H), 7.27-7.23 (m,

1H), 7.16 (t, $J = 8.0$ Hz, 1H), 3.77 (t, $J = 7.0$ Hz, 1H), 1.92-1.81 (m, 2H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.6, 134.4, 130.9, 130.2, 129.6, 128.5, 127.5, 126.8, 125.8, 122.0, 93.1, 81.9, 39.9, 31.5, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(R)-1-Fluoro-4-(3-phenylpent-1-yn-1-yl)benzene (45)

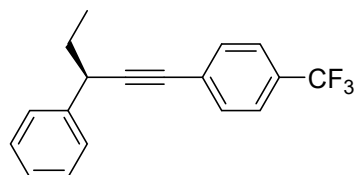


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 1-ethynyl-4-fluorobenzene **2i** (24.0 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **45** as a colorless oil (35.7 mg, 75% yield, 94% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, $\lambda = 254$ nm), t_R (minor) = 24.35 min, t_R (major) = 27.92 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.44-7.39 (m, 4H), 7.36-7.32 (m, 2H), 7.27-7.23 (m, 1H), 7.01-6.96 (m, 2H), 3.77 (t, $J = 7.0$ Hz, 1H), 1.91-1.80 (m, 2H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 162.2 (d, $J = 247.0$ Hz), 141.9, 133.4 (d, $J = 8.1$ Hz), 128.4, 127.5, 126.7, 119.9 (d, $J = 3.5$ Hz), 115.4 (d, $J = 21.9$ Hz), 91.1, 82.2, 39.9, 31.6, 11.8; ^{19}F NMR (376 MHz, CDCl_3) δ : -112.1. The NMR spectra were in accord with that reported in literature.^[5]

(R)-1-(3-Phenylpent-1-yn-1-yl)-4-(trifluoromethyl)benzene (46)



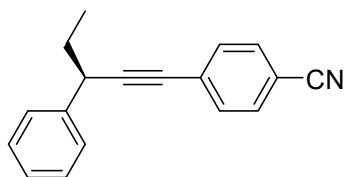
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg,

0.30 mmol, 1.5 equiv.) and 1-ethynyl-4-(trifluoromethyl)benzene **2j** (34.0 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **46** as a colorless oil (46.1 mg, 80% yield, 96% *ee*).

HPLC analysis: Chiralcel IA (hexane/*i*-PrOH = 100/0, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 25.67 min, t_R (major) = 27.61 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.56-7.52 (m, 4H), 7.41-7.39 (m, 2H), 7.36-7.33 (m, 2H), 7.28-7.24 (m, 1H), 3.80 (t, J = 7.2 Hz, 1H), 1.93-1.83 (m, 2H), 1.06 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.5, 131.9, 129.5 (q, J = 32.3 Hz), 128.5, 127.7 (d, J = 1.4 Hz), 127.5, 126.8, 125.1 (q, J = 3.8 Hz), 124.0 (q, J = 270.5 Hz), 94.3, 82.2, 40.0, 31.5, 11.8; ^{19}F NMR (376 MHz, CDCl_3) δ : -62.7. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-4-(3-Phenylpent-1-yn-1-yl)benzonitrile (**47**)

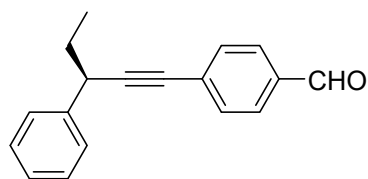


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 4-ethynylbenzonitrile **2k** (25.4 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **47** as a colorless oil (34.8 mg, 71% yield, 95% *ee*).

HPLC analysis: Chiralcel ASH (hexane/*i*-PrOH = 95/5, flow rate 0.8 mL/min, λ = 254 nm), t_R (minor) = 9.30 min, t_R (major) = 11.43 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.58 (d, J = 8.4 Hz, 2H), 7.51 (d, J = 8.8 Hz, 2H), 7.40-7.33 (m, 4H), 7.28-7.25 (m, 1H), 3.80 (t, J = 7.0 Hz, 1H), 1.94-1.83 (m, 2H), 1.05 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.2, 132.2, 131.9, 128.8, 128.5, 127.4, 126.9, 118.6, 111.0, 96.6, 82.0, 40.0, 31.4, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-4-(3-Phenylpent-1-yn-1-yl)benzaldehyde (**48**)

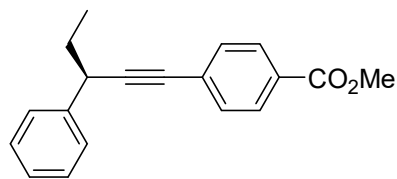


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 4-ethynylbenzaldehyde **2i** (26.0 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **48** as a colorless oil (32.8 mg, 66% yield, 96% *ee*).

HPLC analysis: Chiralcel ASH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.3 mL/min, λ = 290 nm), t_R (minor) = 28.29 min, t_R (major) = 31.21 min.

^1H NMR (400 MHz, CDCl_3) δ : 9.99 (s, 1H), 7.81 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 8.4 Hz, 2H), 7.42-7.40 (m, 2H), 7.35 (t, J = 7.6 Hz, 2H), 7.28-7.24 (m, 1H), 3.82 (t, J = 7.0 Hz, 1H), 1.95-1.84 (m, 2H), 1.06 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 191.5, 141.4, 135.1, 132.2, 130.2, 129.5, 128.5, 127.5, 126.9, 96.2, 82.7, 40.1, 31.4, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

Methyl (*R*)-4-(3-phenylpent-1-yn-1-yl)benzoate (**49**)

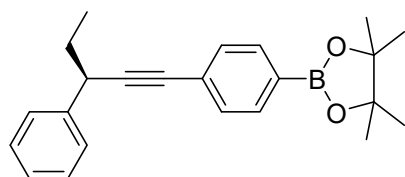


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and methyl 4-ethynylbenzoate **2m** (32.0 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **49** as a colorless oil (39.0 mg, 70% yield, 93% *ee*).

HPLC analysis: Chiralcel IA (hexane/*i*-PrOH = 97/3, flow rate 0.5 mL/min, λ = 254 nm), t_R (minor) = 10.10 min, t_R (major) = 11.31 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.98-7.96 (m, 2H), 7.51-7.49 (m, 2H), 7.42-7.40 (m, 2H), 7.36-7.33 (m, 2H), 7.28-7.23 (m, 1H), 3.91 (s, 3H), 3.80 (t, $J = 7.0$ Hz, 1H), 1.92-1.84 (m, 2H), 1.06 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 166.7, 141.5, 131.6, 129.4, 129.0, 128.6, 128.5, 127.5, 126.8, 94.9, 82.7, 52.2, 40.1, 31.5, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(R)-4,4,5,5-Tetramethyl-2-(4-(3-phenylpent-1-yn-1-yl)phenyl)-1,3,2-dioxaborolane (50)

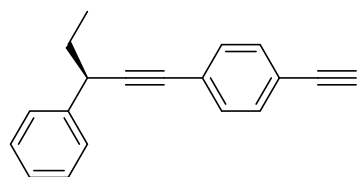


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 2-(4-ethynylphenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane **2n** (45.6 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/100) to yield the product **50** as a colorless oil (43.6 mg, 63% yield, 96% *ee*).

HPLC analysis: Chiralcel IA (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.2 mL/min, $\lambda = 254$ nm), t_R (minor) = 24.78 min, t_R (major) = 29.18 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.73 (d, $J = 8.0$ Hz, 2H), 7.45-7.40 (m, 4H), 7.35-7.32 (m, 2H), 7.26-7.22 (m, 1H), 3.79 (t, $J = 7.0$ Hz, 1H), 1.92-1.81 (m, 2H), 1.34 (s, 12H), 1.05 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.9, 134.5, 130.8, 128.4, 127.5, 126.7, 126.6, 92.9, 83.9, 83.5, 40.0, 31.6, 24.9, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(R)-1-Ethynyl-4-(3-phenylpent-1-yn-1-yl)benzene (51)

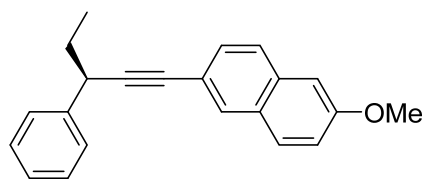


According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (71.9 mg, 0.20 mmol, 1.0 equiv.) and 1,4-diethynylbenzene **2o** (37.8 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **51** as a colorless oil (31.8 mg, 65% yield, 95% *ee*).

HPLC analysis: Chiralcel AD (hexane/*i*-PrOH = 97/3, flow rate 0.7 mL/min, $\lambda = 254$ nm), t_R (minor) = 6.16 min, t_R (major) = 7.61 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.43-7.37 (m, 6H), 7.35-7.32 (m, 2H), 7.27-7.23 (m, 1H), 3.79 (t, $J = 7.0$ Hz, 1H), 3.13 (s, 1H), 1.92-1.81 (m, 2H), 1.05 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.7, 131.9, 131.5, 128.5, 127.5, 126.7, 124.4, 121.3, 93.7, 83.3, 82.9, 78.5, 40.0, 31.5, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-2-Methoxy-6-(3-phenylpent-1-yn-1-yl)naphthalene (52)



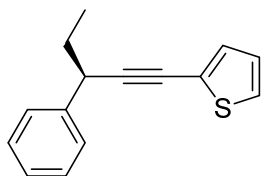
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 2-ethynyl-6-methoxynaphthalene **2p** (36.4 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **52** as a colorless oil (43.9 mg, 73% yield, 95% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 97/3, flow rate 0.8 mL/min, $\lambda = 254$ nm), t_R (major) = 21.20 min, t_R (minor) = 31.44 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.88 (s, 1H), 7.65 (t, $J = 8.6$ Hz, 2H), 7.48-7.44 (m, 3H), 7.35 (t, $J = 7.6$ Hz, 2H), 7.27-7.23 (m, 1H), 7.14-7.08 (m, 2H), 3.90 (s, 3H), 3.82 (t, $J = 7.0$ Hz, 1H), 1.95-1.84 (m, 2H), 1.09 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 158.0, 142.1, 133.8, 131.0, 129.3, 129.1, 128.5, 128.4, 127.6, 126.6, 119.2, 118.8, 105.8, 91.0, 83.8, 55.3, 40.1, 31.7, 11.9. The NMR spectra were in accord with

that reported in literature.^[5]

(R)-2-(3-Phenylpent-1-yn-1-yl)thiophene (53)

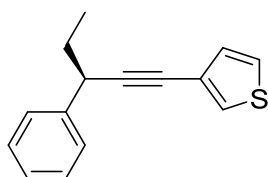


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 2-ethynylthiophene **2q** (21.6 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **53** as a colorless oil (27.6 mg, 61% yield, 96% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), t_R (minor) = 12.52 min, t_R (major) = 13.82 min.

¹H NMR (400 MHz, CDCl₃) δ : 7.40-7.38 (m, 2H), 7.36-7.32 (m, 2H), 7.27-7.23 (m, 1H), 7.20-7.16 (m, 2H), 6.96-6.94 (m, 1H), 3.80 (t, J = 7.0 Hz, 1H), 1.92-1.81 (m, 2H), 1.04 (t, J = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 141.6, 131.2, 128.4, 127.5, 126.8, 126.7, 126.2, 123.9, 95.4, 76.4, 40.2, 31.5, 11.9. The NMR spectra were in accord with that reported in literature.^[5]

(R)-3-(3-Phenylpent-1-yn-1-yl)thiophene (54)

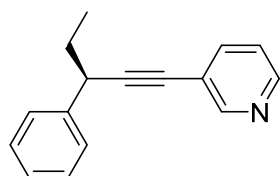


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 3-ethynylthiophene **2c** (21.6 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **54** as a colorless oil (28.1 mg, 62% yield, 97% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 214 nm), t_R (minor) = 17.63 min, t_R (major) = 19.77 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.41-7.39 (m, 3H), 7.35-7.31 (m, 2H), 7.26-7.22 (m, 2H), 7.12-7.10 (m, 1H), 3.76 (t, J = 7.0 Hz, 1H), 1.91-1.80 (m, 2H), 1.04 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.9, 130.1, 128.4, 127.8, 127.5, 126.6, 125.0, 122.8, 91.0, 78.3, 40.0, 31.6, 11.8; HRMS (ESI): m/z calcd for $\text{C}_{15}\text{H}_{15}\text{S}$ $[\text{M}+\text{H}]^+$: 227.0889, found: 227.0888.

(*R*)-4-(3-Phenylpent-1-yn-1-yl)pyridine (**55**)

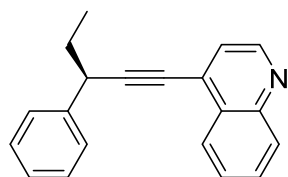


According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 3-ethynylpyridine **2r** (20.6 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/100) to yield the product **55** as a colorless oil (31.4 mg, 71% yield, 96% *ee*).

HPLC analysis: Chiralcel IA (hexane/*i*-PrOH = 97/3, flow rate 0.5 mL/min, λ = 254 nm), t_R (minor) = 12.91 min, t_R (major) = 14.98 min.

^1H NMR (400 MHz, CDCl_3) δ : 8.68 (s, 1H), 8.51 (s, 1H), 7.73-7.70 (m, 1H), 7.42-7.39 (m, 2H), 7.37-7.33 (m, 2H), 7.28-7.21 (m, 2H), 3.80 (t, J = 7.0 Hz, 1H), 1.94-1.83 (m, 2H), 1.06 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 152.4, 148.1, 141.4, 138.5, 128.5, 127.5, 126.8, 122.9, 120.9, 95.2, 80.0, 40.0, 31.4, 11.8; HRMS (ESI): m/z calcd for $\text{C}_{16}\text{H}_{16}\text{N}$ $[\text{M}+\text{H}]^+$: 222.1277, found: 222.1277.

(*R*)-4-(3-Phenylpent-1-yn-1-yl)quinoline (**56**)



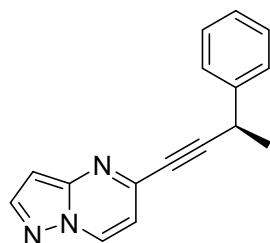
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (107.8 mg, 0.30 mmol, 1.5 equiv.) and 4-ethynylquinoline **2s** (30.6 mg, 0.20 mmol, 1.0 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/30) to yield the product **56** as a colorless oil (35.3 mg, 65% yield, 95% *ee*).

HPLC analysis: Chiralcel OD3 (hexane/*i*-PrOH = 98/2, flow rate 0.3 mL/min, λ = 254 nm), t_R (major) = 30.42 min, t_R (minor) = 32.10 min.

^1H NMR (400 MHz, CDCl_3) δ : 8.85 (d, J = 4.4 Hz, 1H), 8.29 (dd, J = 8.4, 0.8 Hz, 1H), 8.11 (d, J = 8.4 Hz, 1H), 7.76-7.71 (m, 1H), 7.62-7.58 (m, 1H), 7.50-7.47 (m, 3H), 7.41-7.37 (m, 2H), 7.32-7.27 (m, 1H), 3.97 (t, J = 7.0 Hz, 1H), 2.05-1.96 (m, 2H), 1.14 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 149.7, 148.0, 141.1, 130.3, 129.7, 128.6, 128.1, 127.5, 127.04, 126.96, 126.0, 123.8, 101.7, 79.3, 40.4, 31.5, 11.9.

The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-5-(3-Phenylbut-1-yn-1-yl)pyrazolo[1,5- α]pyrimidine (**57**)



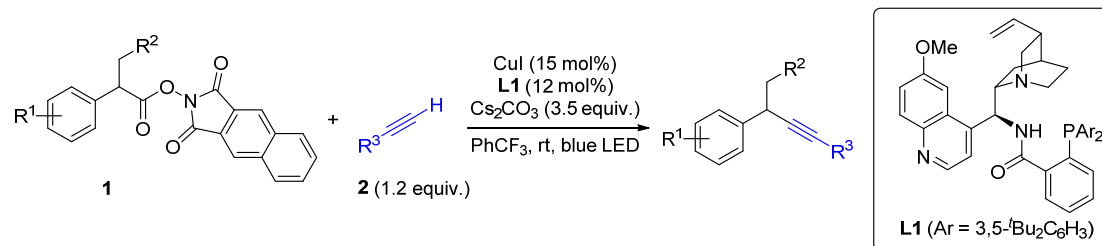
According to **General procedure 3** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylpropanoate **1ah** (51.8 mg, 0.15 mmol, 1.5 equiv.), 5-ethynylpyrazolo[1,5- α]pyrimidine **2t** which was synthesized according to the reference^[3] (14.3 mg, 0.1 mmol, 1.0 equiv.), CuI (4.3 mg, 0.0225 mmol, 15 mol%) and **L1** (22.6 mg, 0.027 mmol, 18 mol%) for 4 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/2) to yield the product **57** as a colorless oil (11.1 mg, 45% yield, 93% *ee*).

HPLC analysis: Chiralcel AD3 (hexane/*i*-PrOH = 95/5, flow rate 0.3 mL/min, λ = 254 nm), t_R (minor) = 48.41 min, t_R (major) = 51.19 min.

^1H NMR (400 MHz, CDCl_3) δ : 8.58 (d, J = 7.2 Hz, 1H), 8.12 (s, 1H), 7.44 (d, J = 7.6

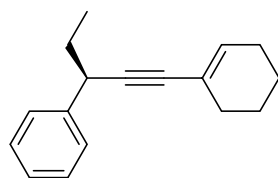
Hz, 2H), 7.36 (t, $J = 7.4$ Hz, 2H), 7.27 (t, $J = 7.2$ Hz, 1H), 6.85 (d, $J = 7.2$ Hz, 1H), 6.68 (s, 1H), 4.05 (q, $J = 7.2$ Hz, 1H), 1.64 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 148.1, 145.6, 142.4, 141.8, 134.4, 128.7, 127.0, 126.9, 110.7, 97.2, 96.9, 81.0, 32.4, 23.7. The NMR spectra were in accord with that reported in literature.^[5]

General procedure 4:



CuI (5.7 mg, 0.03 mmol, 15 mol%), **L1** (20.1 mg, 0.024 mmol, 12 mol%), Cs_2CO_3 (228.1 mg, 0.70 mmol, 3.5 equiv.), *N*-hydroxyphthalimide esters **1** (0.20 mmol, 1.0 equiv.) and anhydrous PhCF_3 (4.0 mL) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere. Then, alkyne **2** (0.24 mmol, 1.2 equiv.) was added to the mixture. The reaction mixture was stirred under the irradiation of a 24 W blue LED for 4 d at room temperature. After the completion of reaction, the reaction mixture was filtered and the precipitate was washed by EtOAc. The filtrate was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography on silica gel to afford the alkylation product.

(*R*)-(1-(Cyclohex-1-en-1-yl)pent-1-yn-3-yl)benzene (**58**)



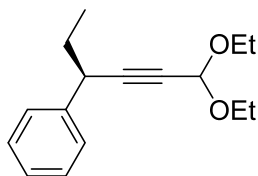
According to **General procedure 4** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (71.9 mg, 0.20 mmol, 1.0 equiv.) and 1-ethynylcyclohex-1-ene **2u** (25.5 mg, 0.24 mmol, 1.2 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **58** as a colorless oil (14.8 mg, 33% yield, 97%

ee).

HPLC analysis: Chiralcel OD3 (hexane/*i*-PrOH = 100/0, flow rate 0.2 mL/min, λ = 214 nm), t_R (minor) = 31.14 min, t_R (major) = 38.85 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.37-7.30 (m, 4H), 7.22 (t, J = 7.0 Hz, 1H), 6.10-6.08 (m, 1H), 3.68 (t, J = 7.0 Hz, 1H), 2.17-2.15 (m, 2H), 2.12-2.09 (m, 2H), 1.84-1.72 (m, 2H), 1.68-1.56 (m, 4H), 1.00 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 142.4, 133.5, 128.3, 127.5, 126.4, 120.9, 88.5, 85.2, 39.8, 31.8, 29.6, 25.6, 22.4, 21.6, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-(6,6-Diethoxyhex-4-yn-3-yl)benzene (59)

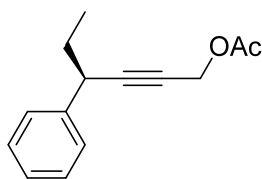


According to **General procedure 4** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (71.9 mg, 0.20 mmol, 1.0 equiv.) and 3,3-diethoxyprop-1-yne **2v** (30.8 mg, 0.24 mmol, 1.2 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/50) to yield the product **59** as a colorless oil (24.1 mg, 49% yield, 97% *ee*).

HPLC analysis: Chiralcel OD3 (hexane/*i*-PrOH = 99/1, flow rate 0.3 mL/min, λ = 214 nm), t_R (major) = 20.31 min, t_R (minor) = 25.24 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.35-7.29 (m, 4H), 7.23 (t, J = 6.6 Hz, 1H), 5.34 (s, 1H), 3.81-3.72 (m, 2H), 3.65-3.56 (m, 3H), 1.84-1.77 (m, 2H), 1.26-1.22 (m, 6H), 0.99 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 141.2, 128.3, 127.5, 126.7, 91.5, 87.4, 78.5, 60.7, 60.6, 39.2, 31.2, 15.1, 11.7. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-4-Phenylhex-2-yn-1-yl acetate (60)

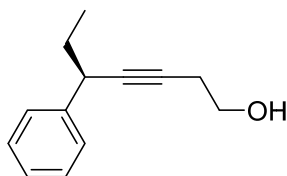


According to **General procedure 4** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (71.9 mg, 0.20 mmol, 1.0 equiv.) and prop-2-yn-1-yl acetate **2w** (23.5 mg, 0.24 mmol, 1.2 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/50) to yield the product **60** as a colorless oil (22.1 mg, 51% yield, 95% *ee*).

HPLC analysis: Chiralcel OD3 (hexane/*i*-PrOH = 99/1, flow rate 0.3 mL/min, λ = 214 nm), t_R (major) = 29.92 min, t_R (minor) = 34.28 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.33-7.29 (m, 4H), 7.26-7.21 (m, 1H), 4.74 (d, J = 2.0 Hz, 2H), 3.62-3.58 (m, 1H), 2.10 (s, 3H), 1.82-1.75 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 170.4, 141.3, 128.4, 127.4, 126.7, 88.8, 52.8, 39.3, 31.2, 20.8, 11.7. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-5-Phenylhept-3-yn-1-ol (**61**)



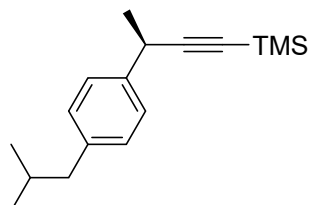
According to **General procedure 4** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (71.9 mg, 0.20 mmol, 1.0 equiv.) and but-3-yn-1-ol **2x** (16.8 mg, 0.24 mmol, 1.2 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1/10) to yield the product **61** as a colorless oil (11.3 mg, 30% yield, 97% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 95/5, flow rate 0.7 mL/min, λ = 214 nm), t_R (major) = 11.42 min, t_R (minor) = 12.69 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.35-7.30 (m, 4H), 7.23 (t, J = 6.6 Hz, 1H), 3.72 (t, J = 5.8 Hz, 2H), 3.56 (t, J = 7.0 Hz, 1H), 2.52 (td, J = 6.2, 1.6 Hz, 2H), 1.87 (s, 1H),

1.82-1.70 (m, 2H), 0.98 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 142.3, 128.4, 127.4, 126.6, 84.1, 79.2, 61.4, 39.4, 31.6, 23.3, 11.8. The NMR spectra were in accord with that reported in literature.^[5]

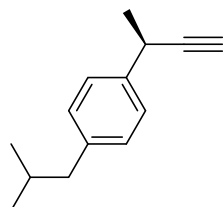
(*R*)-(3-(4-Isobutylphenyl)but-1-yn-1-yl)trimethylsilane (62)



According to **General procedure 4** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-isobutylphenyl)propanoate **1b** (80.3 mg, 0.20 mmol, 1.0 equiv.) and ethynyltrimethylsilane **2y** (23.6 mg, 0.24 mmol, 1.2 equiv.) for 4 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **62** as a colorless oil (25.8 mg, 50% yield).

^1H NMR (400 MHz, CDCl_3) δ : 7.28 (d, $J = 8.0$ Hz, 2H), 7.10 (d, $J = 8.0$ Hz, 2H), 3.77 (q, $J = 7.2$ Hz, 1H), 2.46 (d, $J = 7.2$ Hz, 2H), 1.91-1.80 (m, 1H), 1.47 (d, $J = 7.2$ Hz, 3H), 0.91 (d, $J = 6.4$ Hz, 6H), 0.19 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.2, 139.9, 129.2, 126.5, 109.8, 86.0, 45.0, 32.4, 30.2, 24.7, 22.40, 22.38, 0.2. The NMR spectra were in accord with that reported in literature.^[5]

(*S*)-1-(But-3-yn-2-yl)-4-isobutylbenzene (62')

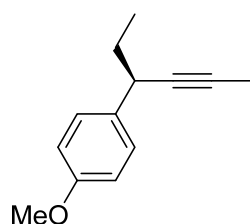


To a stirred solution of **62** (24.4 mg, 0.094 mmol, 1.0 equiv.) in MeOH (1.5 mL) was added K_2CO_3 (26 mg, 0.189 mmol, 2.0 equiv.) and the reaction mixture was stirred for 4 h. After completion of reaction, the mixture was concentrated under reduced pressure and purified by column chromatography (petroleum ether) to yield the desired product **62'** as a colorless oil (15.6 mg, 89% yield, 94% *ee*).

HPLC analysis: Chiralcel OD3 (hexane/*i*-PrOH = 100/0, flow rate 0.3 mL/min, λ = 214 nm), t_R (major) = 17.64 min, t_R (minor) = 22.26 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.30 (d, J = 8.0 Hz, 2H), 7.11 (d, J = 8.0 Hz, 2H), 3.78-3.72 (m, 1H), 2.46 (d, J = 7.2 Hz, 2H), 2.26 (d, J = 2.4 Hz, 1H), 1.90-1.80 (m, 1H), 1.51 (d, J = 7.2 Hz, 3H), 0.91 (d, J = 6.4 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 140.1, 139.8, 129.3, 126.5, 87.4, 69.9, 45.0, 31.2, 30.2, 24.2, 22.4. The NMR spectra were in accord with that reported in literature.^[5]

(*R*)-1-(Hex-4-yn-3-yl)-4-methoxybenzene (63)



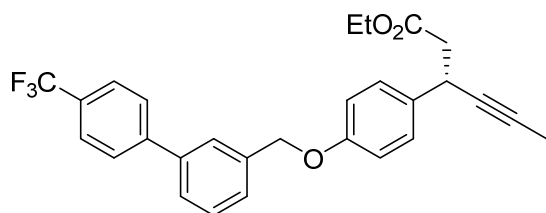
According to **General procedure 4** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-(4-methoxyphenyl)butanoate **S34** (39.0 mg, 0.10 mmol, 1.0 equiv.) and prop-1-yne **2z** (0.5 mL, 0.50 mmol, 5.0 equiv., 1.0 M in THF) for 4 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **63** as a colorless oil (10.9 mg, 58% yield, 93% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 230 nm), t_R (minor) = 11.31 min, t_R (major) = 17.02 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.26-7.24 (m, 2H), 6.85 (d, J = 8.4 Hz, 2H), 3.79 (s, 3H), 3.45 (t, J = 6.4 Hz, 1H), 1.86 (s, 3H), 1.74-1.67 (m, 2H), 0.95 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 158.2, 134.9, 128.4, 113.7, 81.1, 78.1, 55.3, 38.6, 31.7, 11.8, 3.6; HRMS (ESI): m/z calcd for $\text{C}_{13}\text{H}_{17}\text{O}$ $[\text{M}+\text{H}]^+$: 189.1274, found: 189.1273.

Ethyl

(*S*)-3-(4-((4'-(trifluoromethyl)-[1,1'-biphenyl]-3-yl)methoxy)phenyl)hex-4-ynoate (64)

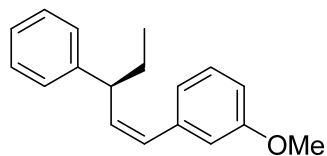


According to **General procedure 2** with 1-(1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl) 4-ethyl 2-(4-((4'-(trifluoromethyl)-[1,1'-biphenyl]-3-yl)methoxy)phenyl)succinate **S64** (66.8 mg, 0.1 mmol, 1.0 equiv.), prop-1-yne **2z** (0.5 mL, 0.5 mmol, 5.0 equiv., 1.0 M in THF), CuI (3.8 mg, 0.02 mmol, 20 mol%) and **L3** (19.3 mg, 0.024 mmol, 24 mol%) for 3 d, the reaction mixture was purified by column chromatography on silica gel (dichloromethane/petroleum ether = 1/1.5) to yield the product **64** as a colorless oil (12.6 mg, 27% yield, 76% *ee*).

HPLC analysis: Chiralcel IB (hexane/*i*-PrOH = 98/2, flow rate 1.0 mL/min, $\lambda = 254$ nm), t_R (minor) = 10.55 min, t_R (major) = 11.62 min.

^1H NMR (400 MHz, CDCl_3) δ : 7.70 (s, 4H), 7.65 (s, 1H), 7.56 (d, $J = 7.2$ Hz, 1H), 7.48 (d, $J = 7.6$ Hz, 2H), 7.30 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 8.4$ Hz, 2H), 5.12 (s, 2H), 4.16-4.06 (m, 3H), 2.77-2.71 (m, 1H), 2.67-2.61 (m, 1H), 1.82 (d, $J = 2.4$ Hz, 3H), 1.21 (t, $J = 7.0$ Hz, 3H); ^{19}F NMR (376 MHz, CDCl_3) δ : -62.4. The NMR spectra were in accord with that reported in literature.^[5]

(*S,Z*)-1-Methoxy-3-(3-phenylpent-1-en-1-yl)benzene (65)



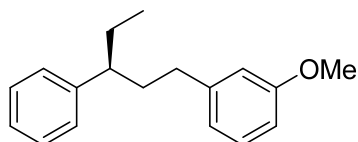
To a solution of IPrCuCl (7.3 mg, 0.015 mmol, 10 mol%), Pd(OAc)₂ (1.7 mg, 0.0075 mmol, 5 mol%), **L** (5.7 mg, 0.015 mmol, 10 mol%), NaO^tBu (28.8 mg, 0.3 mmol, 2 equiv.), MeOH (24.0 mg, 0.75 mmol, 5 equiv.) and **39** (37.6 mg, 0.15 mmol, 1.0 equiv., 97% *ee*) in toluene (1.5 mL) was added TMDSO (1,1,3,3-tetramethyldisiloxane) (40.3 mg, 0.3 mmol, 2 equiv.) under argon atmosphere. Then, the reaction mixture was stirred at 60 °C for 4 h. Subsequently, the reaction mixture was filtered through a short plug of silica gel with EtOAc (3 × 10 mL). The

organic solvent was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography (petroleum ether) on silica gel to afford the product **65** as a colorless oil (30.7 mg, 81% yield, 97% *ee*).^[9]

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99/1, flow rate 1.0 mL/min, λ = 240 nm), t_R (minor) = 10.92 min, t_R (major) = 22.96 min.

¹H NMR (400 MHz, CDCl₃) δ : 7.31 (t, J = 7.4 Hz, 2H), 7.25-7.18 (m, 4H), 6.83 (d, J = 7.6 Hz, 1H), 6.80-6.77 (m, 2H), 6.51 (d, J = 11.6 Hz, 1H), 5.88-5.82 (m, 1H), 3.75 (s, 3H), 3.73-3.68 (m, 1H), 1.80-1.66 (m, 2H), 0.84 (t, J = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 159.4, 145.2, 138.9, 136.0, 129.1, 128.8, 128.5, 127.4, 126.1, 121.2, 113.9, 112.5, 55.1, 45.5, 30.7, 12.0; HRMS (ESI): m/z calcd for C₁₈H₂₁O [M+H]⁺: 253.1587, found: 253.1585.

(*S*)-1-Methoxy-3-(3-phenylpentyl)benzene (**66**)



To a solution of **39** (37.6 mg, 0.15 mmol, 1.0 equiv., 97% *ee*) in THF (2.5 mL) was added Pd/C (10.0 mg, 10% w/w Pd on carbon) under argon atmosphere. Subsequently, the reaction flask was evacuated and refilled with hydrogen through a balloon. The reaction mixture was stirred at room temperature under the hydrogen atmosphere for 4 h. Then, the reaction mixture was filtered and rinsed with EtOAc (3 \times 20 mL). The filtrate was concentrated by rotary evaporator under vacuum and the residue was purified by column chromatography on silica gel (petroleum ether) to give the product **66** as a colorless oil (31.7 mg, 83% yield, 96% *ee*).

HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99/1, flow rate 1.0 mL/min, λ = 214 nm), t_R (minor) = 10.65 min, t_R (major) = 13.23 min.

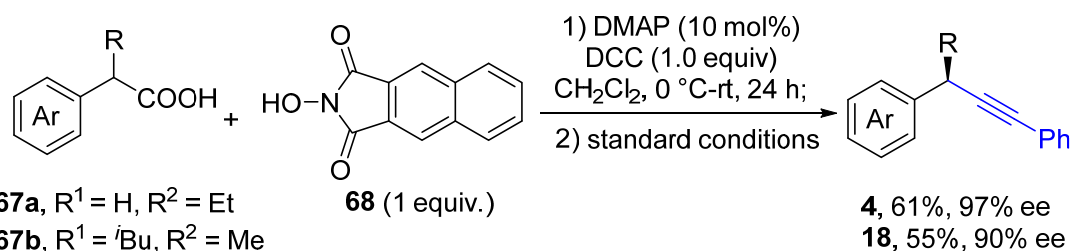
¹H NMR (400 MHz, CDCl₃) δ : 7.31 (t, J = 7.4 Hz, 2H), 7.24-7.14 (m, 4H), 6.71-6.69 (m, 2H), 6.65 (s, 1H), 3.77 (s, 3H), 2.48-2.38 (m, 3H), 2.02-1.83 (m, 2H), 1.74-1.66 (m, 1H), 1.61-1.54 (m, 1H), 0.76 (t, J = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ : 159.5, 145.4, 144.3, 129.1, 128.3, 127.8, 125.9, 120.8, 114.1, 110.9, 55.1, 47.3, 38.0, 33.9, 29.8, 12.1; HRMS (ESI): m/z calcd for C₁₈H₂₃O [M+H]⁺: 255.1743, found:

255.1740.

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9. Synthetic applications

9.1 One-pot synthesis



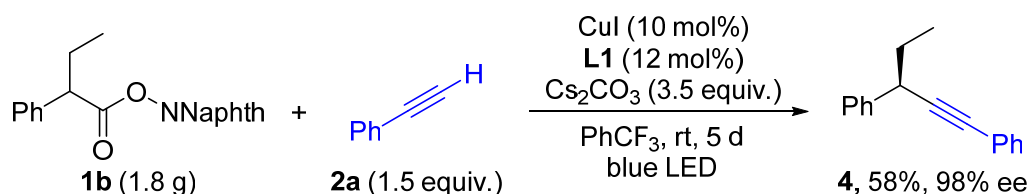
Step 1: According to **General procedure 1** with alkyl carboxylic acids **67a** and **67b** (0.20 mmol, 1.0 equiv.) and 2-hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione **68** (42.6 mg, 0.20 mmol, 1.0 equiv.) for 24 h, the reaction mixture was filtrated through a pad of diatomite. The precipitate was washed with CH₂Cl₂ (3 × 50 mL). The filtrate was removed by rotary evaporator under vacuum to give the crude ester without further purification.

Step 2: According to **General procedure 2** with the crude ester (0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether) to yield the product **4** (colorless oil, 26.9 mg, 61% yield, 97% *ee*) and **18** (colorless oil, 28.9 mg, 55% yield, 90% *ee*), respectively.

HPLC analysis of **4**: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 1.0 mL/min, λ = 254 nm), *t_R* (minor) = 11.48 min, *t_R* (major) = 16.96 min.

HPLC analysis of **18**: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, λ = 254 nm), *t_R* (minor) = 15.97 min, *t_R* (major) = 26.49 min.

9.2 Large-scale synthesis



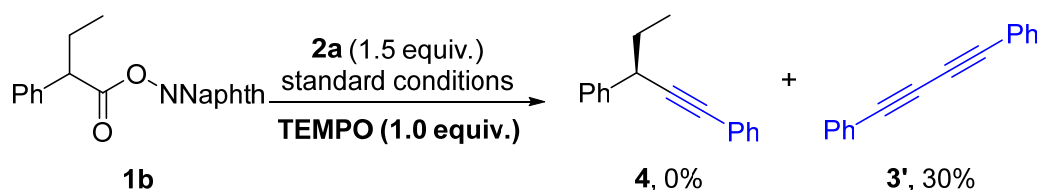
CuI (95.2 mg, 0.5 mmol, 10 mol%), **L1** (502 mg, 0.6 mmol, 12 mol%), Cs₂CO₃ (5.7 g, 17.5 mmol, 3.5 equiv.), 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (1.8 g, 5.0 mmol, 1.0 equiv.) and anhydrous PhCF₃ (100 mL) were sequentially added to an oven-dried flask equipped with a magnetic stir bar

under argon atmosphere. Then, ethynylbenzene **2a** (766.1 mg, 7.5 mmol, 1.5 equiv.) was added to the mixture. The reaction mixture was stirred under the irradiation of a 24 W blue LED for 5 d at room temperature. After the completion of reaction, the reaction mixture was filtered and the precipitate was washed by EtOAc (3×30 mL). The filtrate was removed by rotary evaporator under vacuum and the residue was purified by flash column chromatography on silica gel with petroleum ether to afford the product **4** as a colorless oil (638.9 mg, 58% yield, 98% *ee*).

HPLC analysis: Chiralcel ODH (hexane/*i*-PrOH = 99.5/0.5, flow rate 1.0 mL/min, $\lambda = 254$ nm), t_R (minor) = 11.16 min, t_R (major) = 16.35 min.

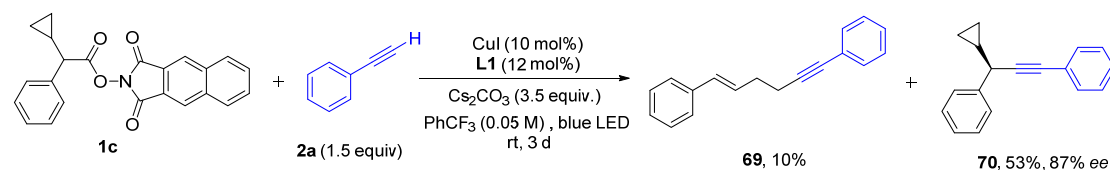
10. Mechanistic Study

10.1 Radical-trapping experiment



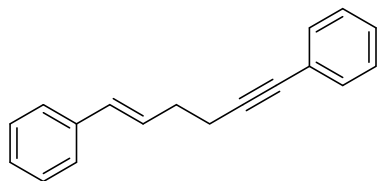
CuI (3.8 mg, 0.02 mmol, 10 mol%), **L1** (20.1 mg, 0.024 mmol, 12 mol%), Cs₂CO₃ (228.1 mg, 0.70 mmol, 3.5 equiv.), 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-phenylbutanoate **1b** (71.9 mg, 0.20 mmol, 1.0 equiv.), TEMPO (31.2 mg, 0.20 mmol 1.0 equiv.) and anhydrous PhCF₃ (4.0 mL) were sequentially added to an oven-dried Schlenk tube equipped with a magnetic stir bar under argon atmosphere. Then, ethynylbenzene **2a** (30.6 mg, 0.30 mmol, 1.5 equiv.) was added to the mixture. The reaction mixture was stirred under the irradiation of a 24 W blue LED for 3 d at room temperature. After the completion of reaction, the reaction mixture was filtered and the precipitate was washed by EtOAc. The filtrate was removed by rotary evaporator under vacuum and the residue was analyzed by ¹H NMR. **4** was not detected in the reaction mixture. Furthermore, **3'** was isolated as a colorless solid (9.1 mg, 30% yield) and 2-phenylbutanoic acid via the hydrolysis of **1b** could be detected.

10.2 Radical clock experiments



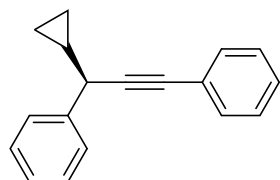
According to **General procedure 2** with 1,3-dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl 2-cyclopropyl-2-phenylacetate **1c** (74.3 mg, 0.20 mmol, 1.0 equiv.) and ethynylbenzene **2a** (30.6 mg, 0.3 mmol, 1.5 equiv.) for 3 d, the reaction mixture was purified by column chromatography on silica gel (petroleum ether).

(*E*)-Hex-1-en-5-yne-1,6-diylidibenzene (69)



69 was isolated as a colorless oil (4.6 mg, 10% yield). ^1H NMR (400 MHz, CDCl_3) δ : 7.41-7.36 (m, 4H), 7.32-7.26 (m, 5H), 7.21 (t, $J = 7.2$ Hz, 1H), 6.50 (d, $J = 16.0$ Hz, 1H), 6.36-6.29 (m, 1H), 2.60-2.52 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ : 137.5, 131.6, 131.1, 128.8, 128.5, 128.2, 127.6, 127.1, 126.1, 123.9, 89.4, 81.2, 32.3, 19.8; HRMS (ESI): m/z calcd for $\text{C}_{18}\text{H}_{17}$ $[\text{M}+\text{H}]^+$: 233.1325, found: 233.1324.

(R)-(3-Cyclopropylprop-1-yne-1,3-diyl)dibenzene (70)



70 was isolated as a colorless oil (24.6 mg, 53% yield, 87% *ee*). HPLC analysis: Chiralcel OJH (hexane/*i*-PrOH = 99.5/0.5, flow rate 0.7 mL/min, $\lambda = 254$ nm), t_{R} (minor) = 22.39 min, t_{R} (major) = 26.84 min; ^1H NMR (400 MHz, CDCl_3) δ : 7.51-7.49 (m, 2H), 7.46-7.43 (m, 2H), 7.38-7.35 (m, 2H), 7.31-7.27 (m, 4H), 3.70 (d, $J = 6.4$ Hz, 1H), 1.28-1.20 (m, 1H), 0.63-0.52 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ : 142.0, 131.7, 128.4, 128.2, 127.8, 127.5, 126.8, 123.6, 89.6, 83.2, 41.3, 17.5, 4.1, 3.1. The NMR spectra were in accord with that reported in literature.^[3]

10.3 Stern-Volmer luminescence quenching experiments

Emission intensities were recorded using FLS-980 Spectrofluorometer equipped with MCP-PMT detector from Edinburgh Instruments for all experiments. The solution was prepared by adding Cu(I)-phenylacetylide, an appropriate amount of quencher, and PhCF_3 to a 5.0 mL of quartz cuvette. The concentration of Cu(I)-phenylacetylide was 5.0×10^{-3} M. The cuvette was sealed with a septum. After degassing with nitrogen for 30 min, the solution was irradiated at 466 nm, and emission was detected at 582 nm.

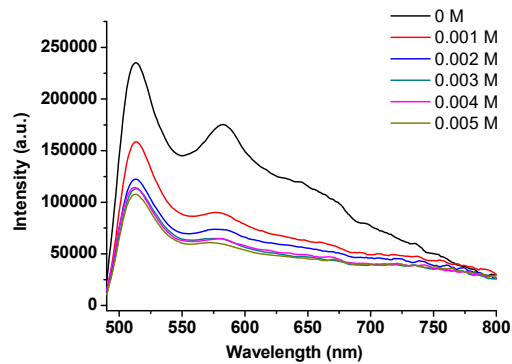


Figure S1a. Cu(I)-phenylacetylide emission quenching by **1b**.

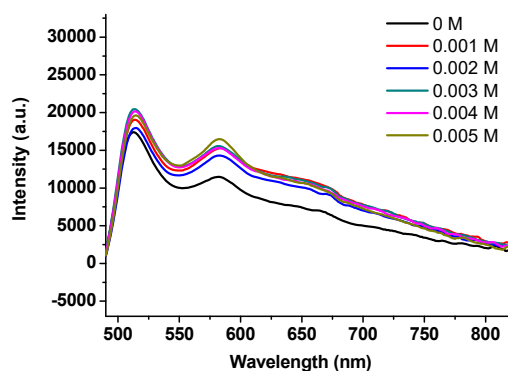


Figure S1b. Cu(I)-phenylacetylide emission quenching by ethynylbenzene (**2a**).

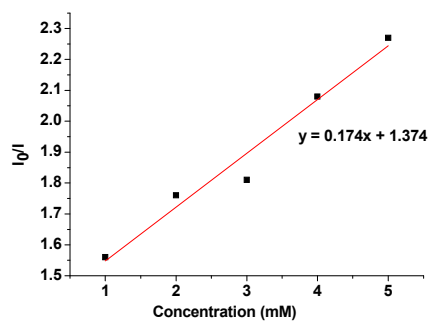
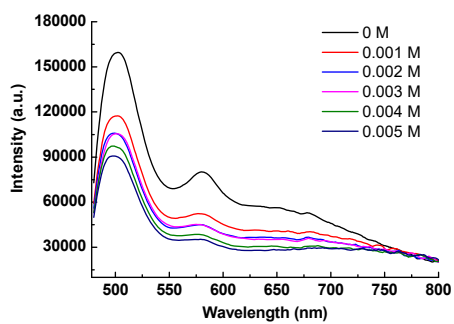


Figure S1c. Cu(I)-phenylacetylide emission quenching by **1aa**.

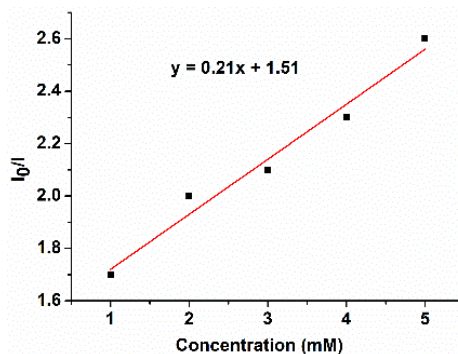
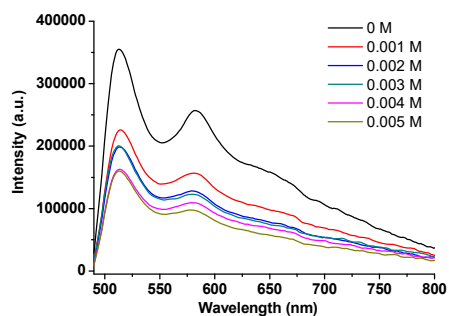


Figure S1d. Cu(I)-phenylacetylide emission quenching by **1ab**.

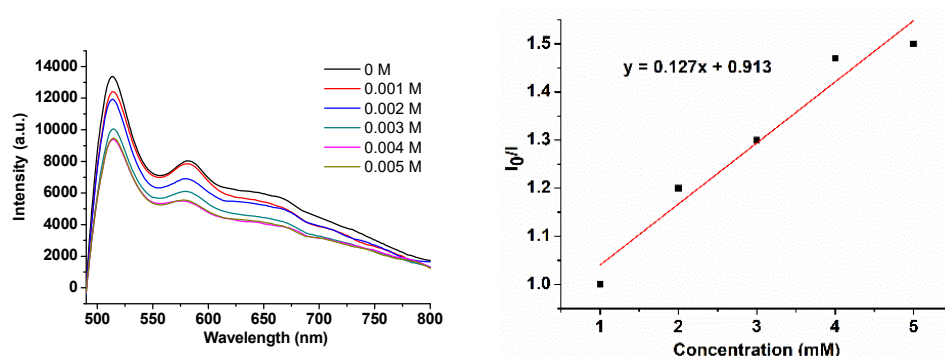


Figure S1e. Cu(I)-phenylacetylide emission quenching by **1ac**.

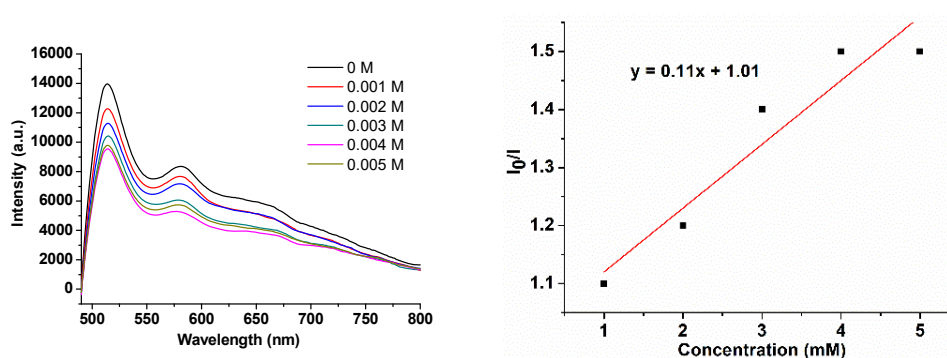


Figure S1f. Cu(I)-phenylacetylide emission quenching by **1ad**.

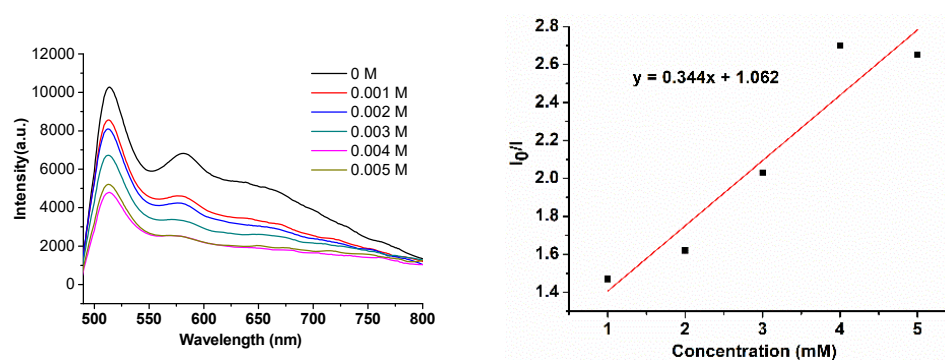


Figure S1g. Cu(I)-phenylacetylide emission quenching by **1ae**.

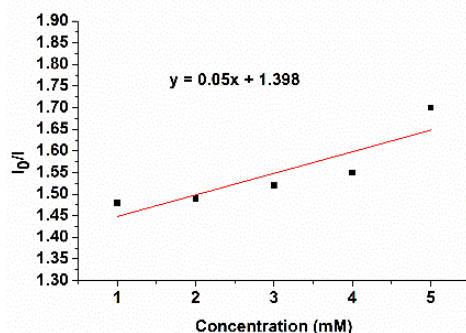
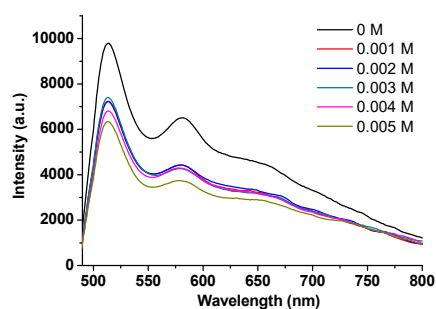


Figure S1h. Cu(I)-phenylacetylide emission quenching by **1af**.

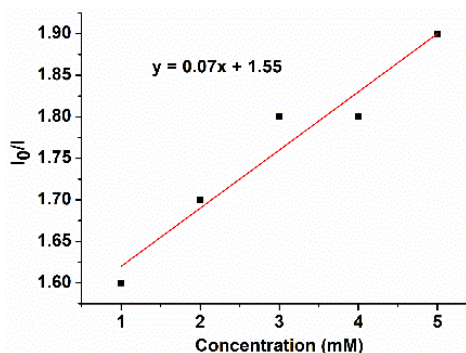
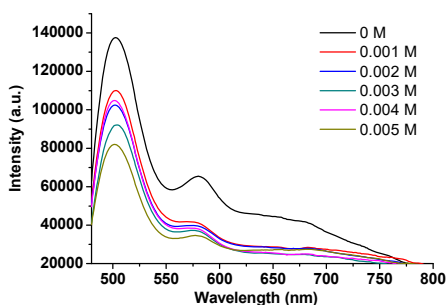


Figure S1i. Cu(I)-phenylacetylide emission quenching by **1ag**.

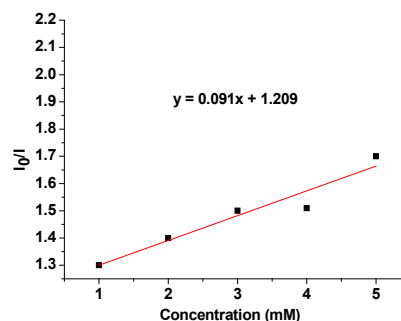
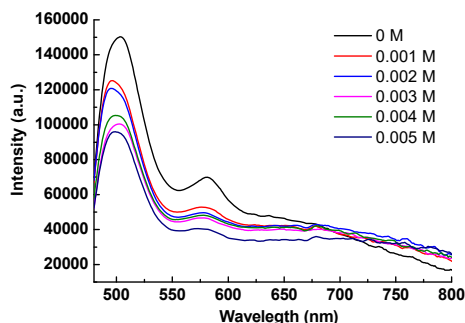


Figure S1j. Cu(I)-phenylacetylide emission quenching by **1ah**.

10.4 Cyclic voltammetry experiments

Cyclic voltammetry experiment was performed using a CH Instruments Electrochemical Workstation model PalmSens4. The electrochemical cell was equipped with a glassy carbon working electrode, a Pt mesh counter electrode, and a Ag/AgNO₃ (10 mM AgNO₃ in MeCN) reference electrode. A solution of **1b** in MeCN (0.001 M) was tested with 0.1 M Bu₄NPF₆ as the supporting electrolyte and ferrocene was using as an internal standard. Scan rate = 0.1 V/s, 2 sweep segments, a sample

interval of 0.001 V.

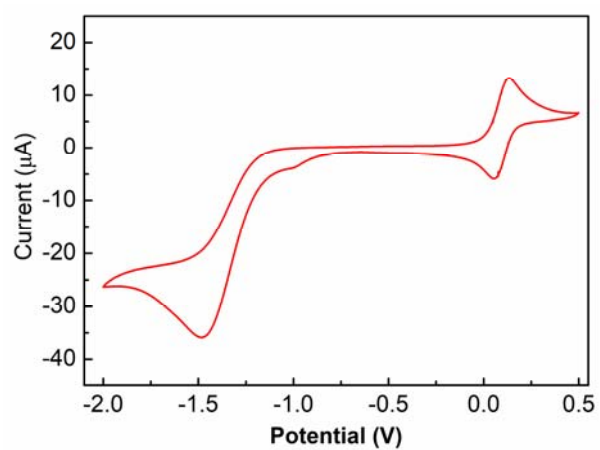
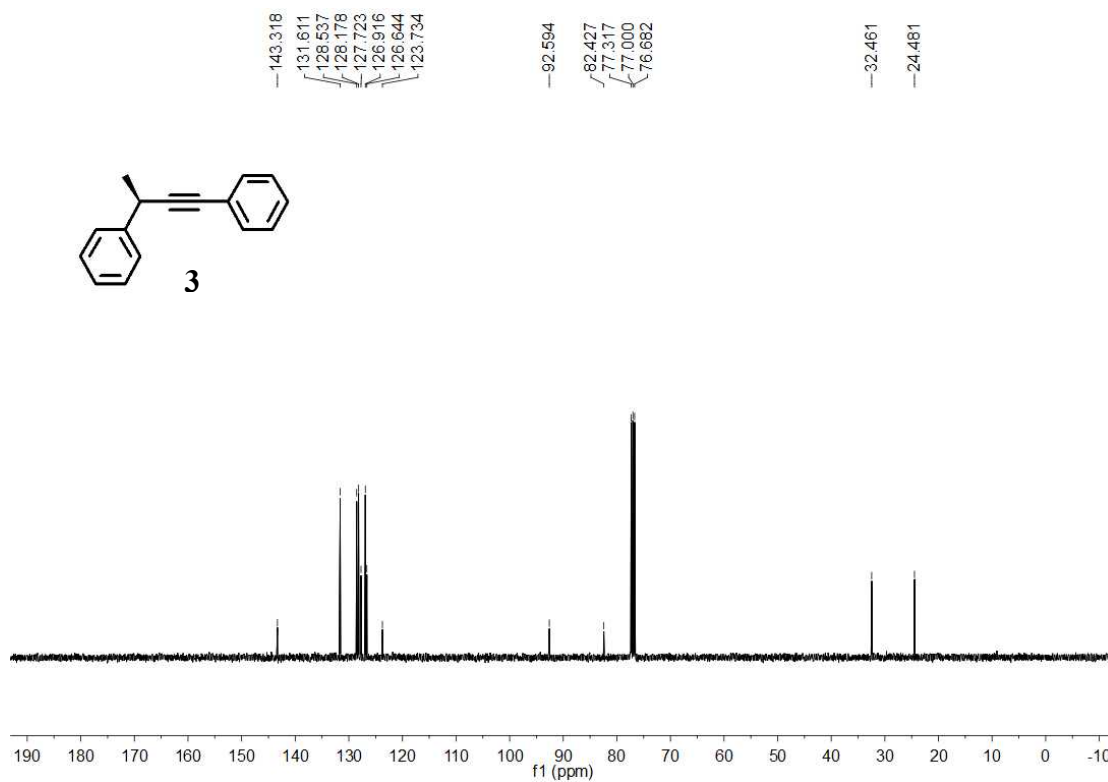
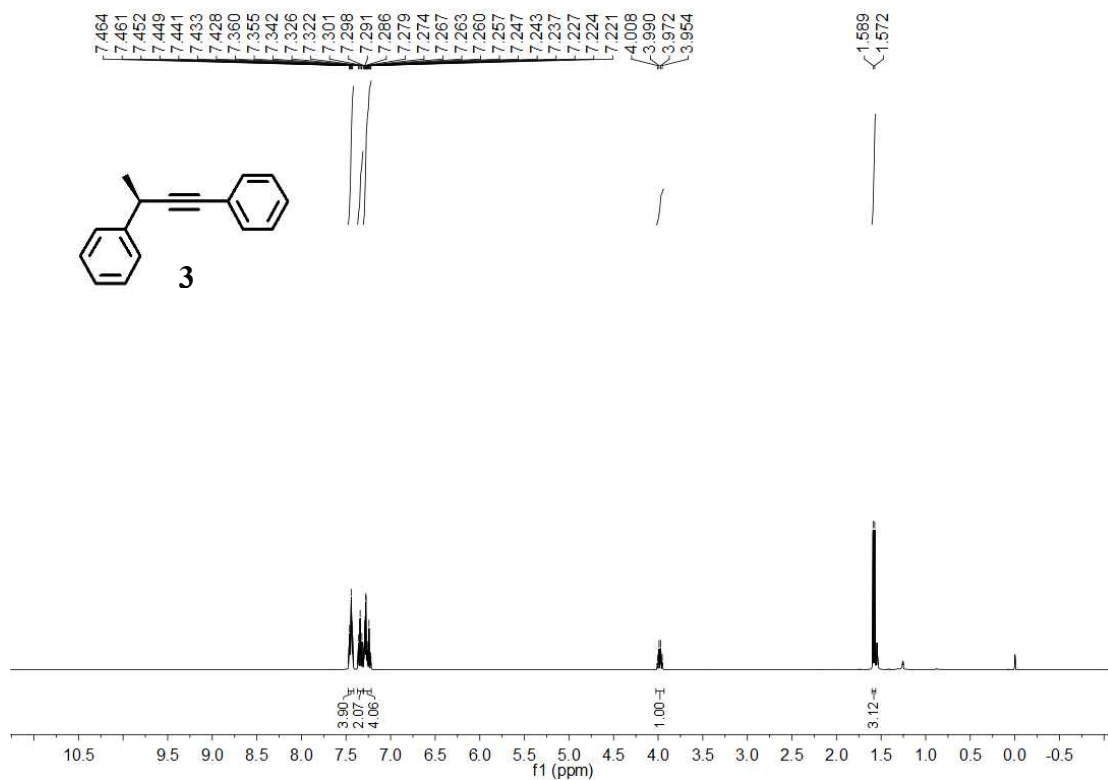


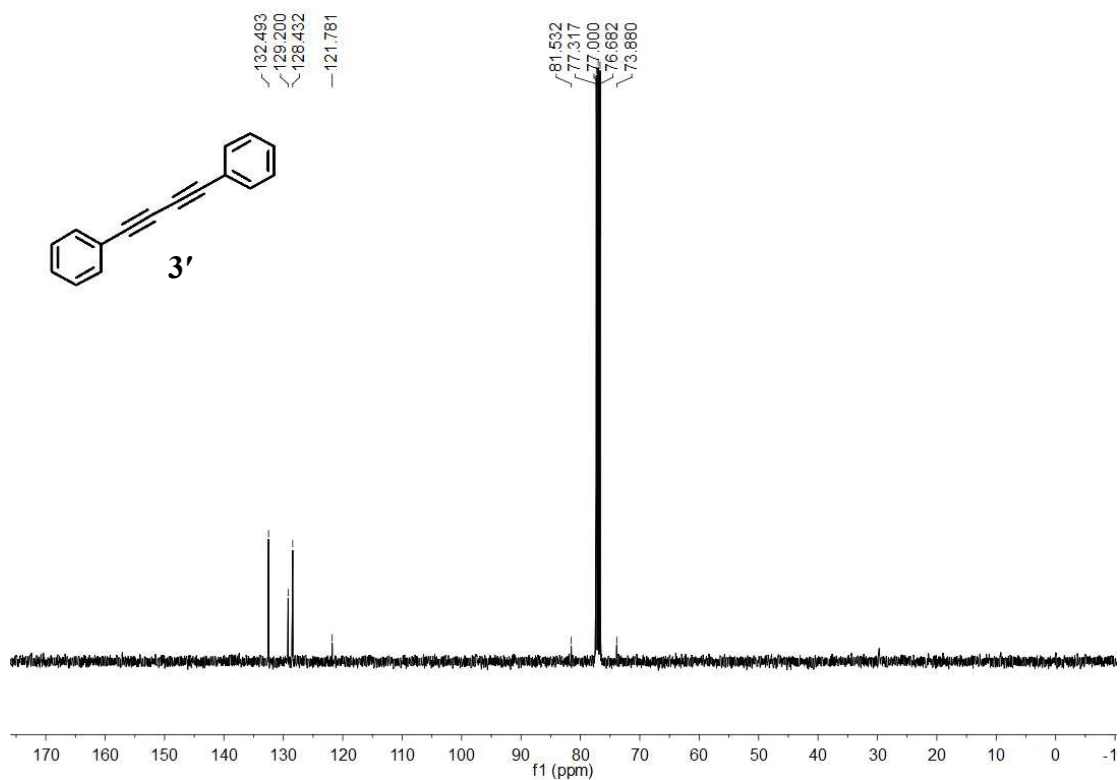
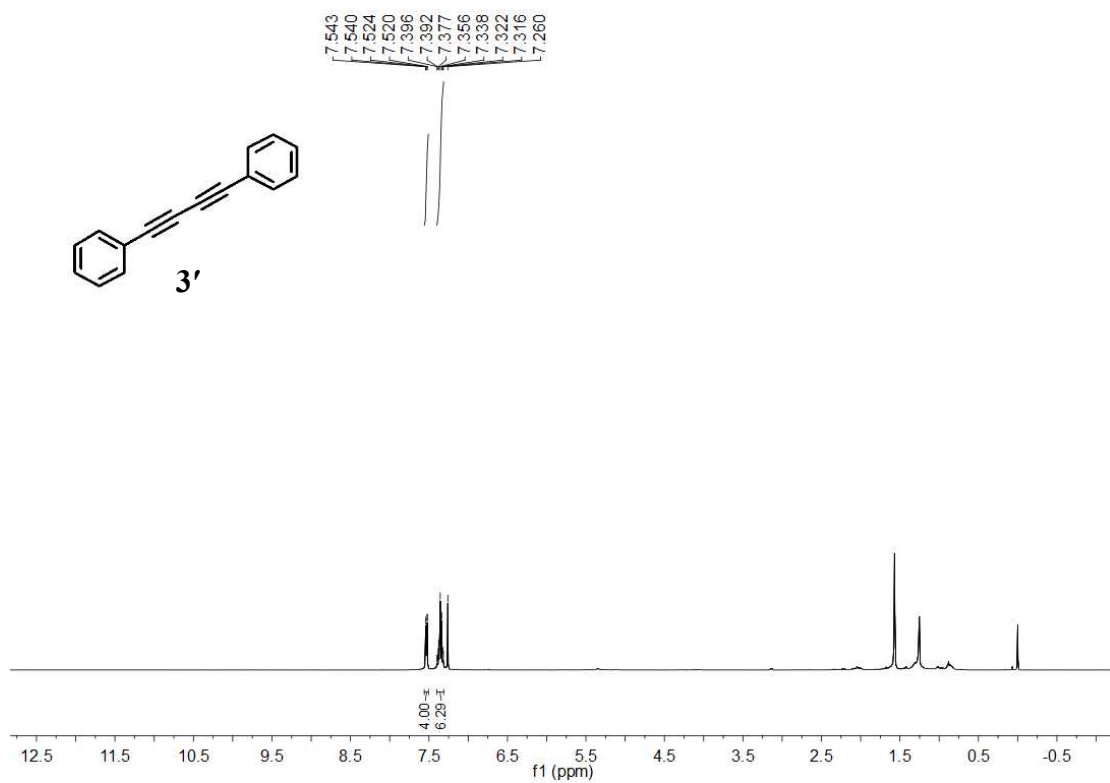
Figure S1. Cyclic voltammogram of **1b** in MeCN. $E_p = -1.567$ V vs. Fc/Fc⁺. E_p (vs. SCE) = -1.567 V + 0.393 V = -1.174 V.

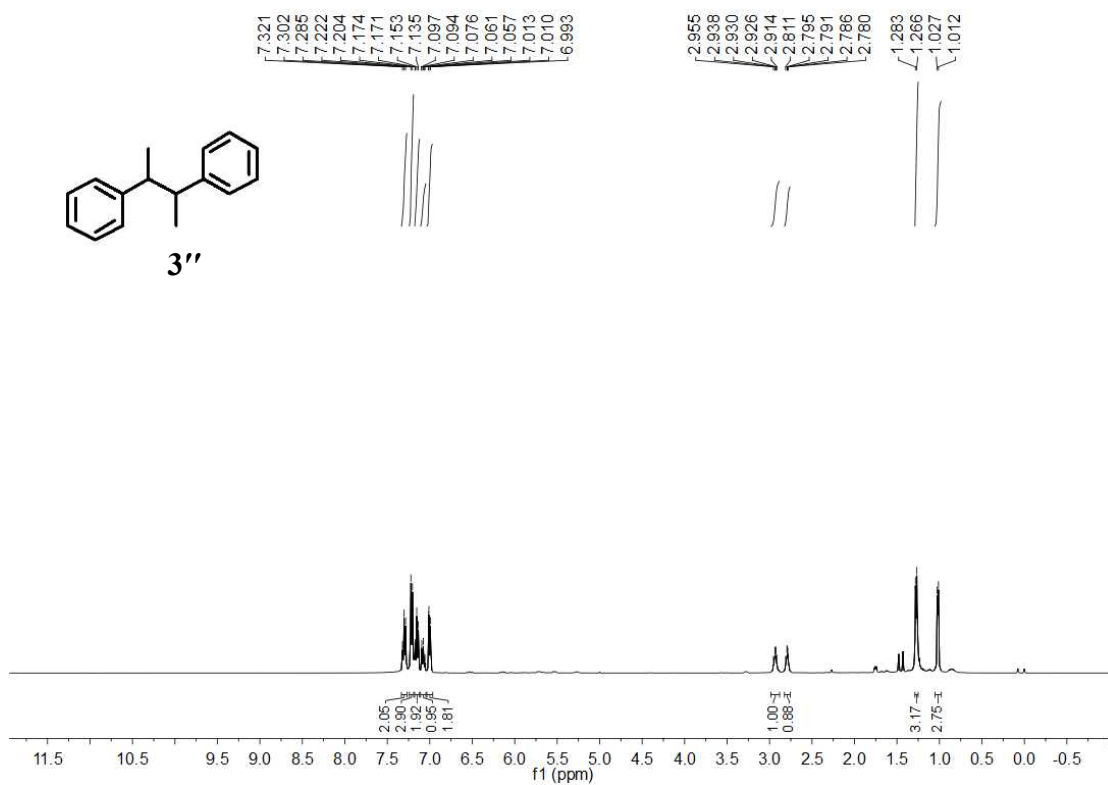
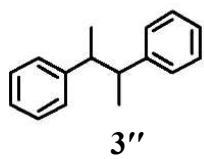
11. References

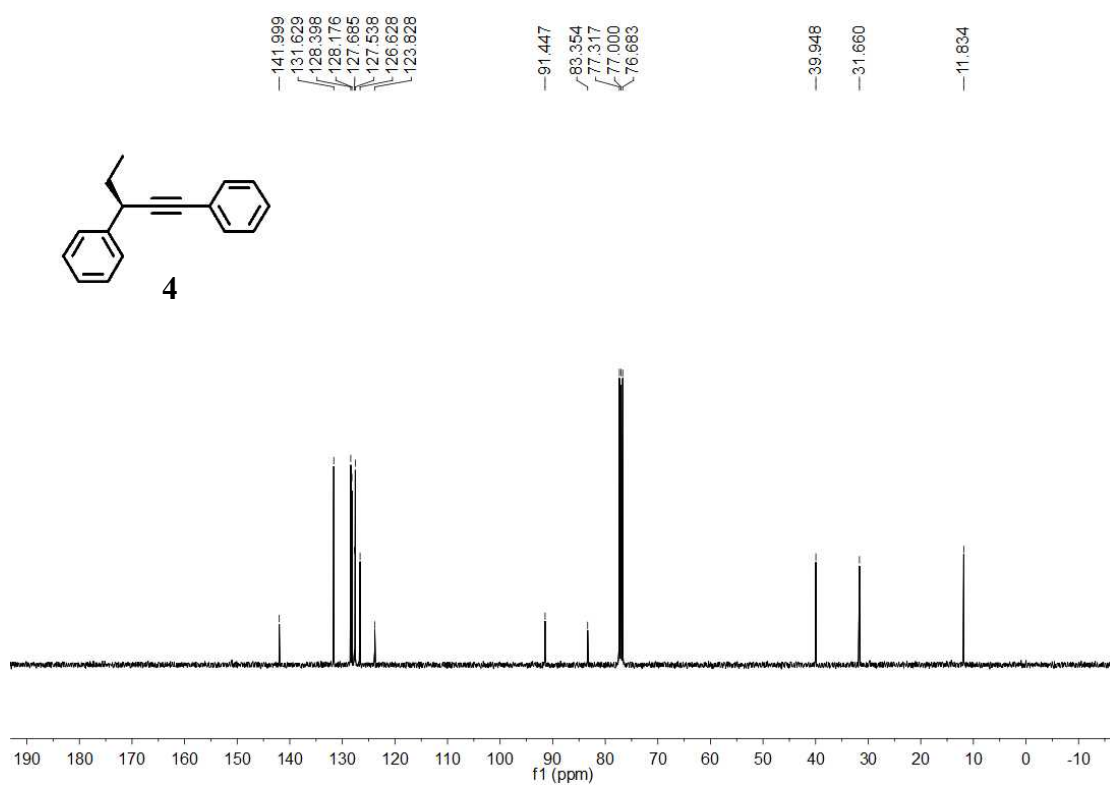
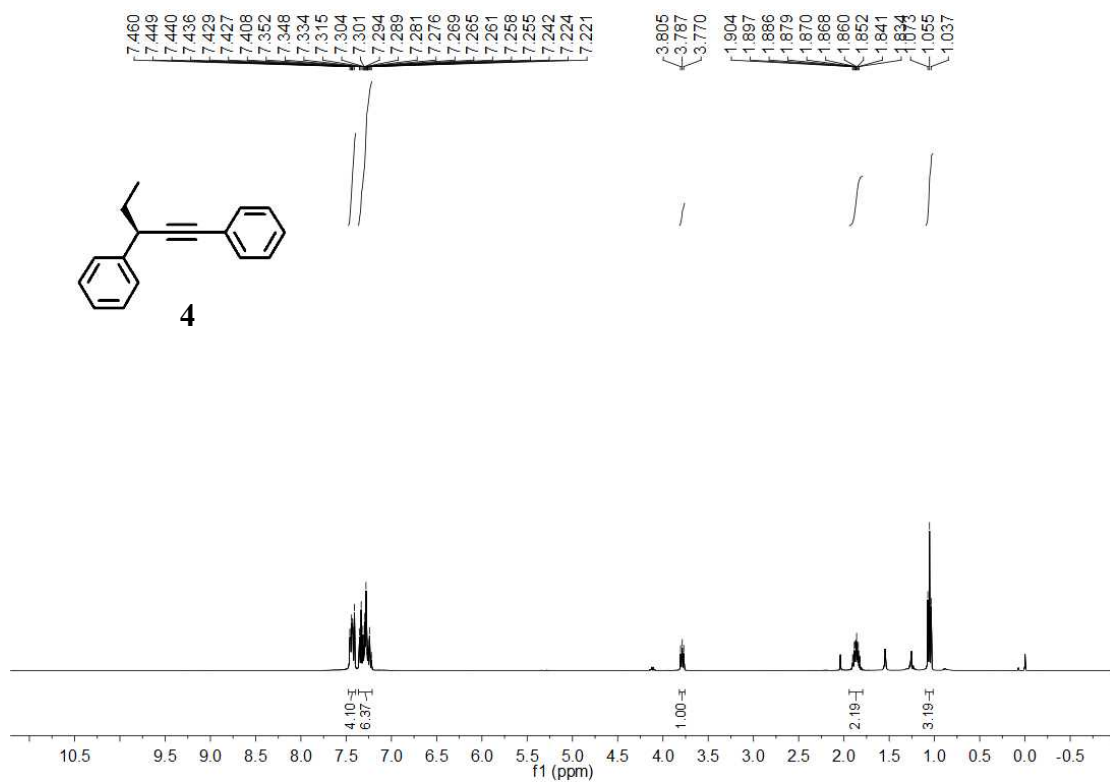
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- [2] Y. Duan, J.-H. Lin, J.-C. Xiao, Y.-C. Gu, *Org. Lett.* **2016**, *18*, 2471.
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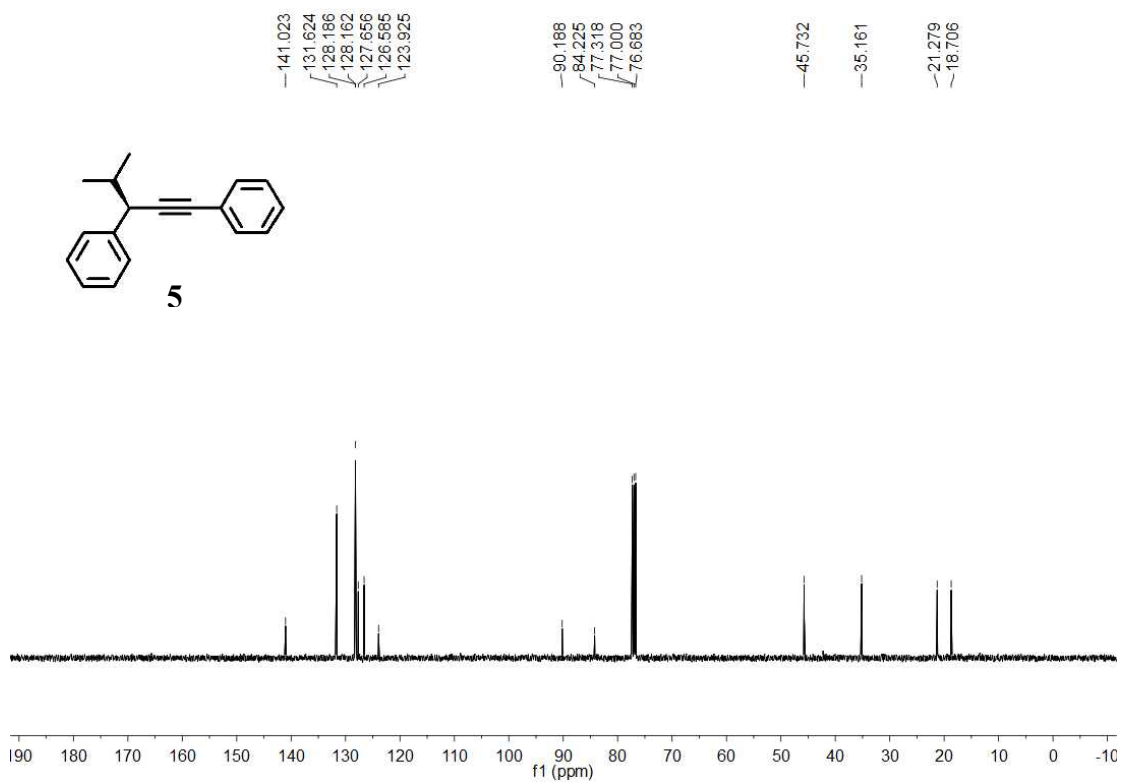
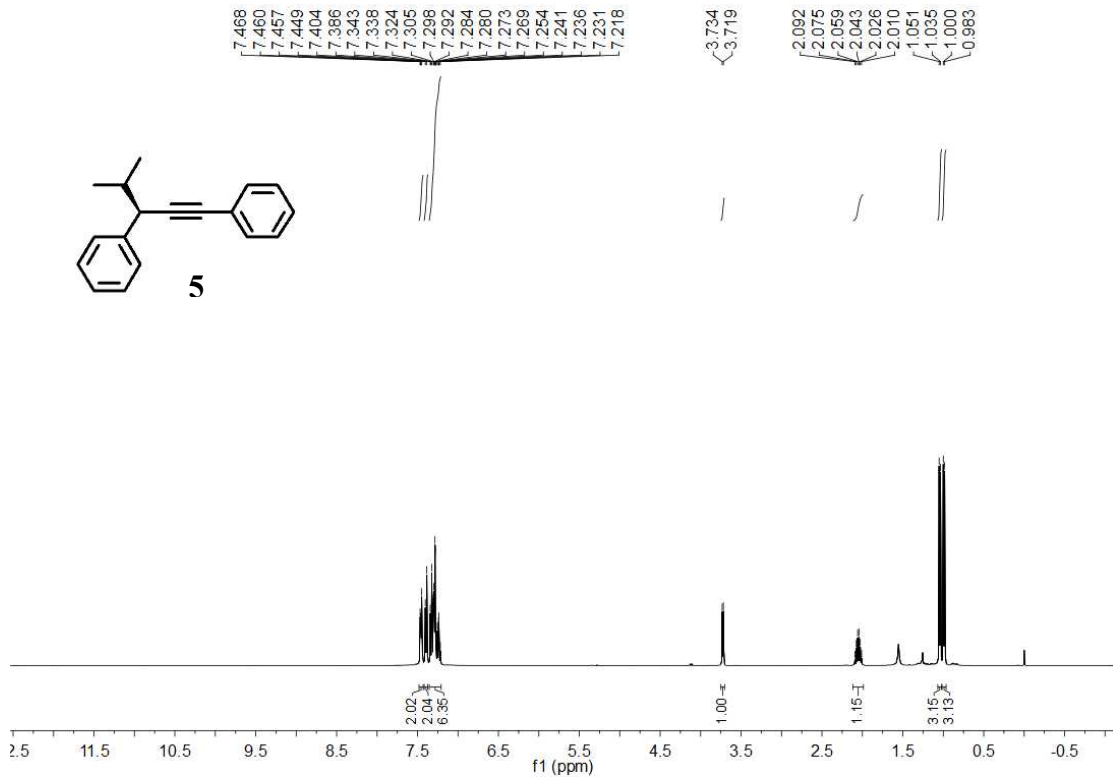
12. NMR Spectra

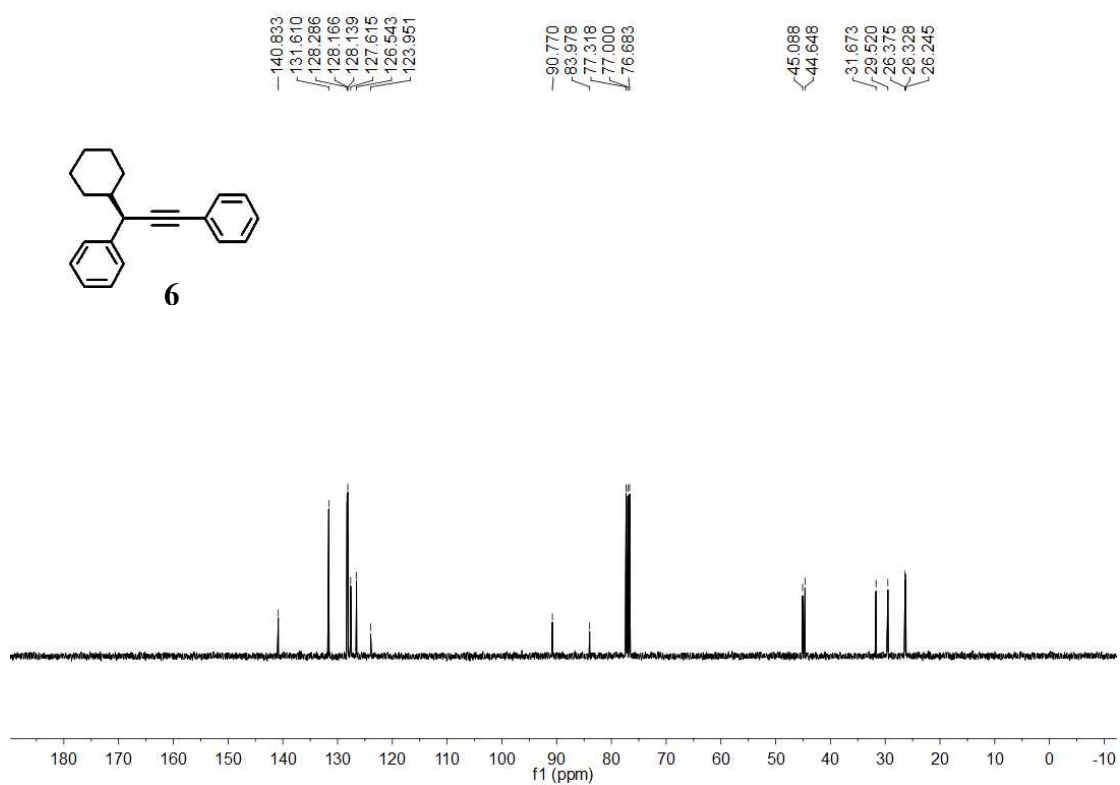
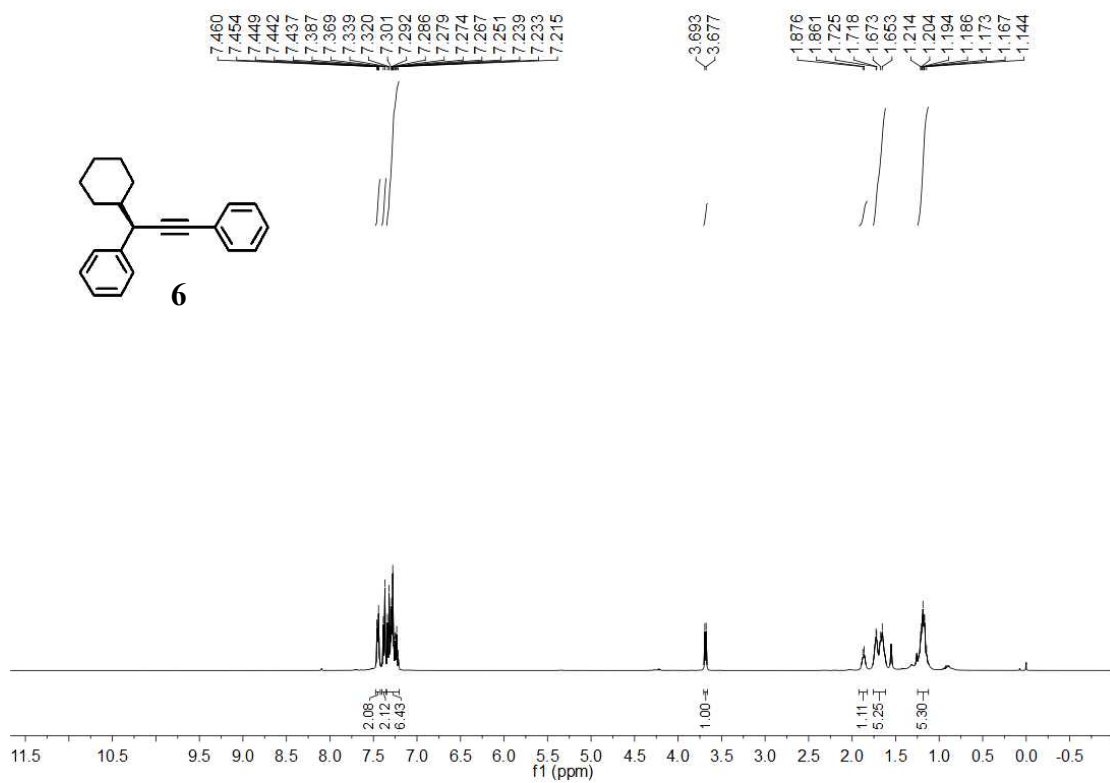


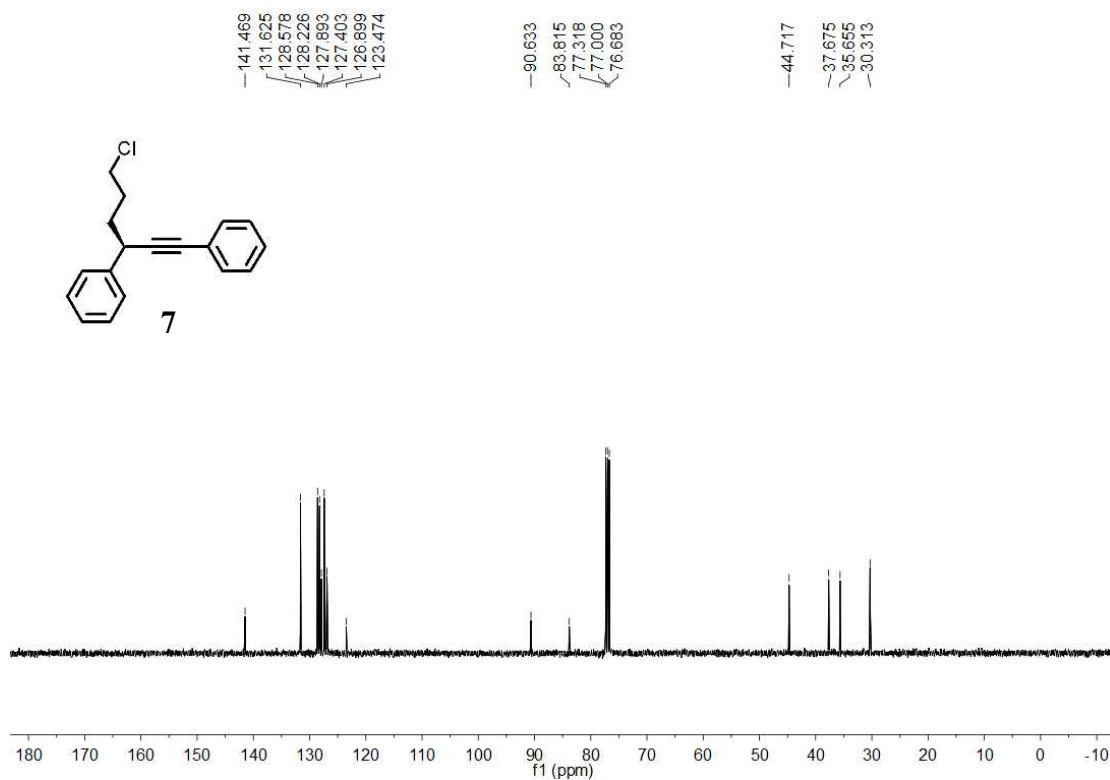
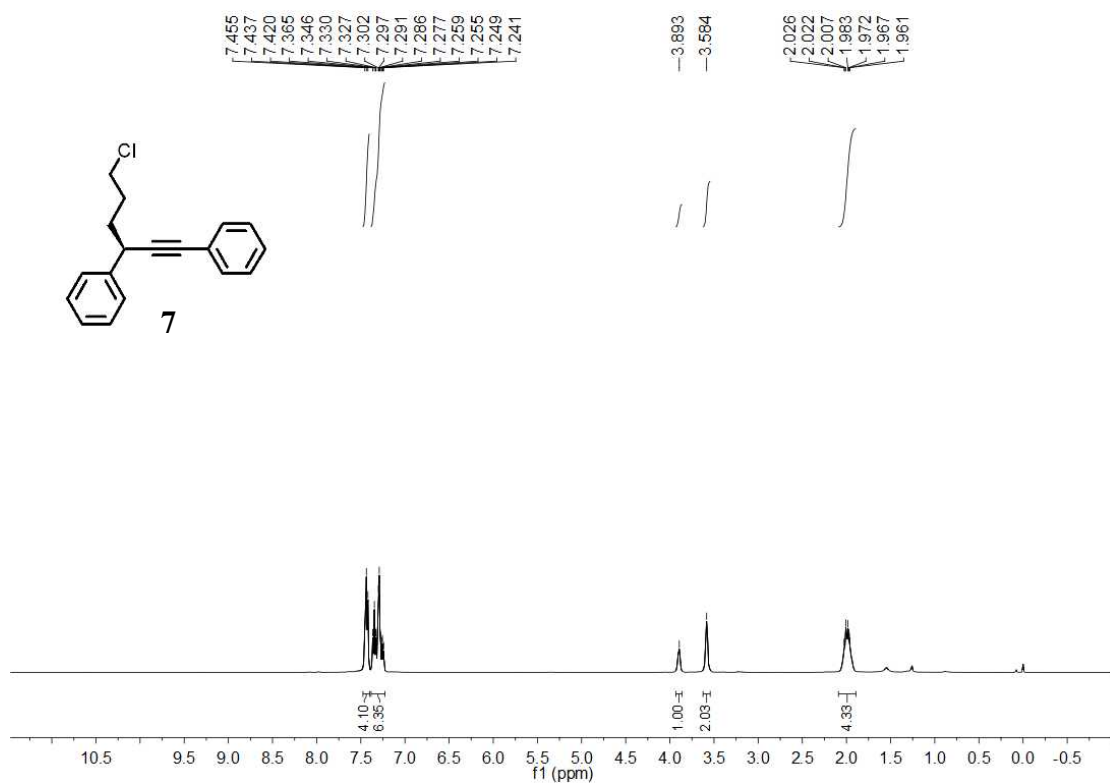


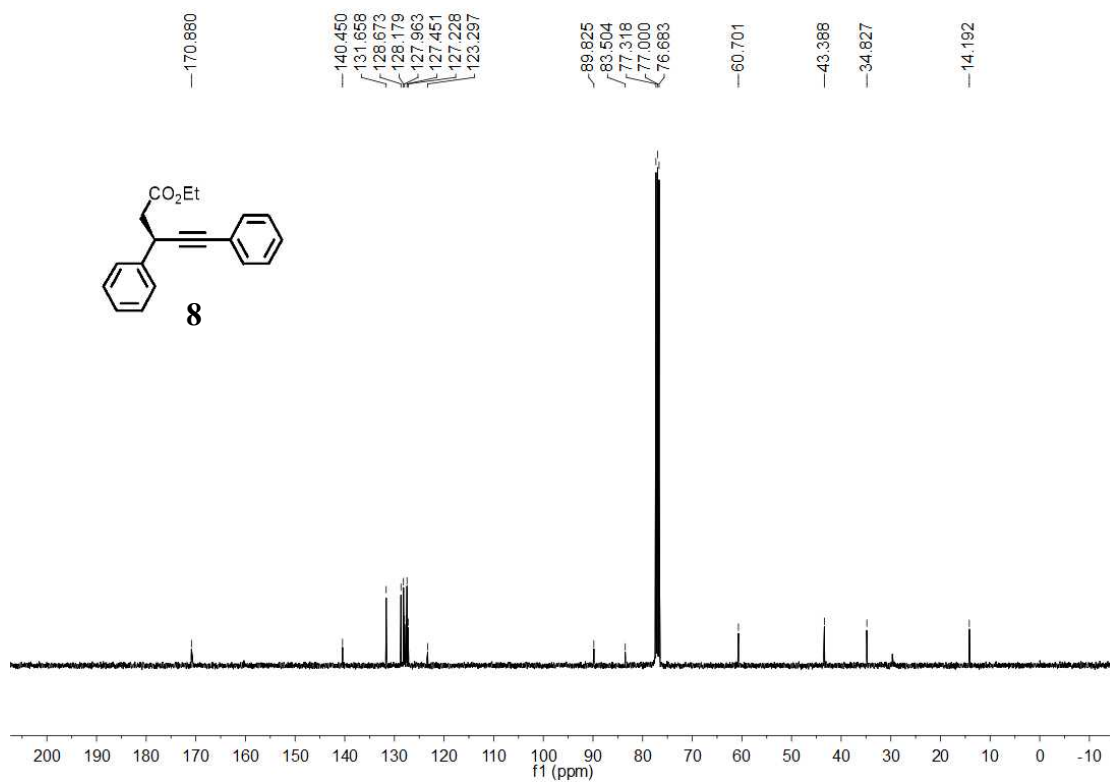
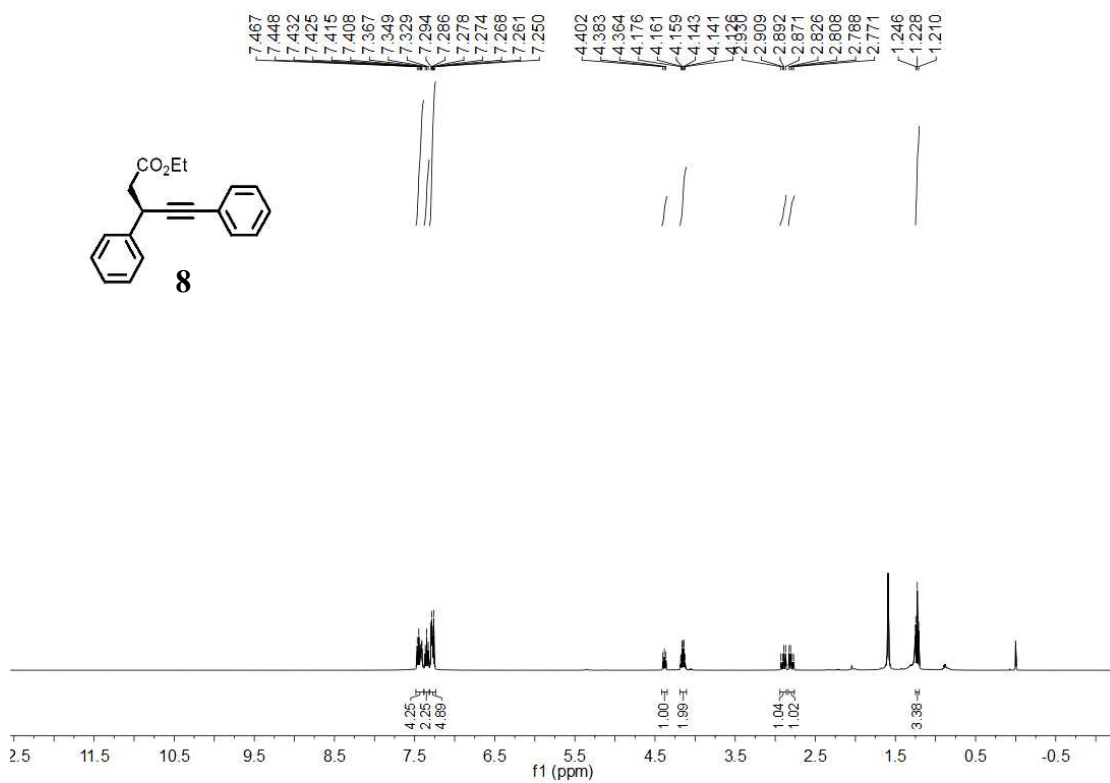


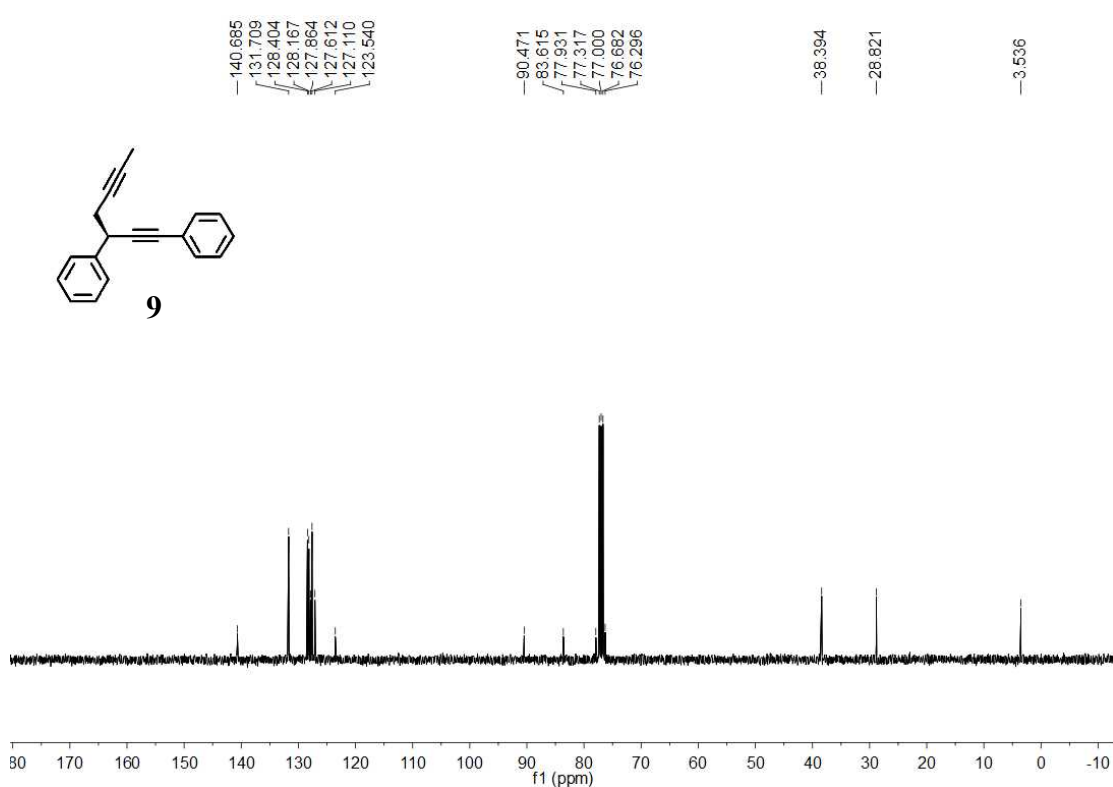
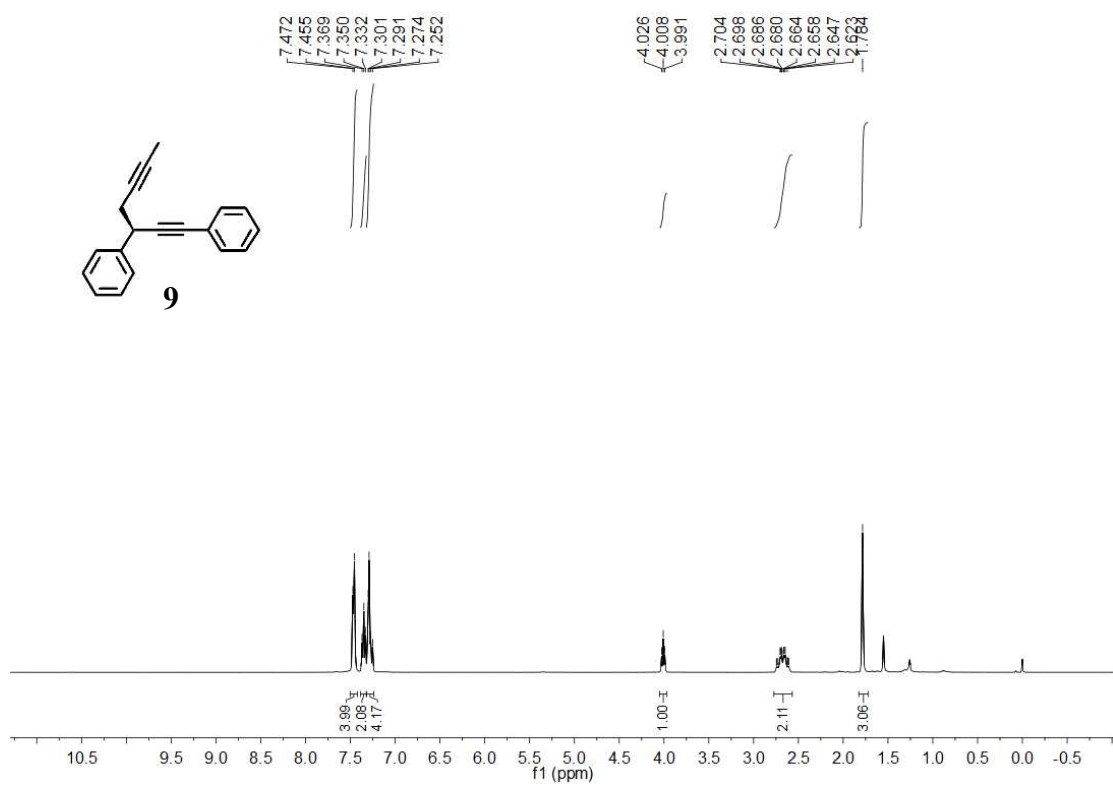


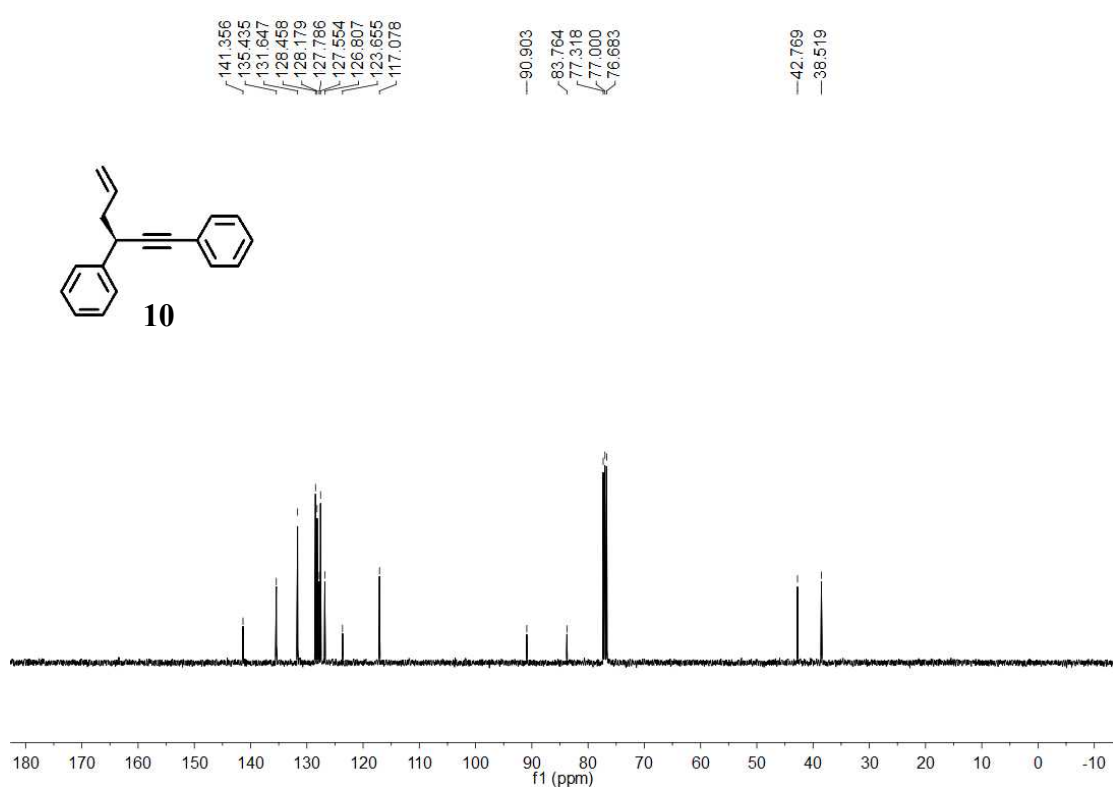
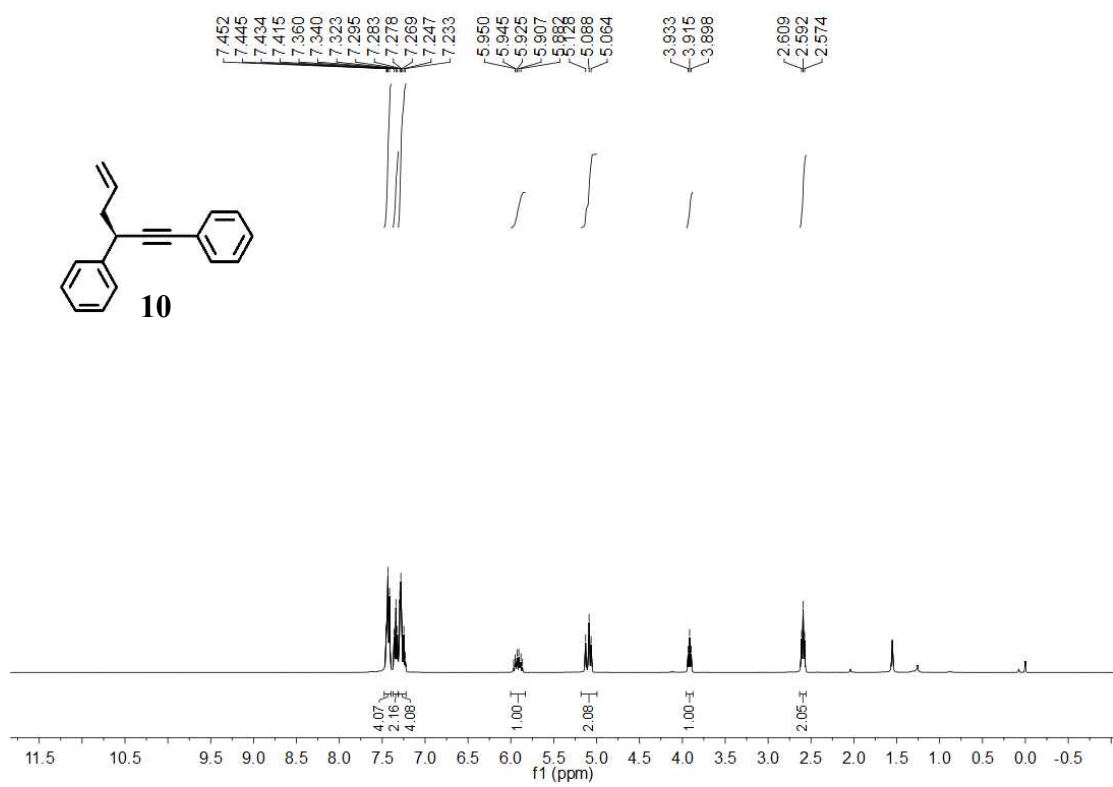


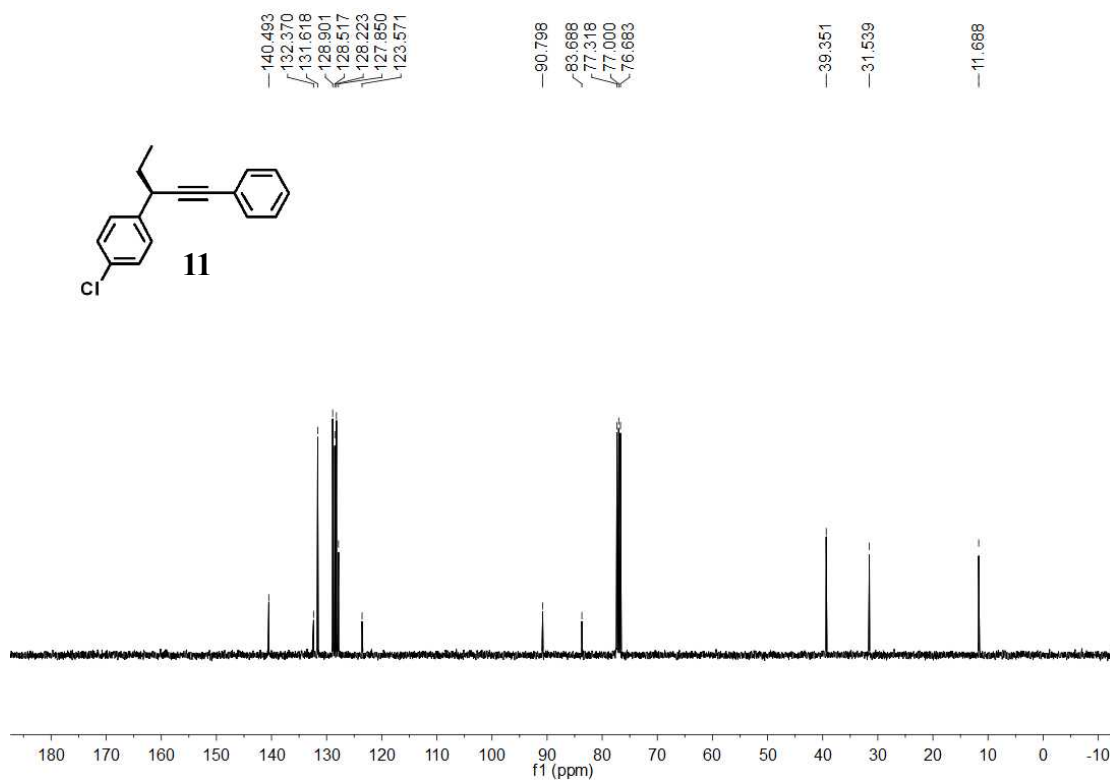
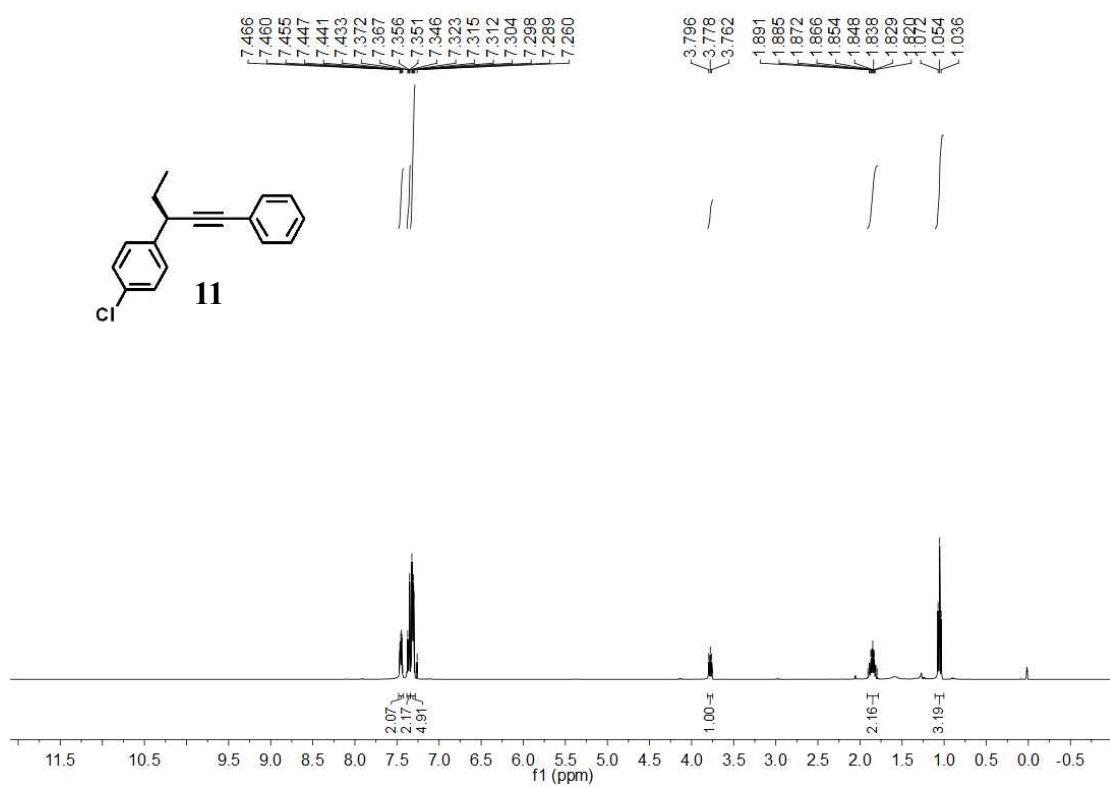


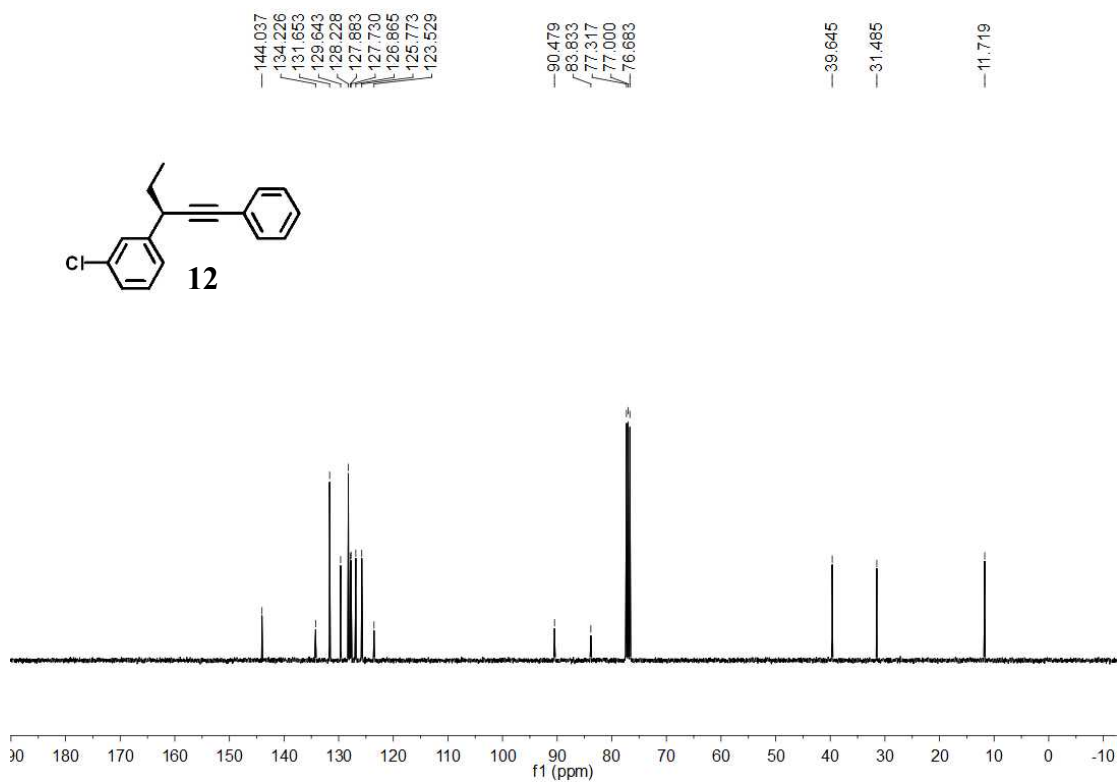
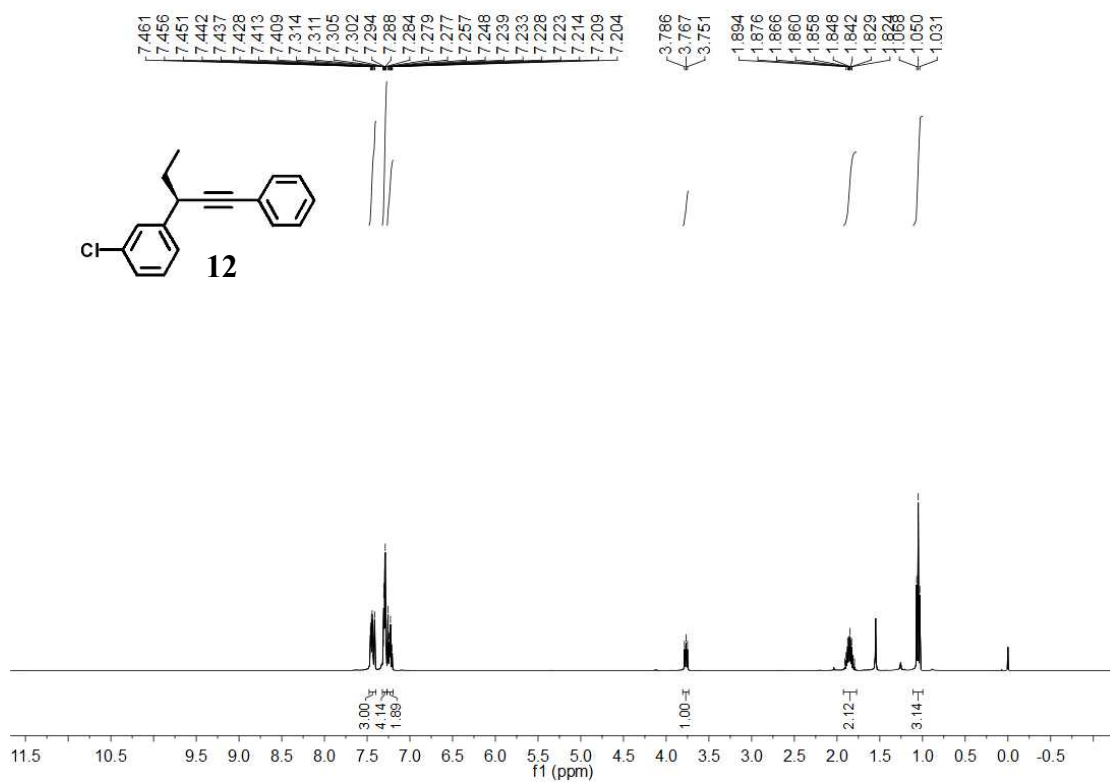


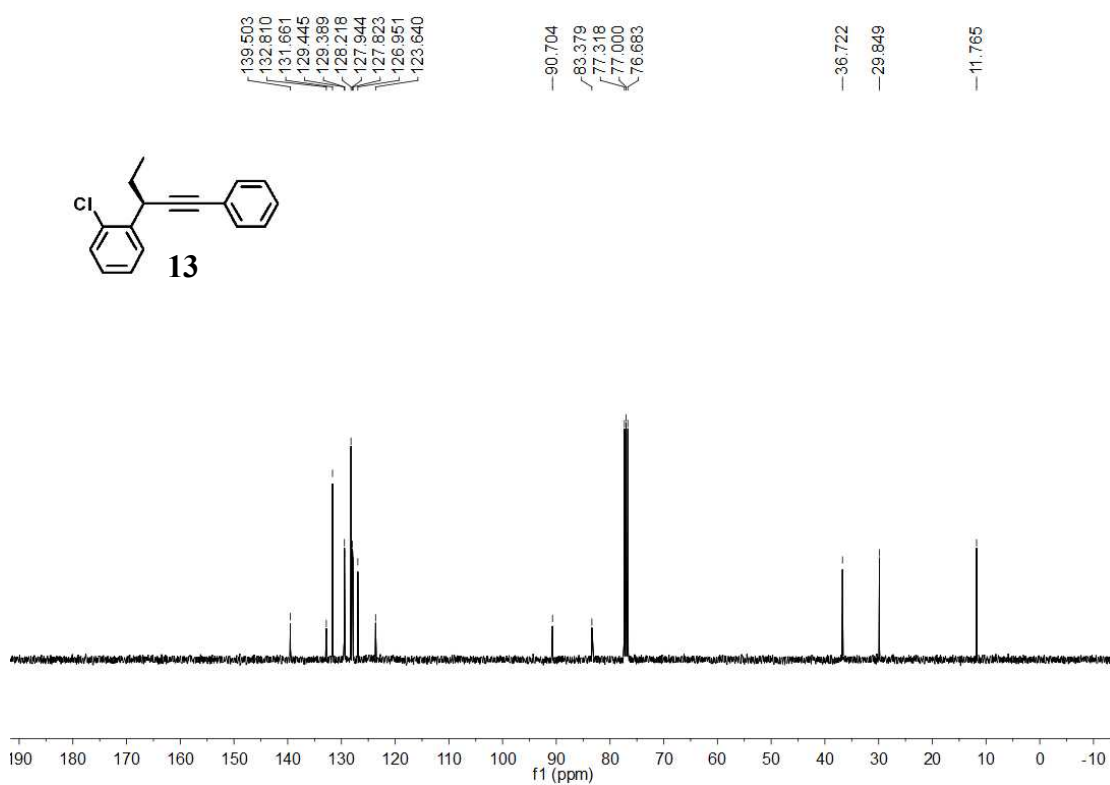
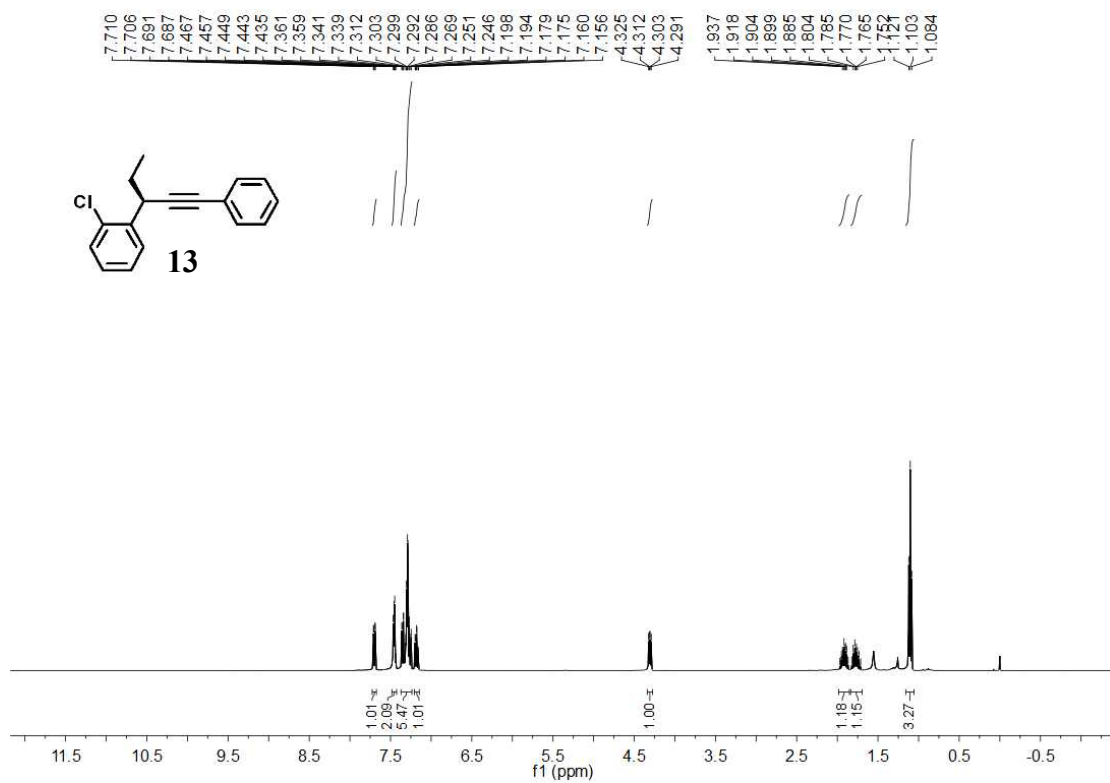


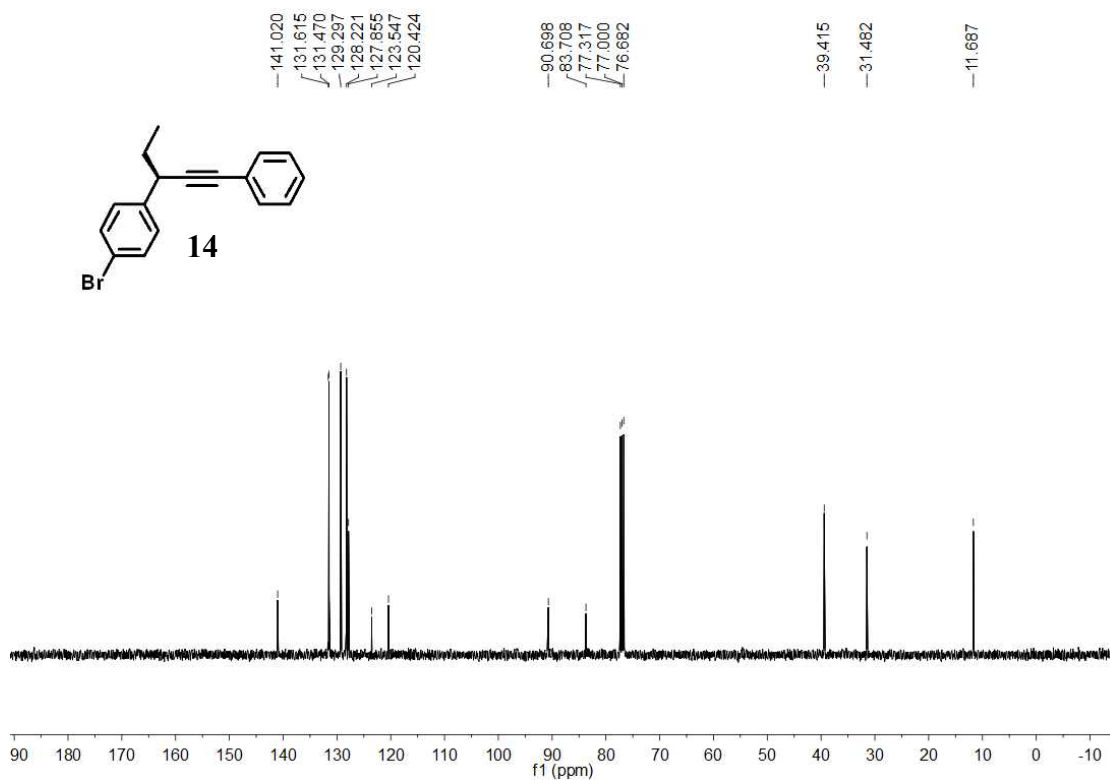
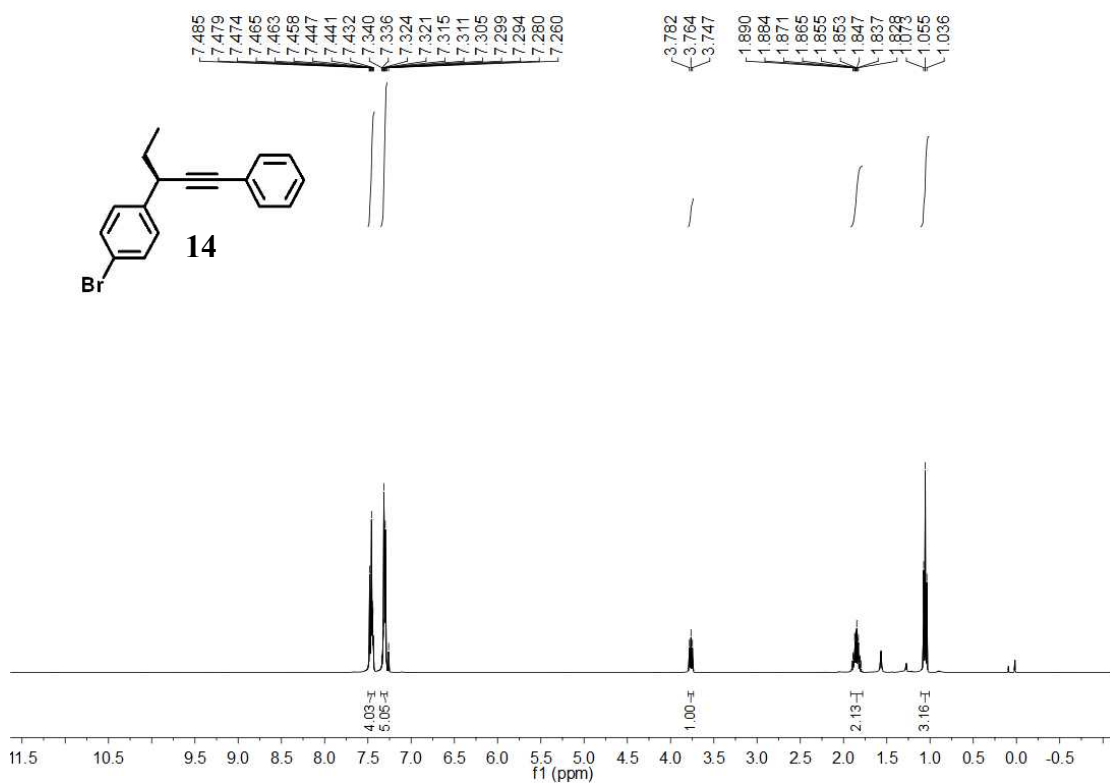


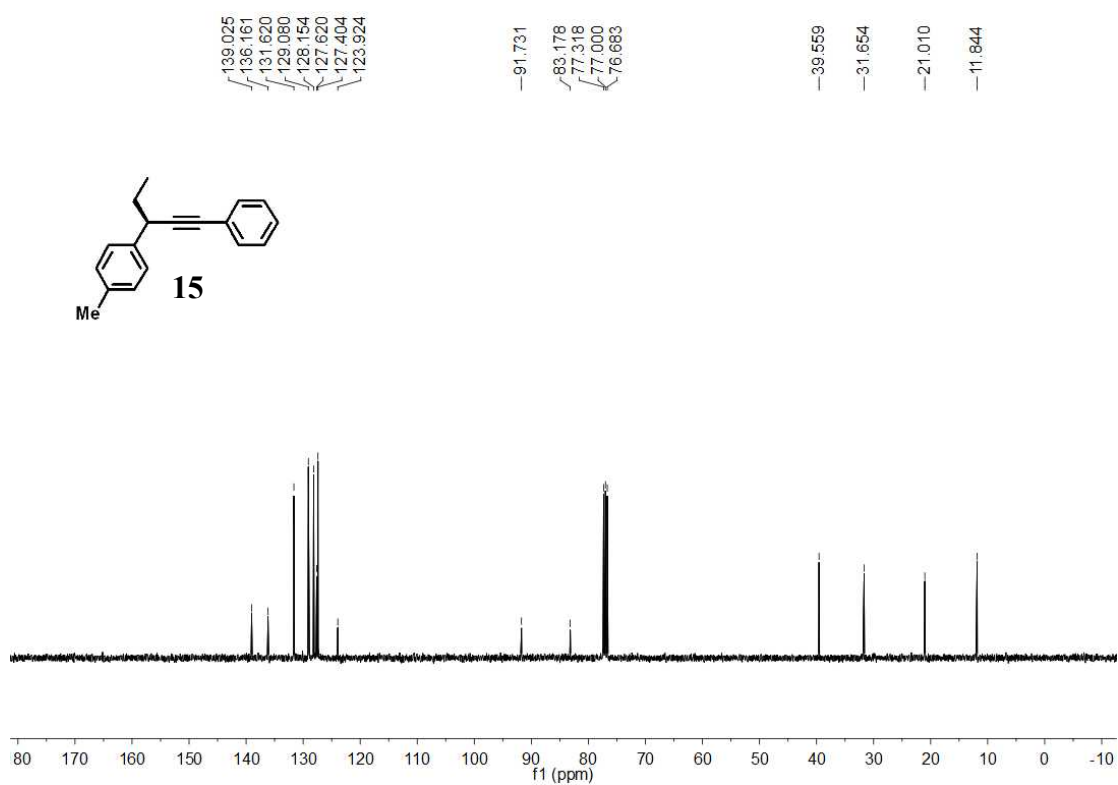
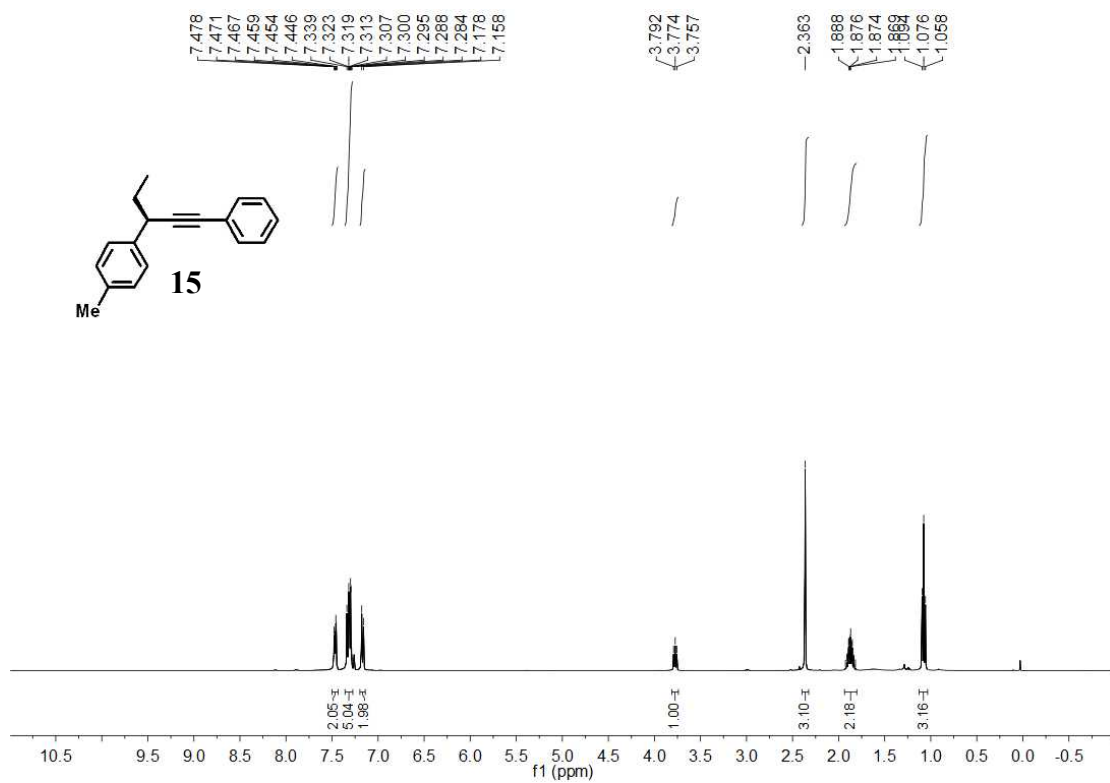


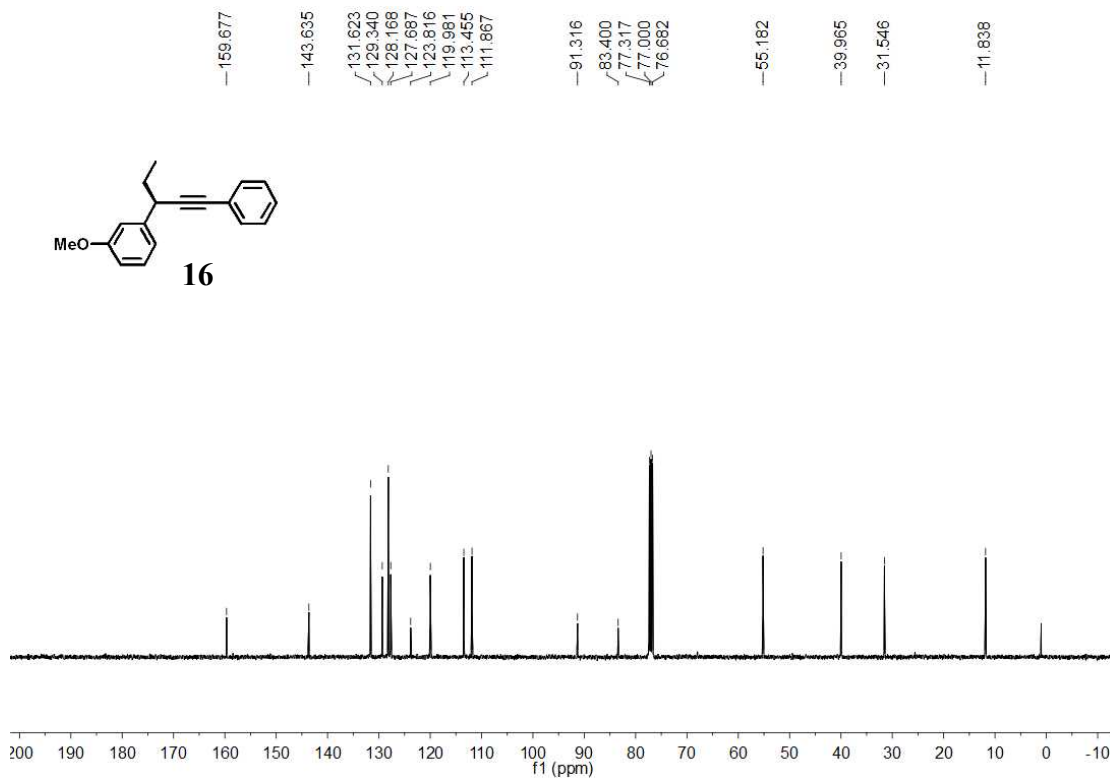
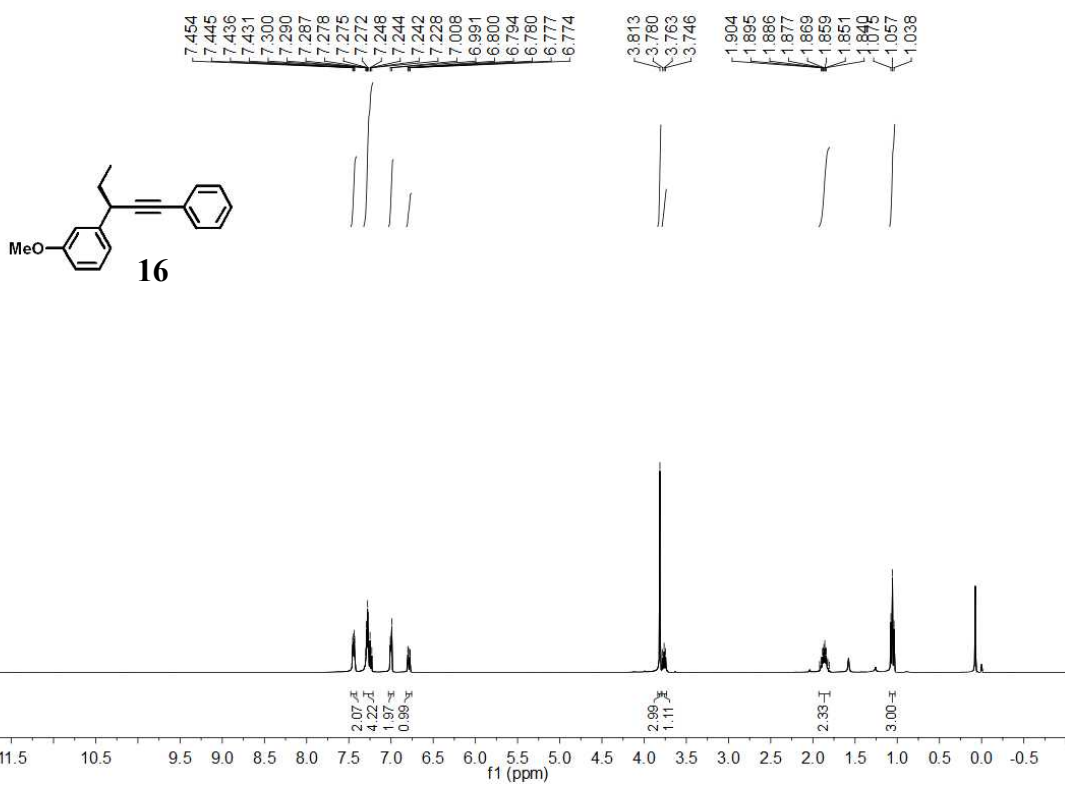


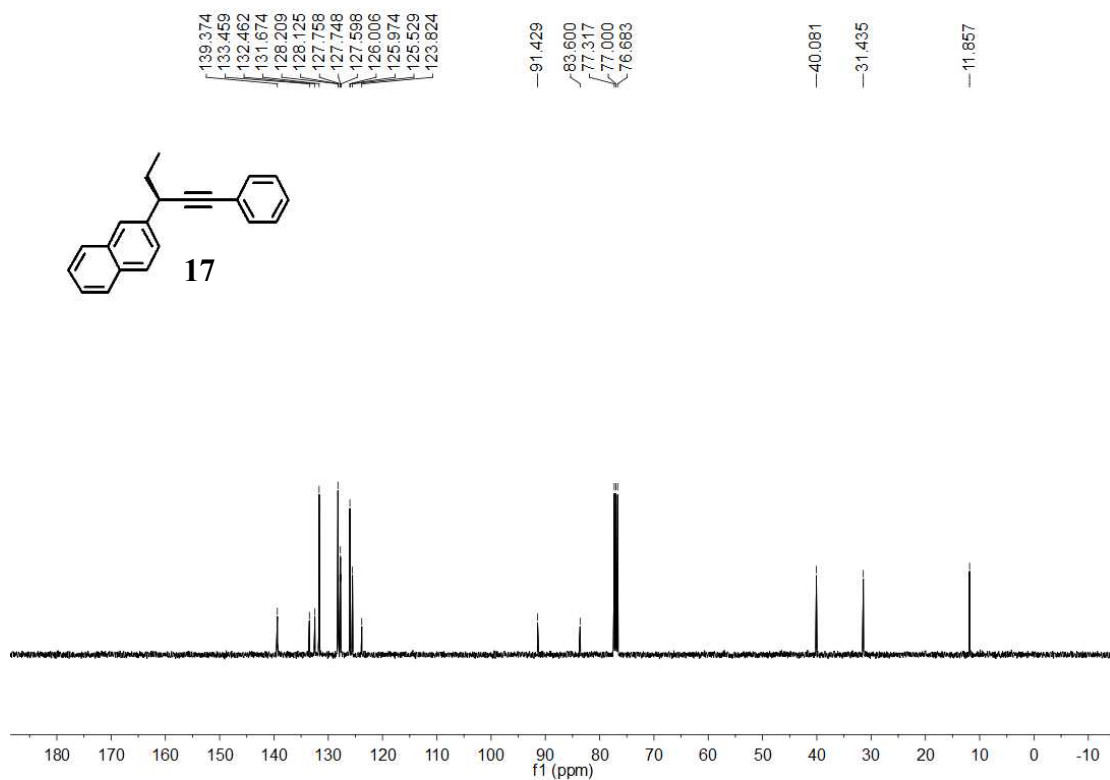
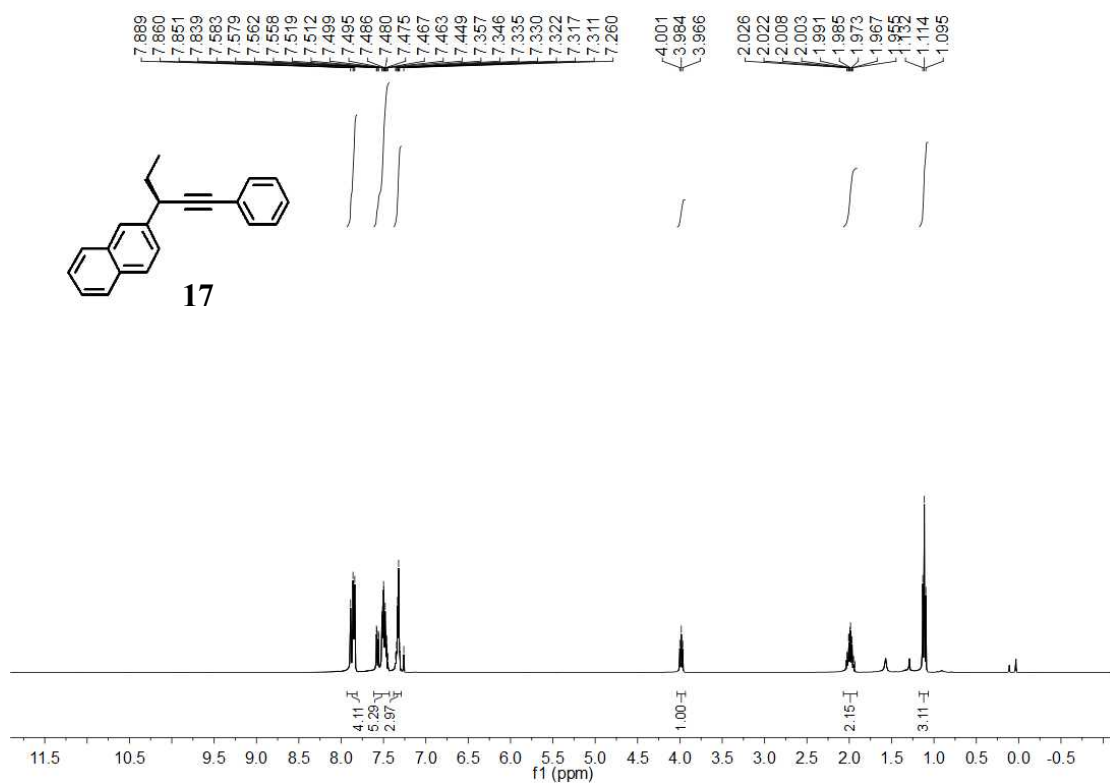


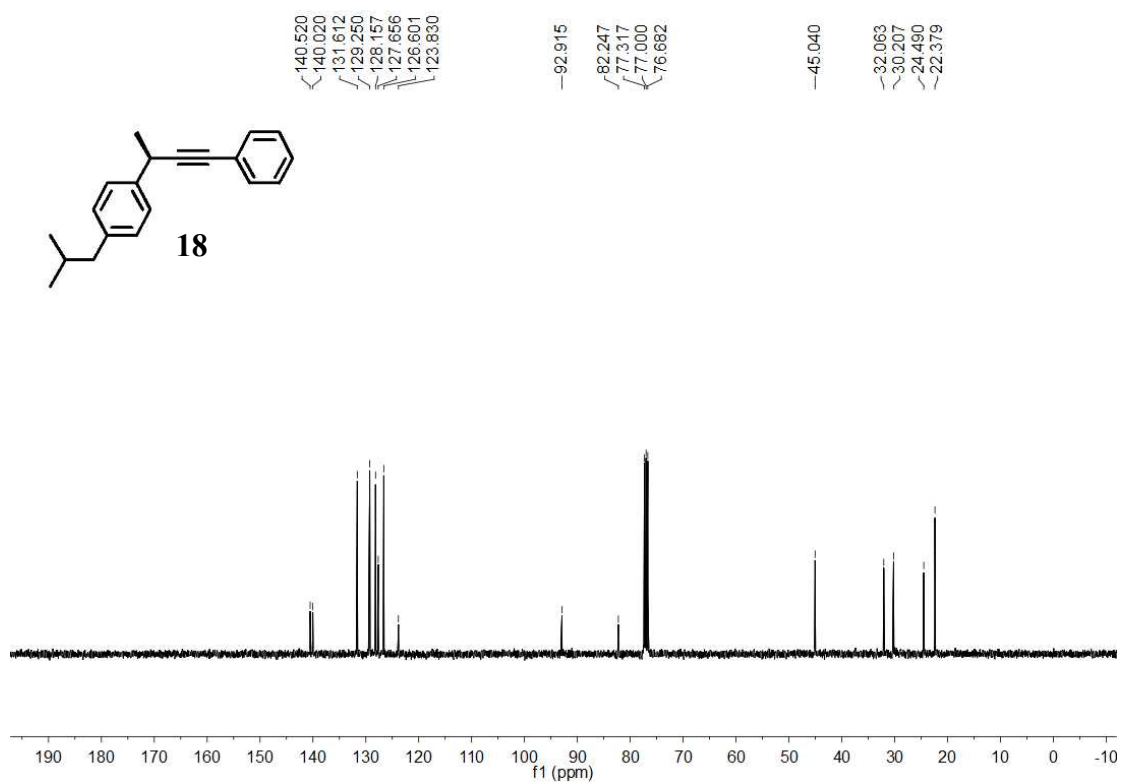
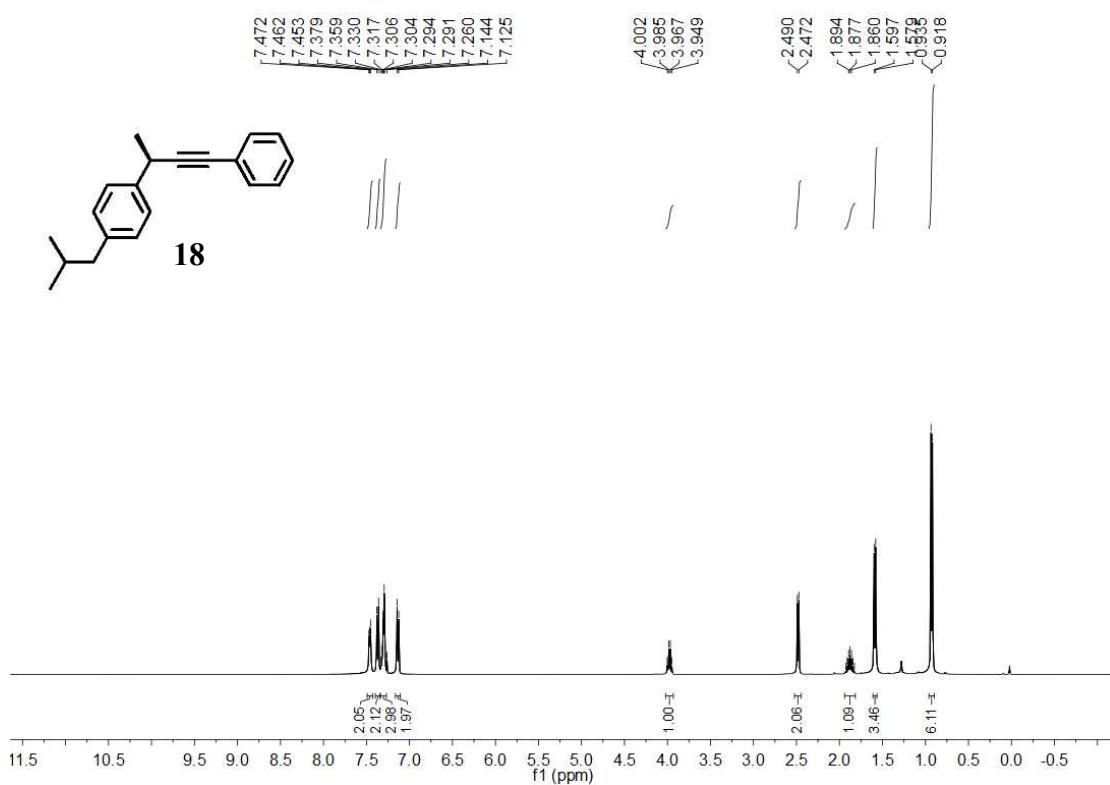


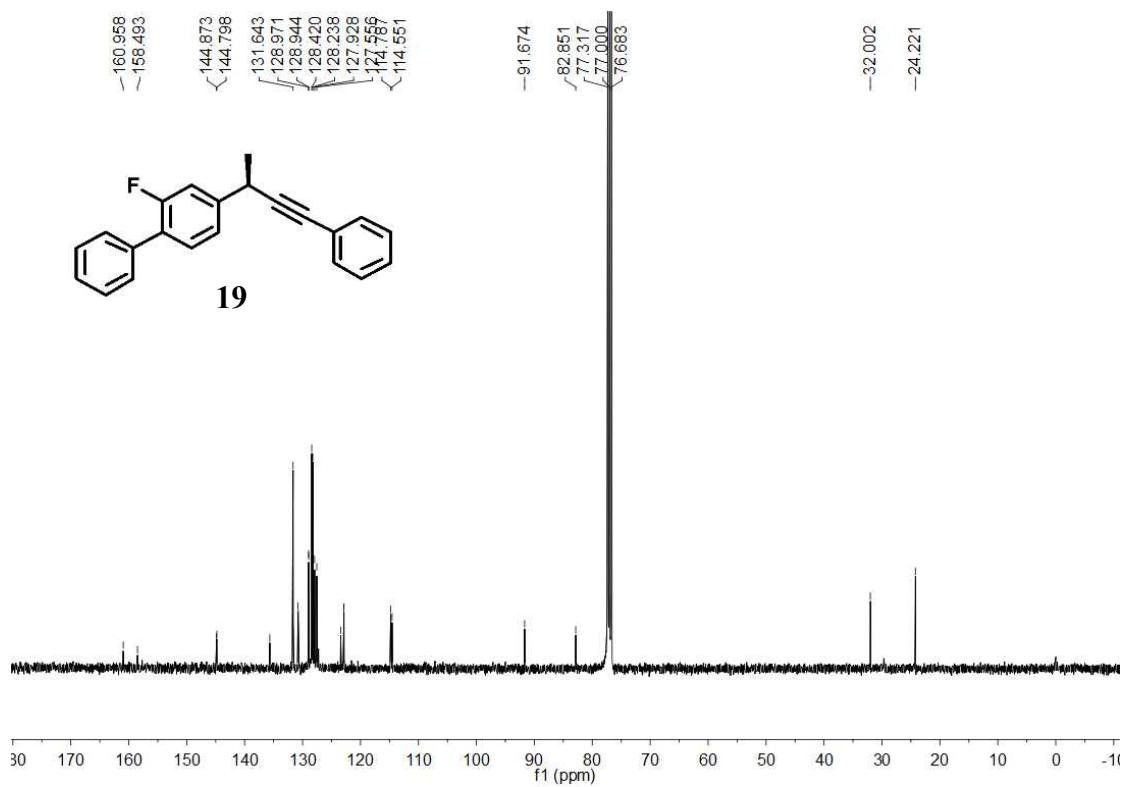
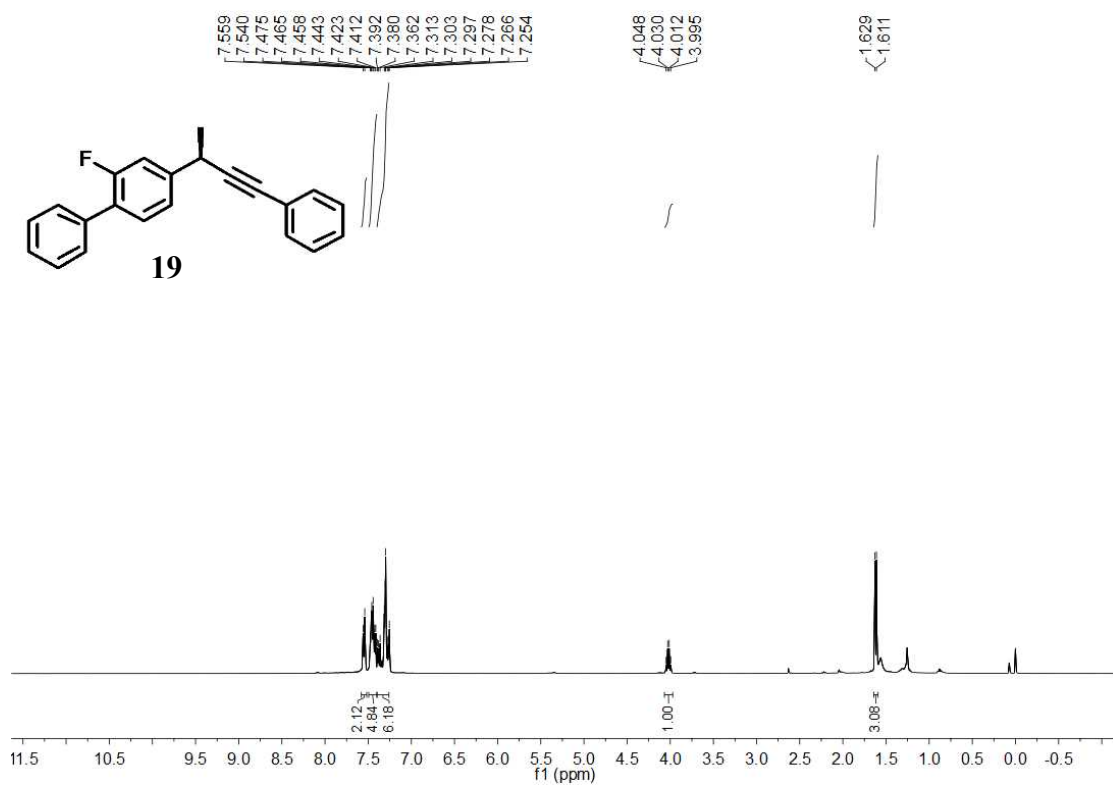


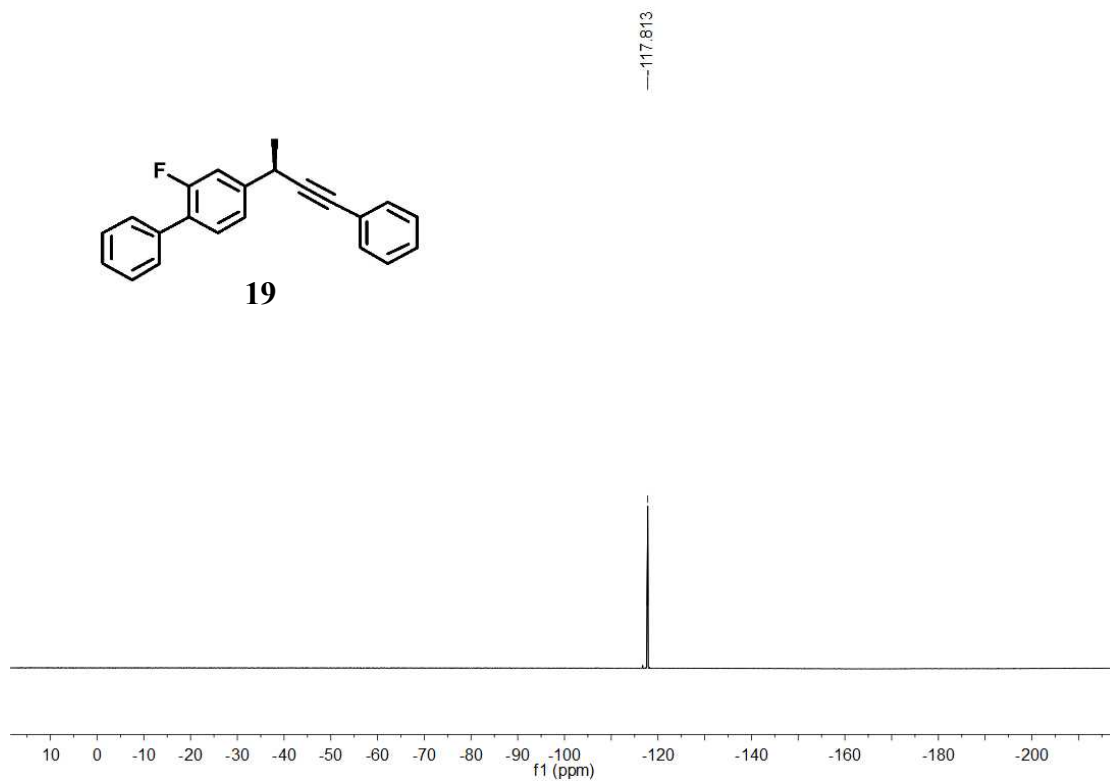
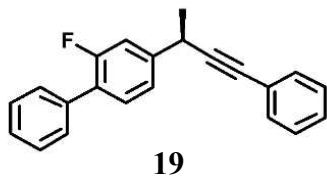


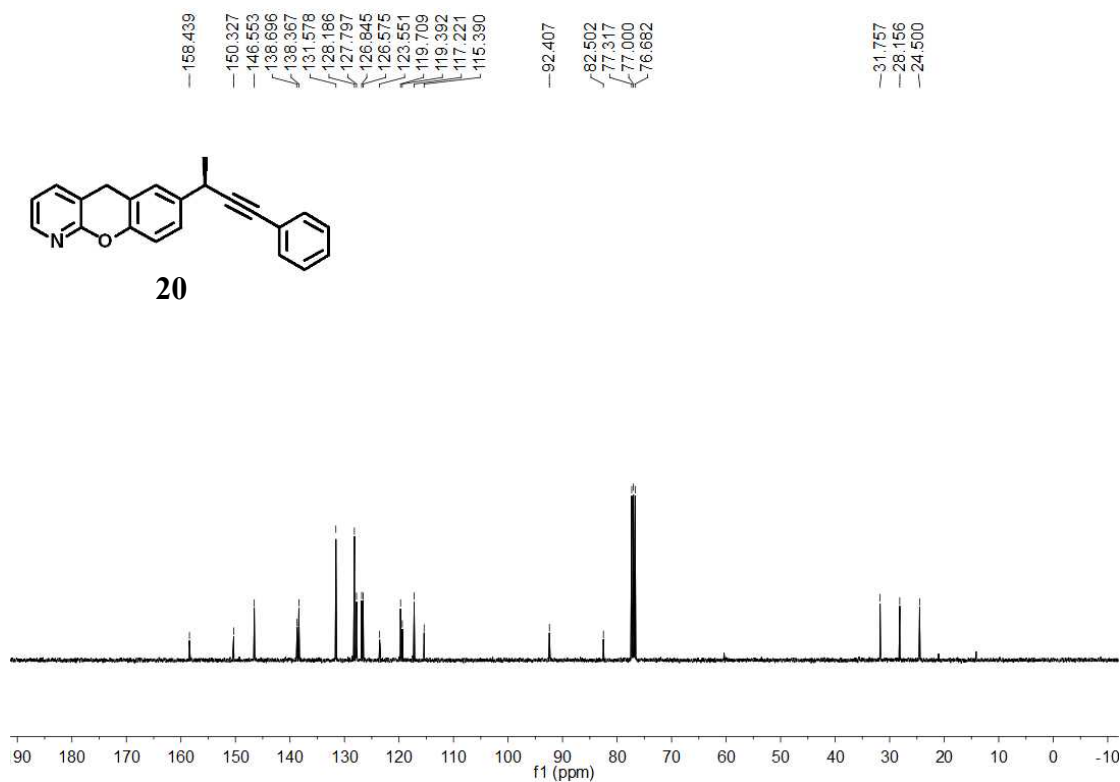
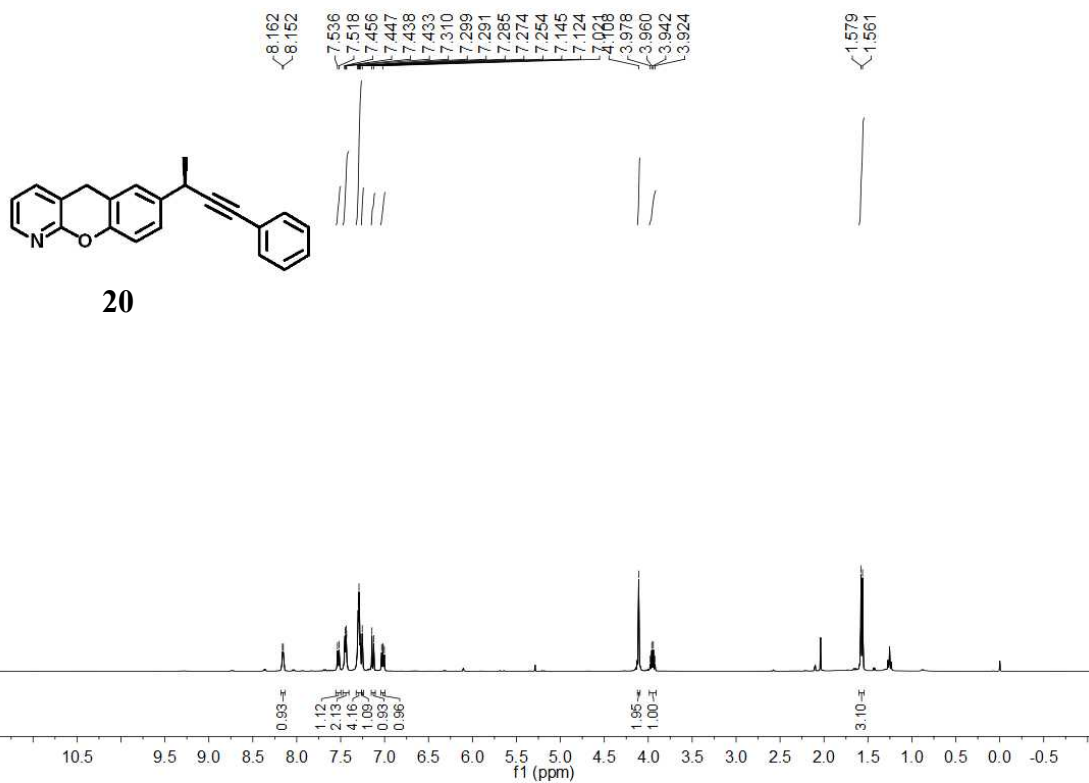


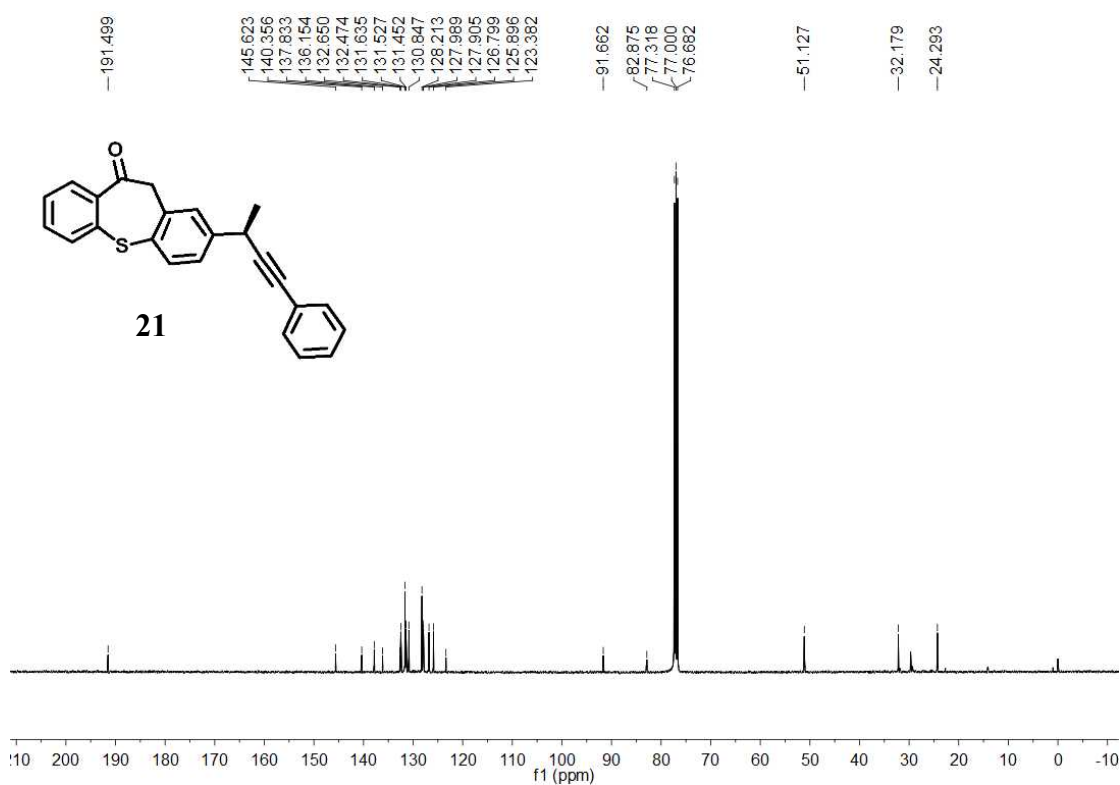
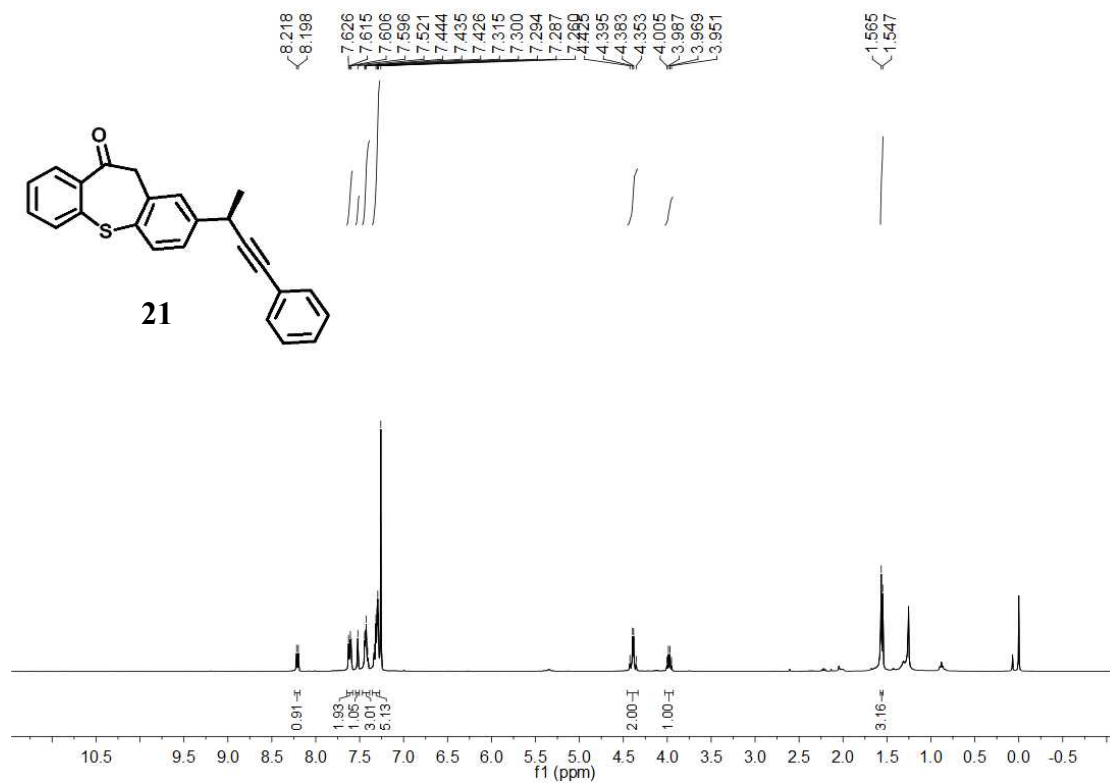


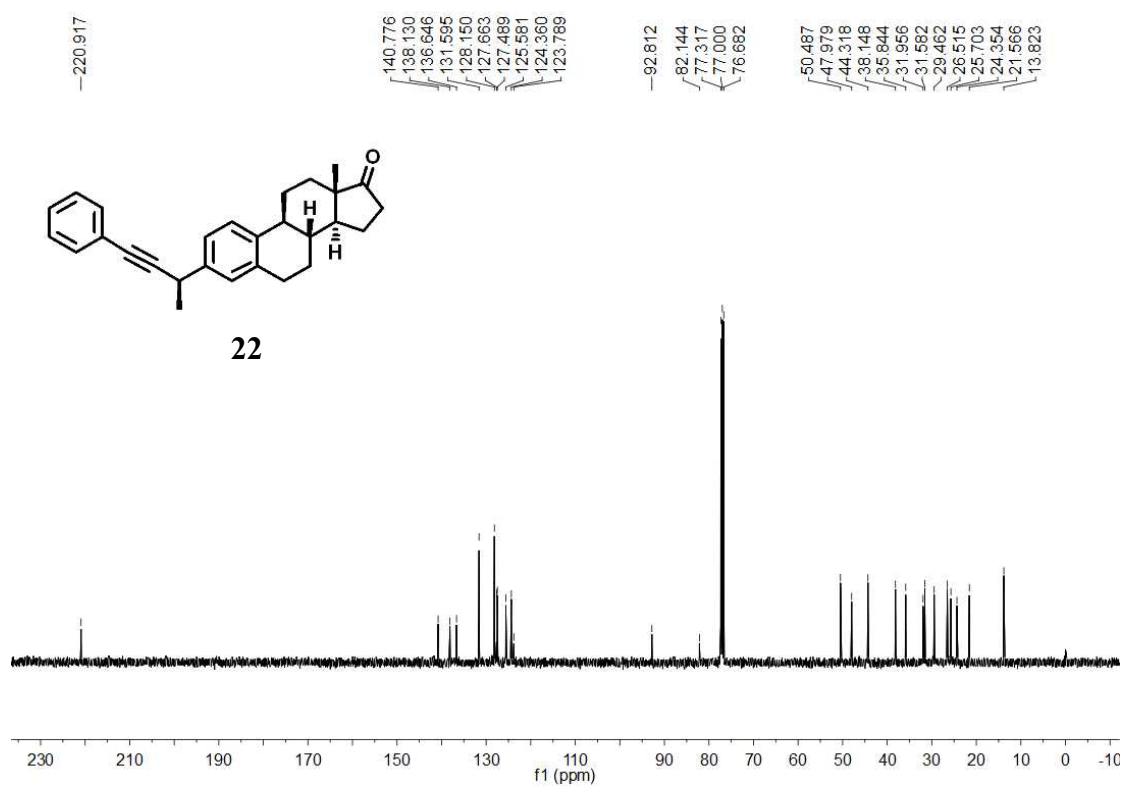
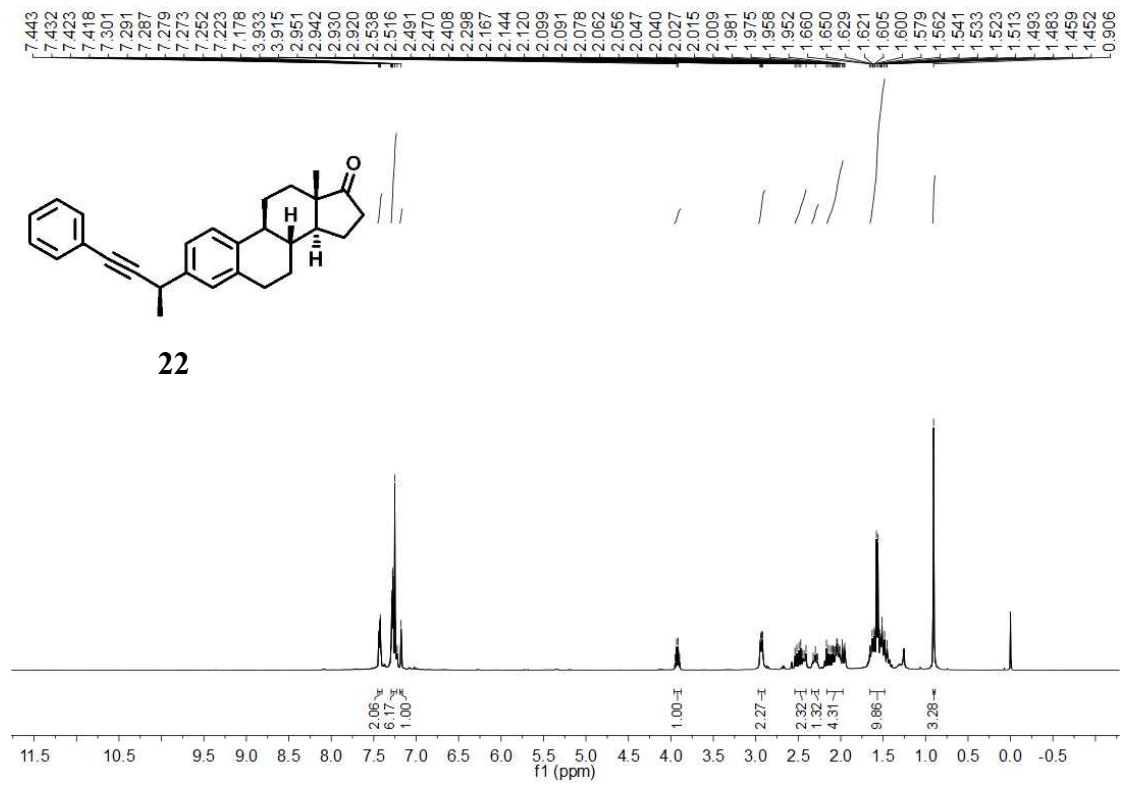


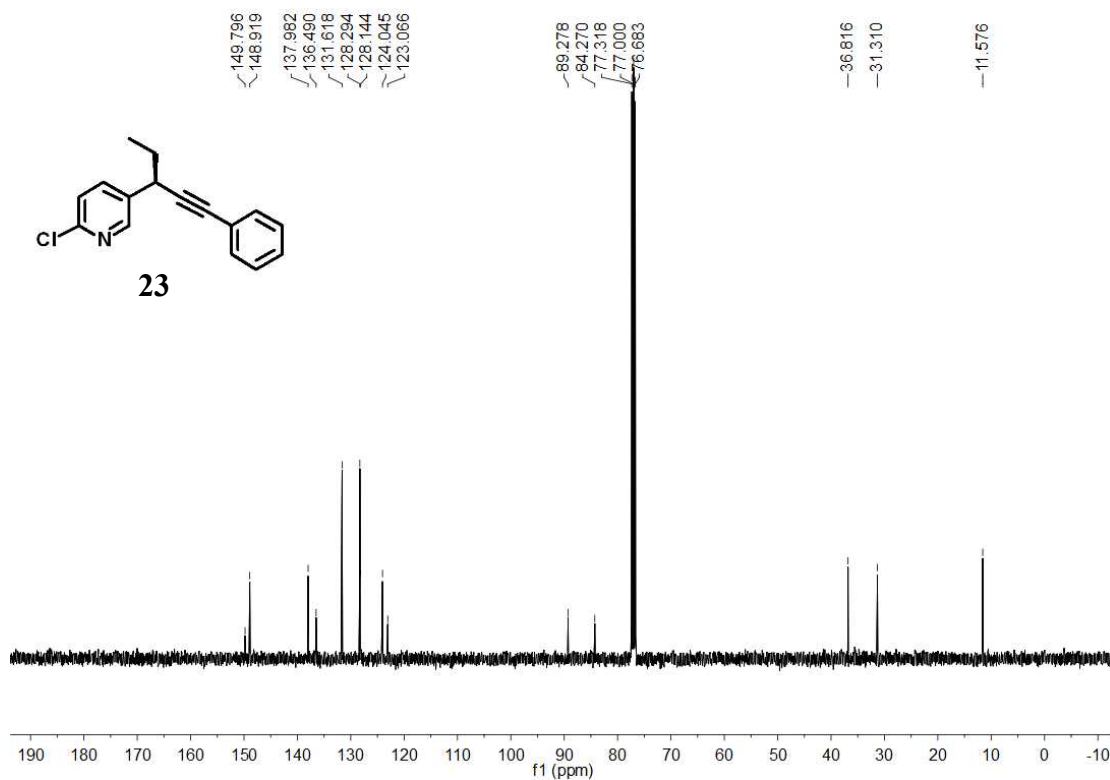
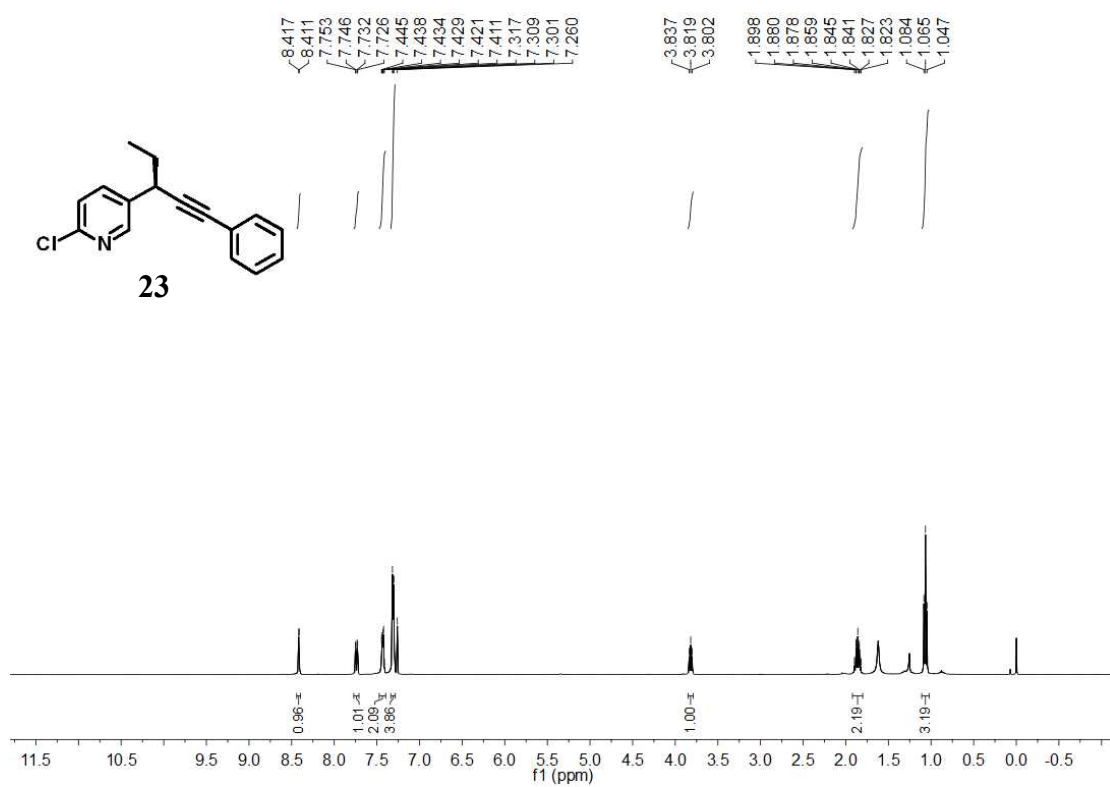


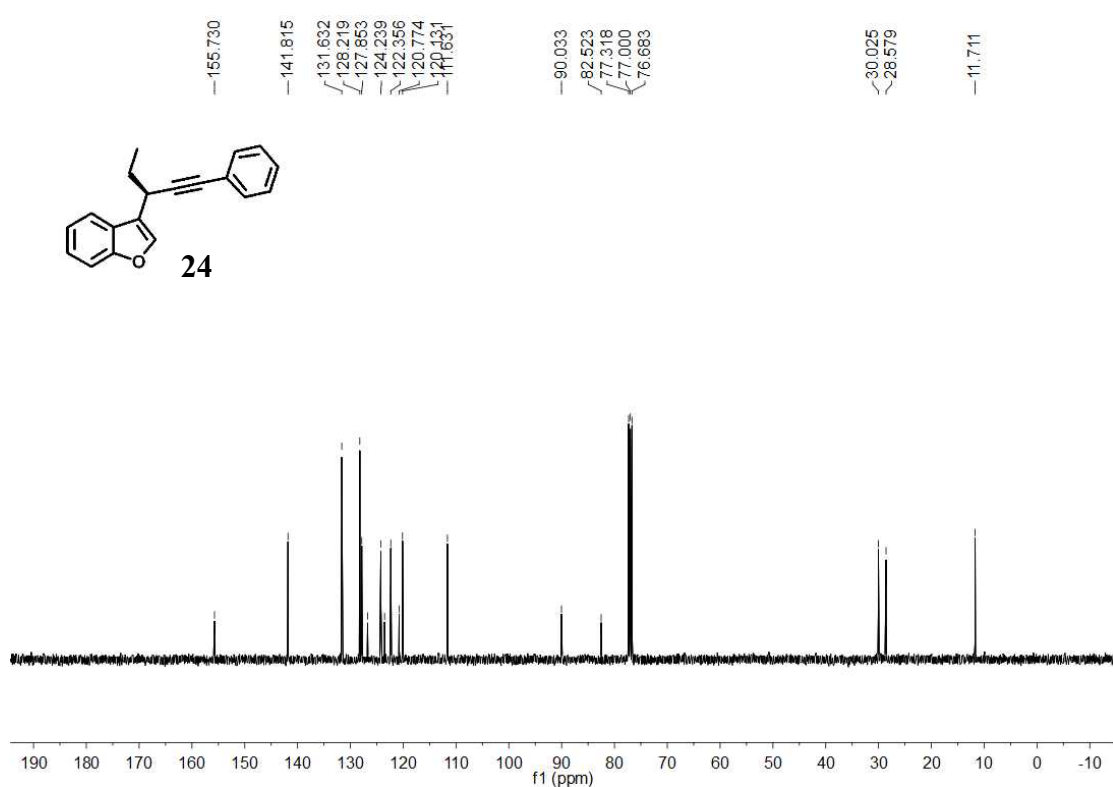
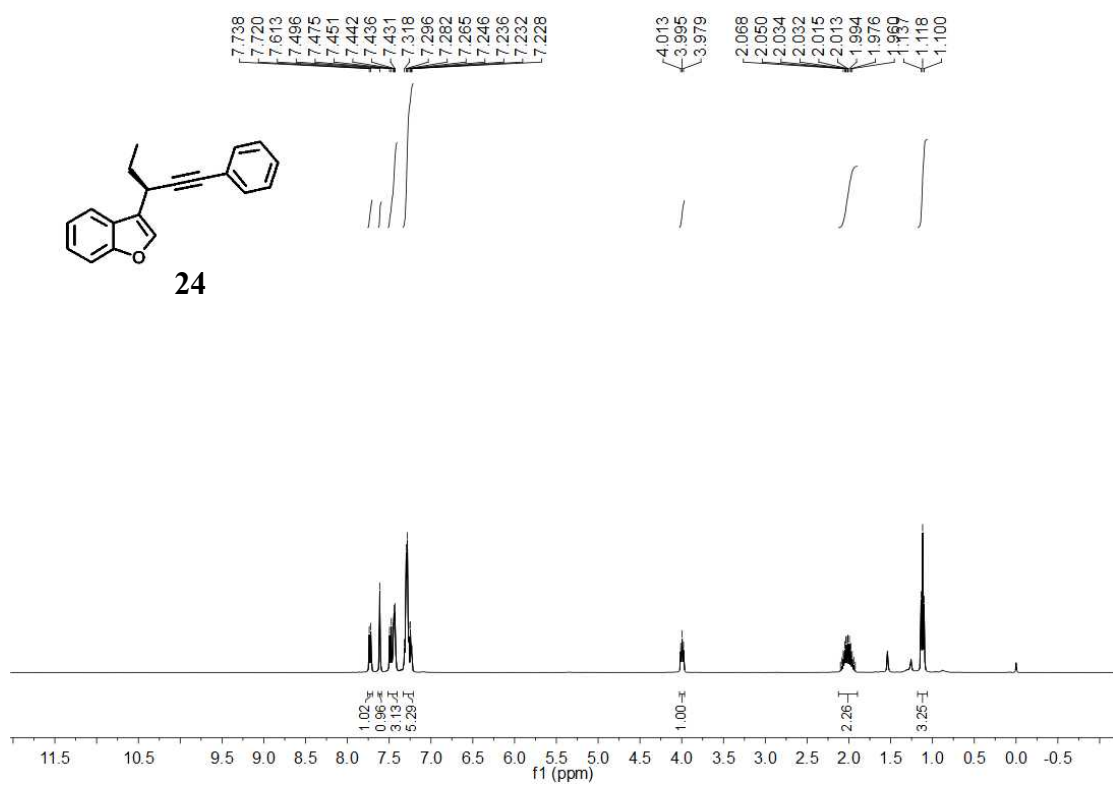


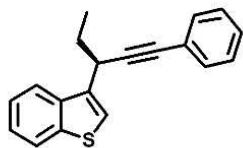










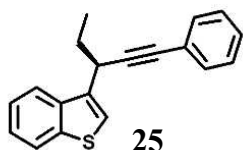
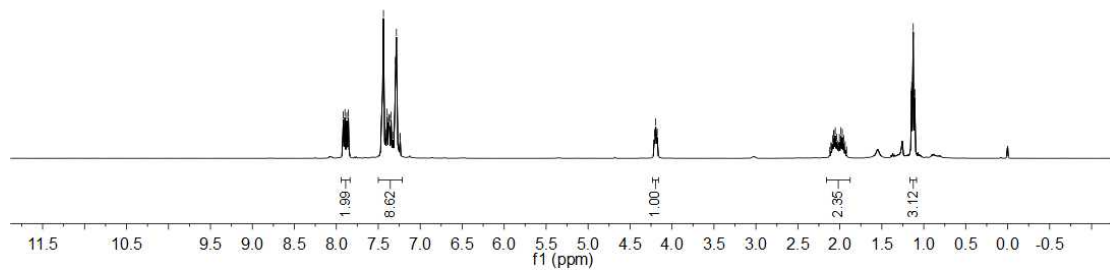


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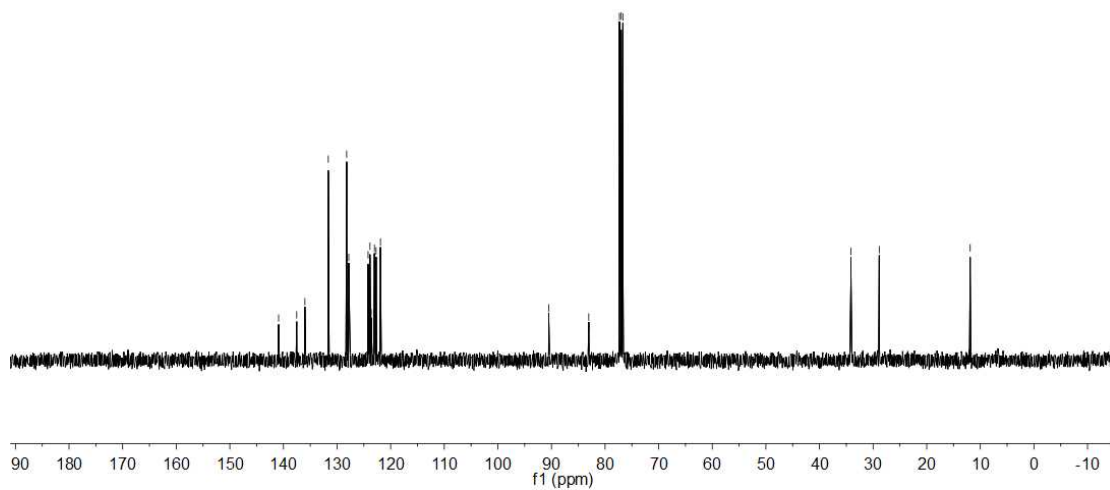
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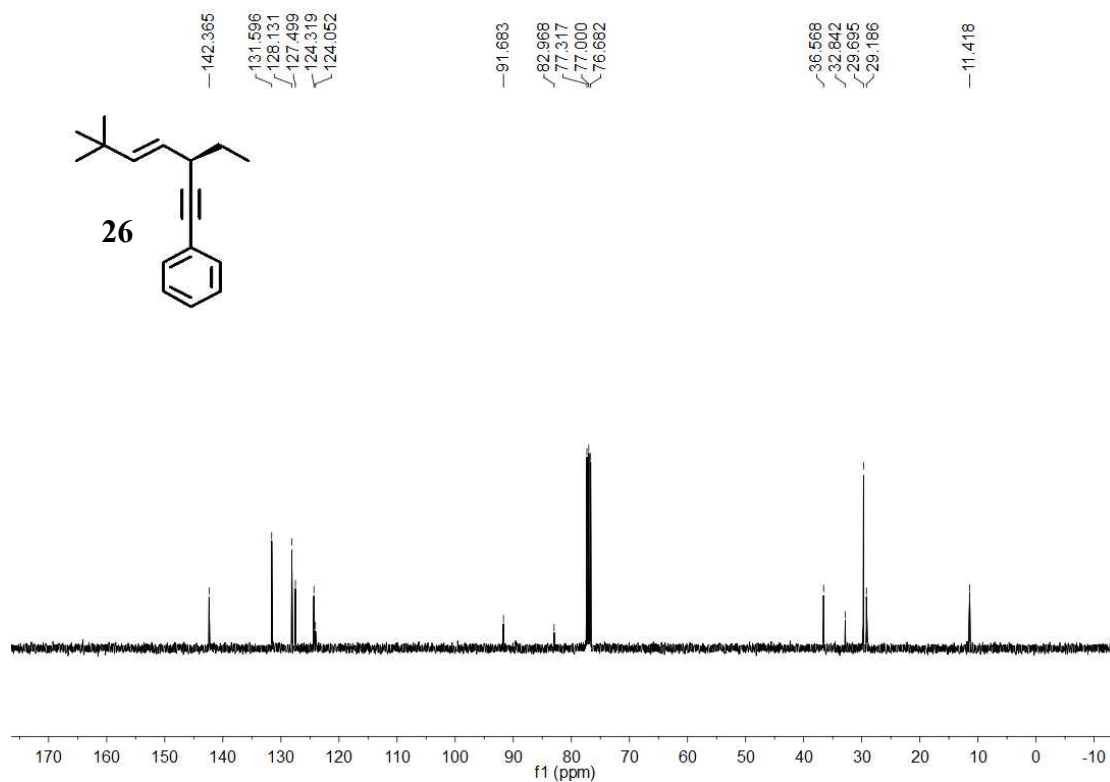
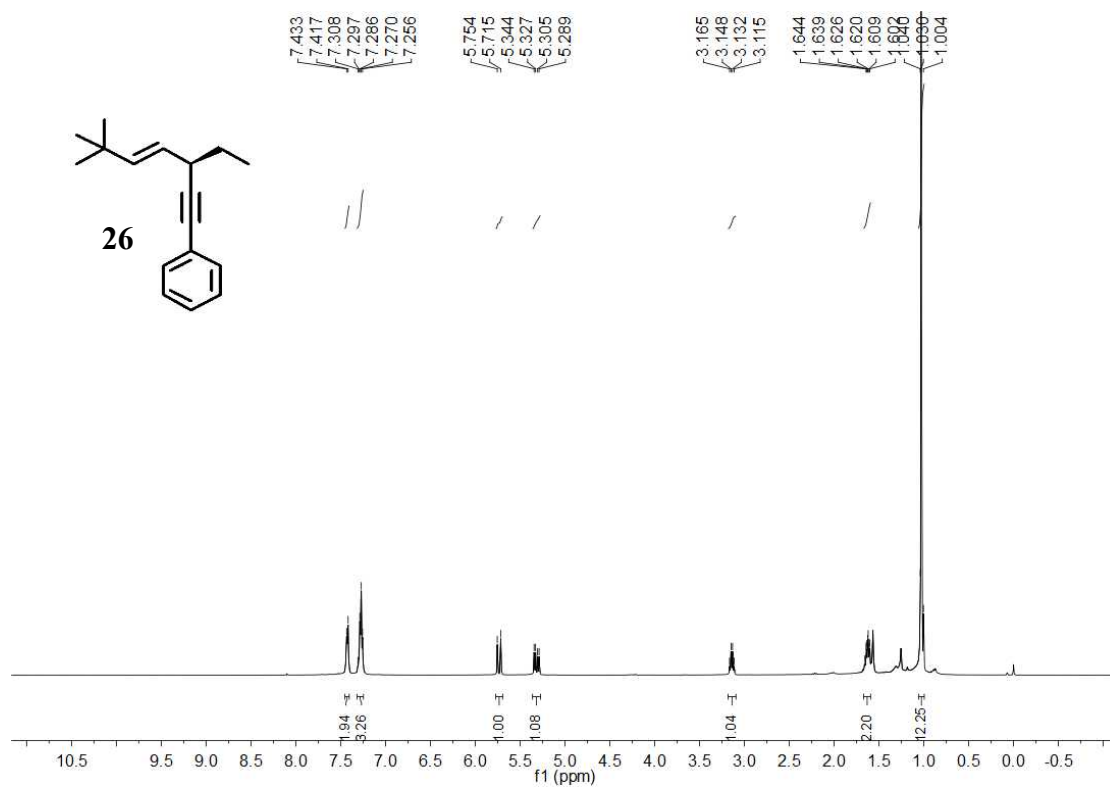
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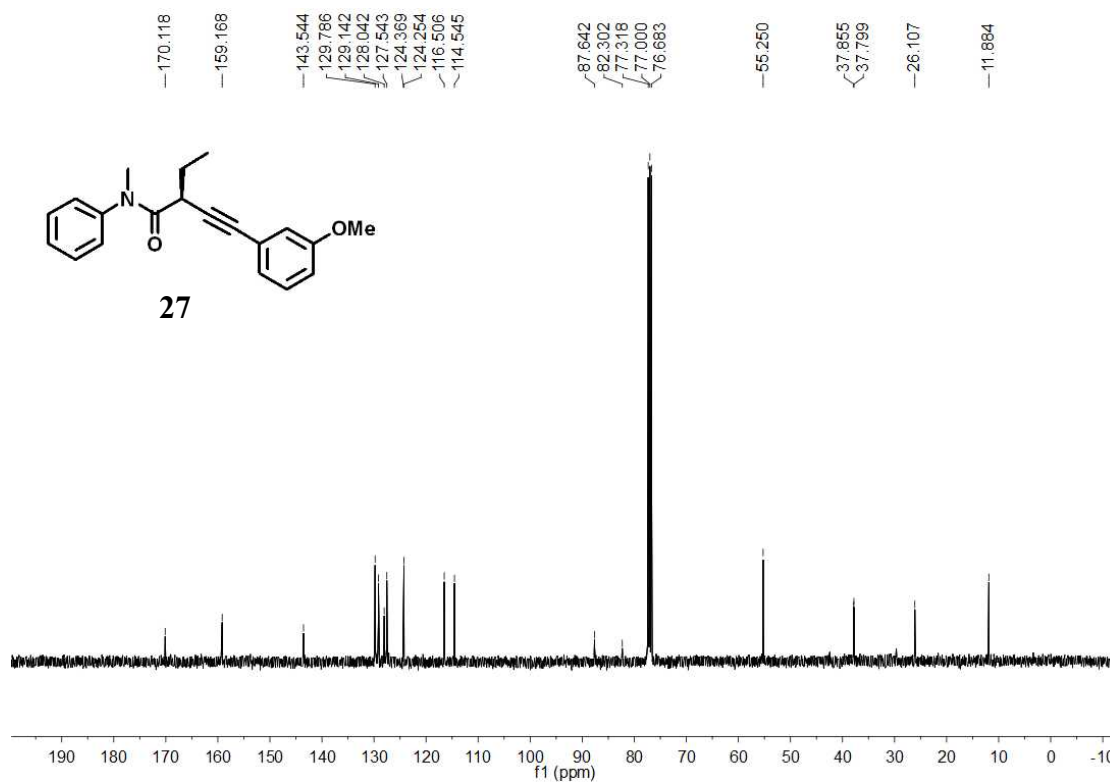
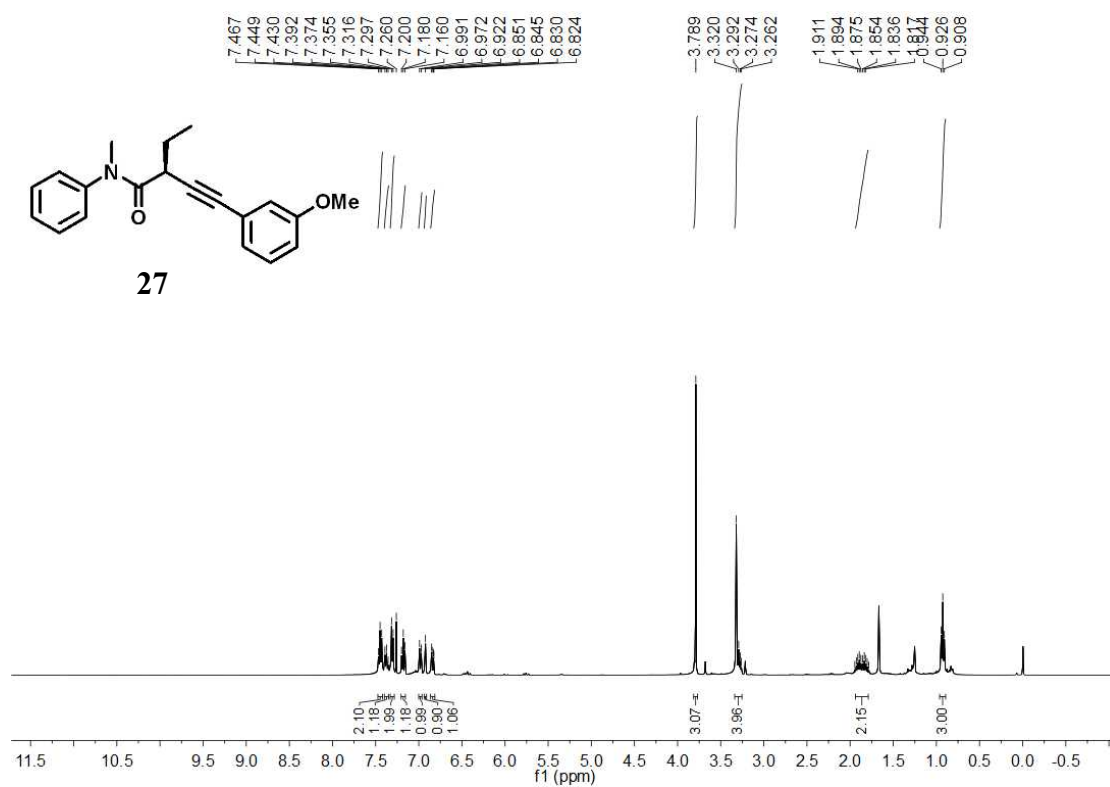
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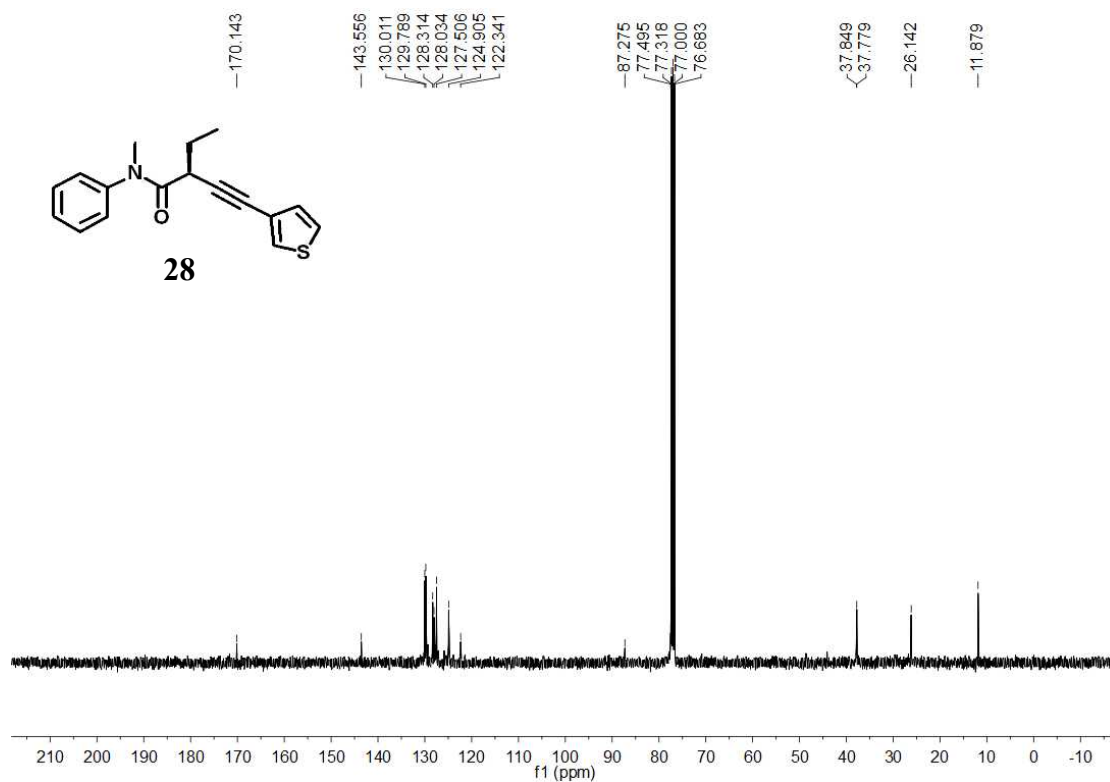
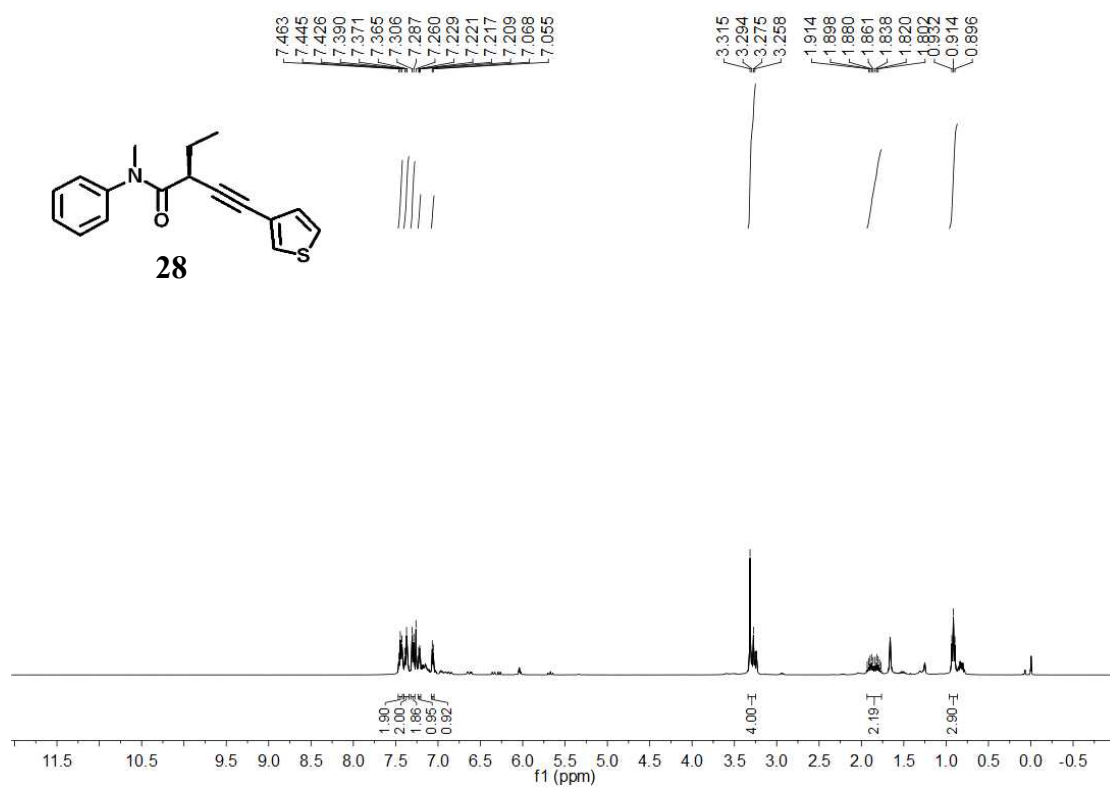
34.148
28.854

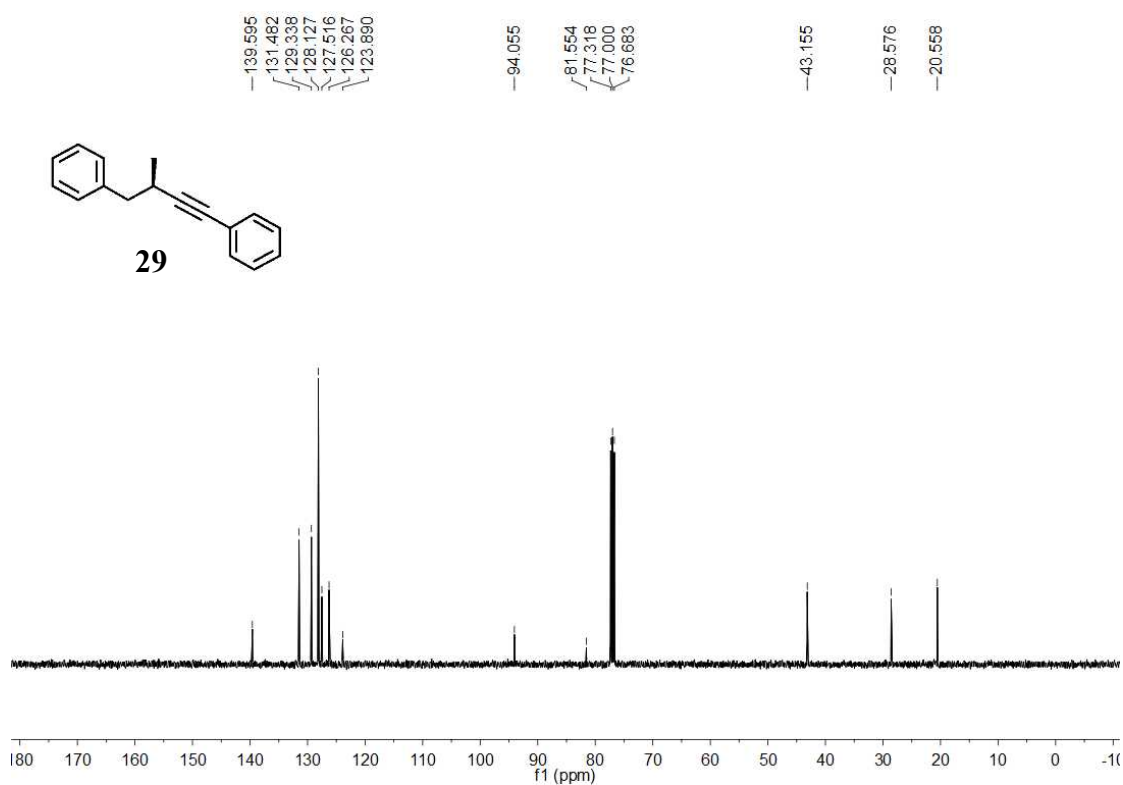
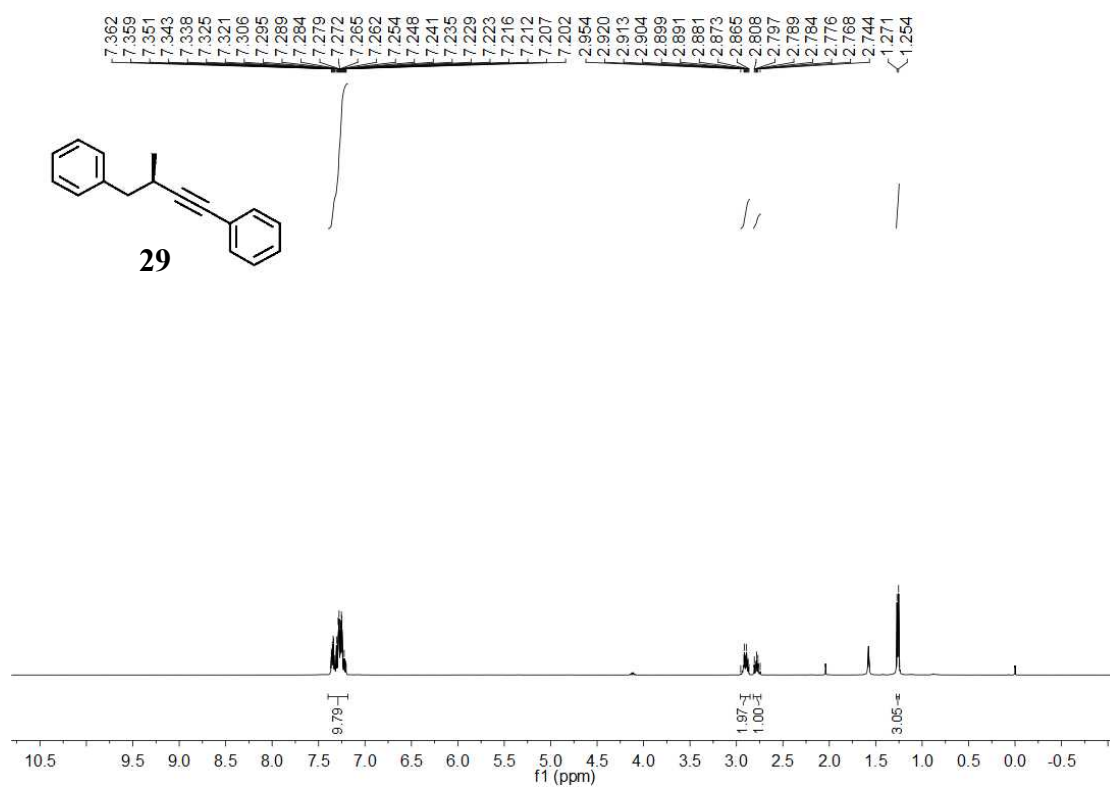
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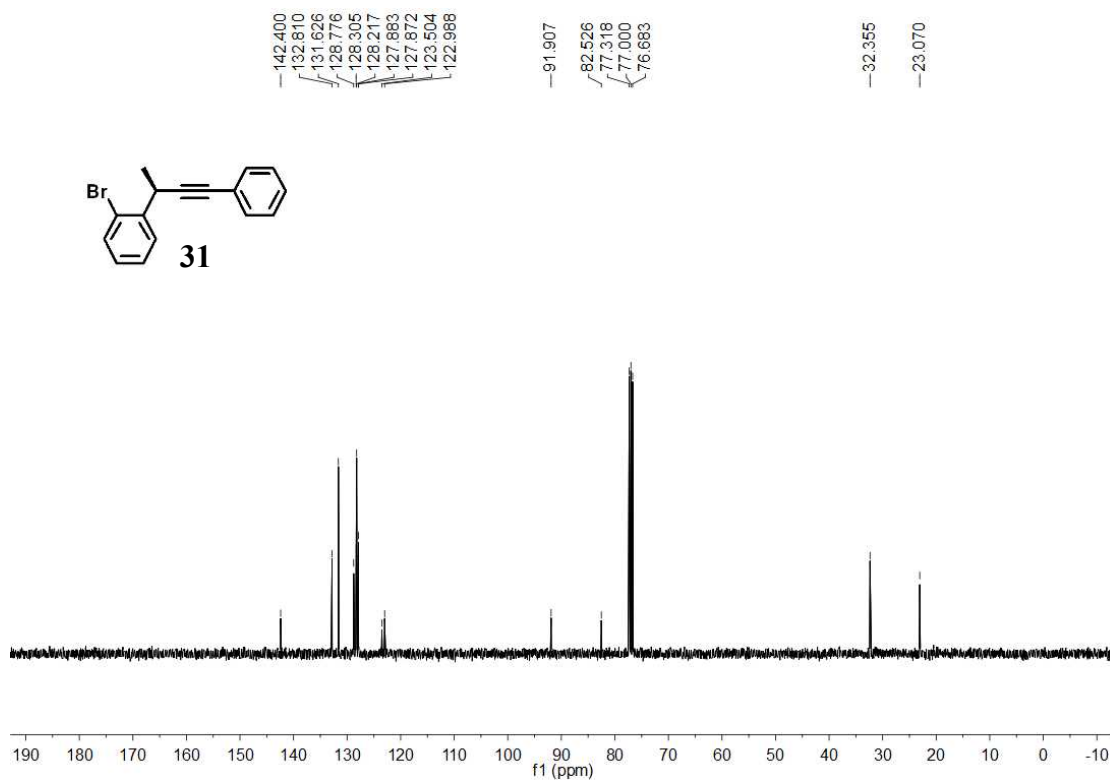
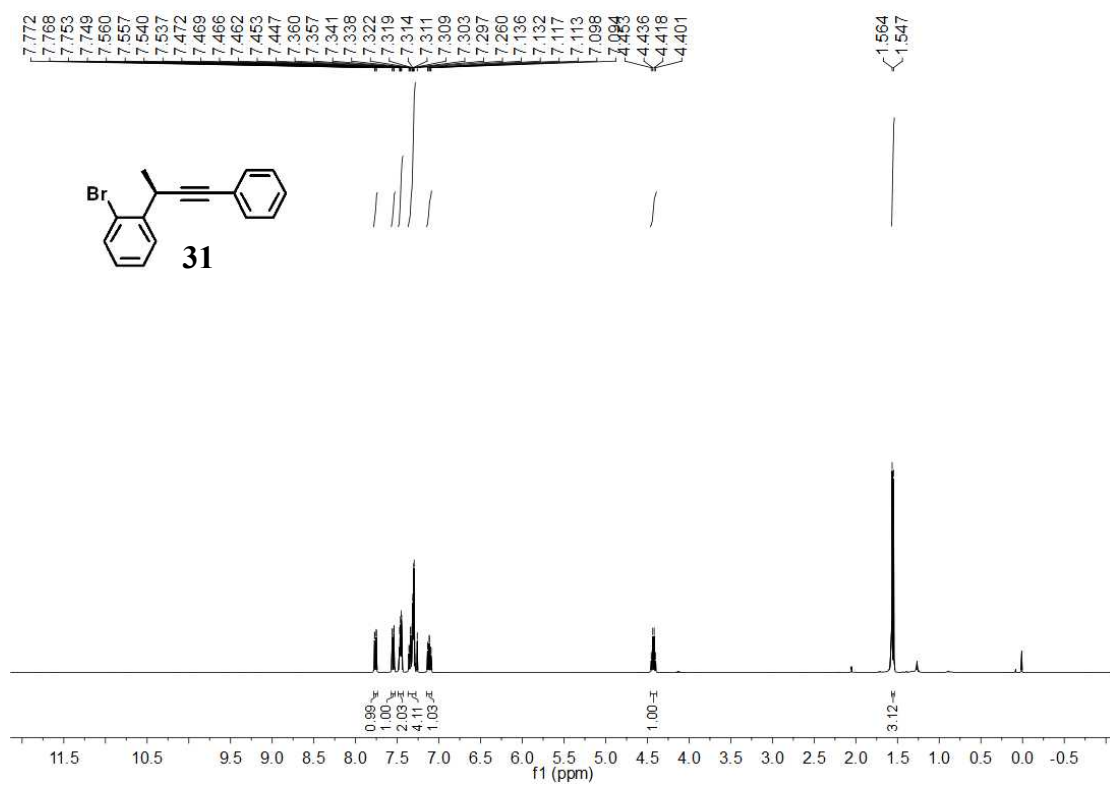


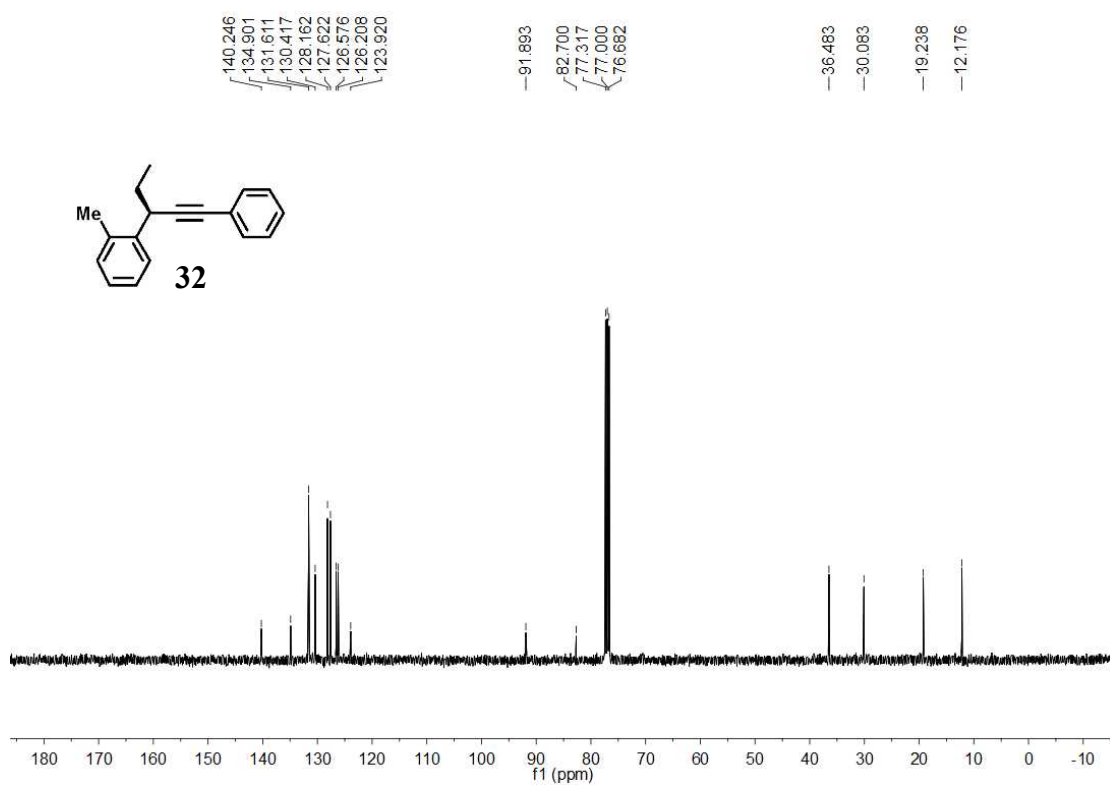
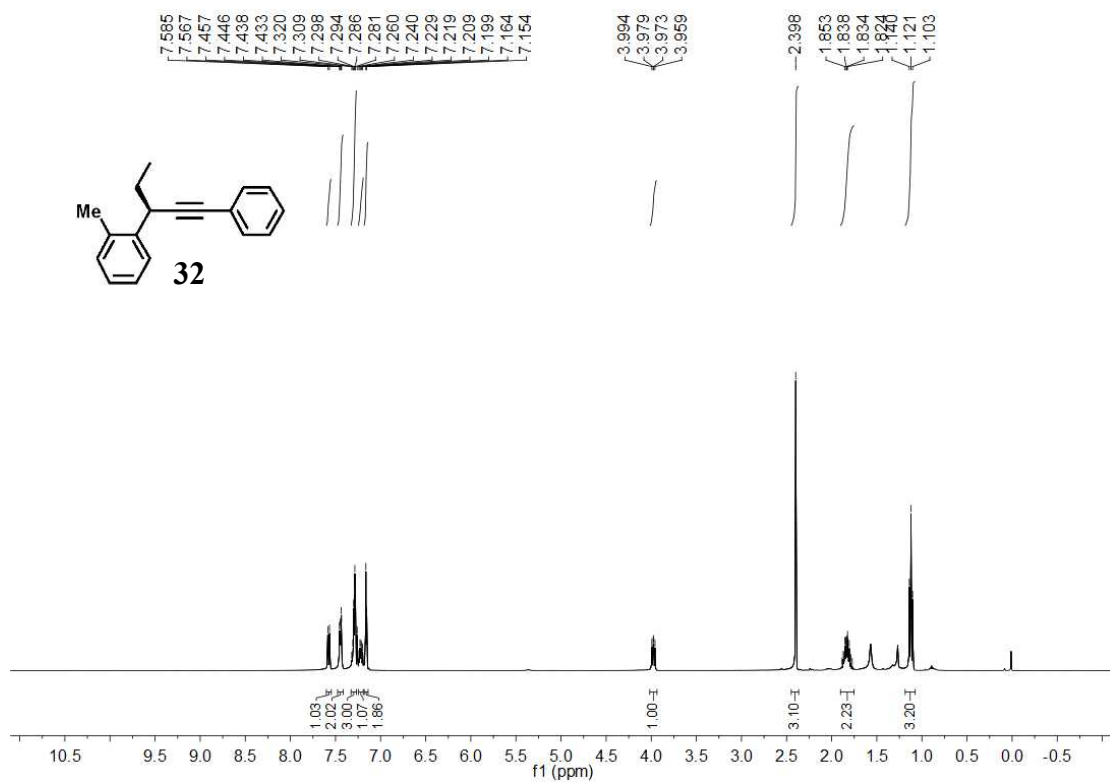


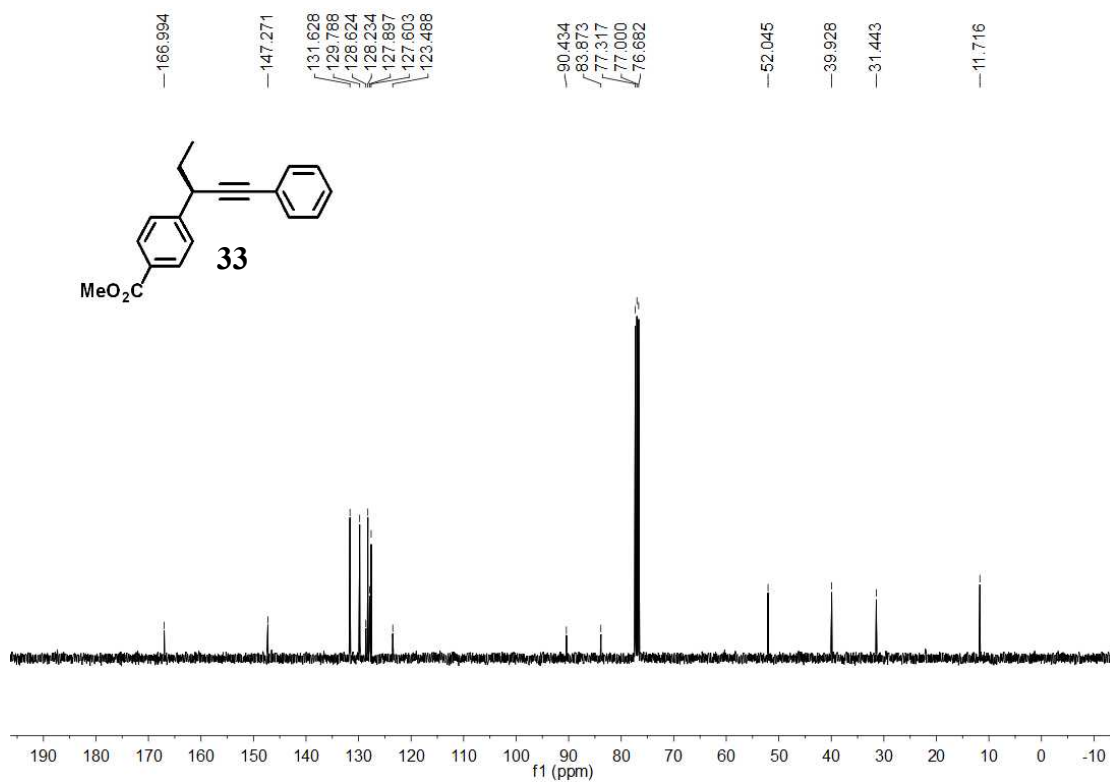
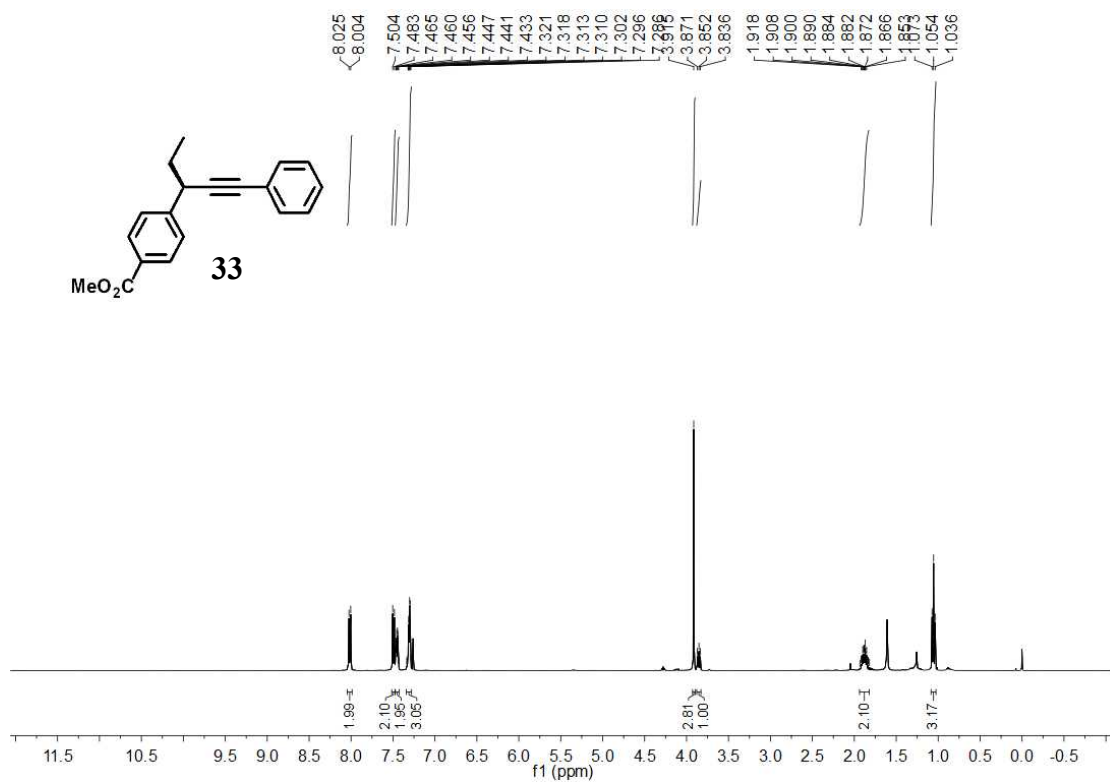


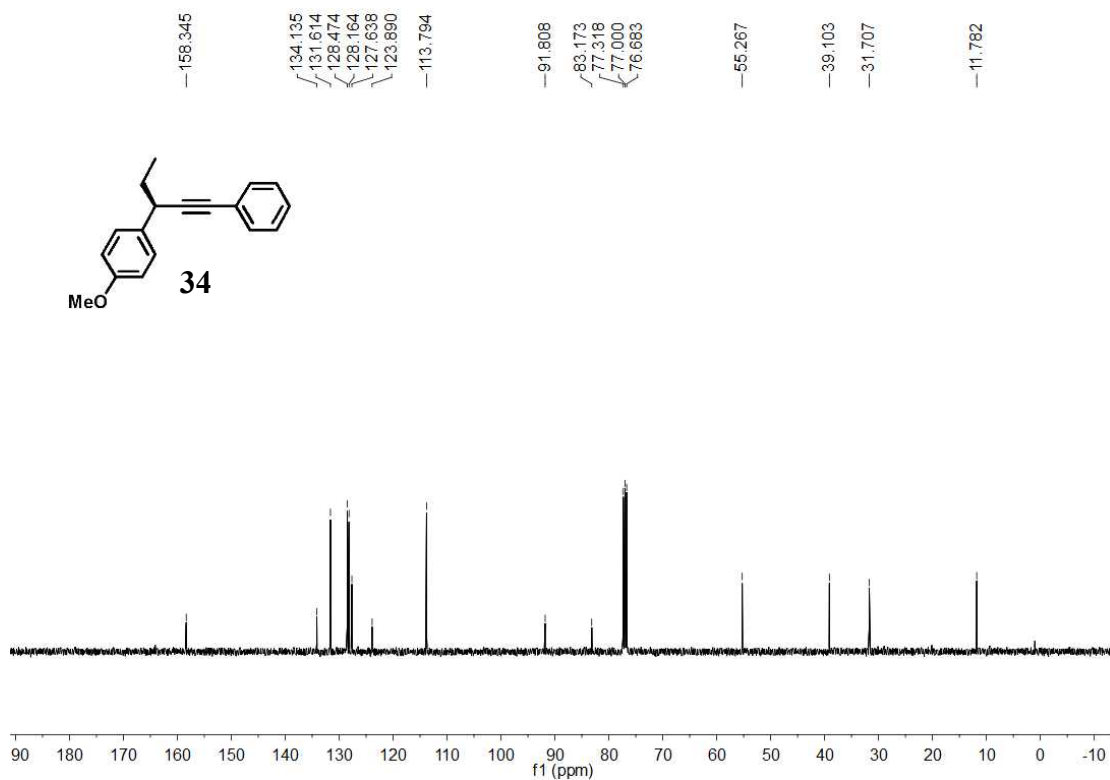
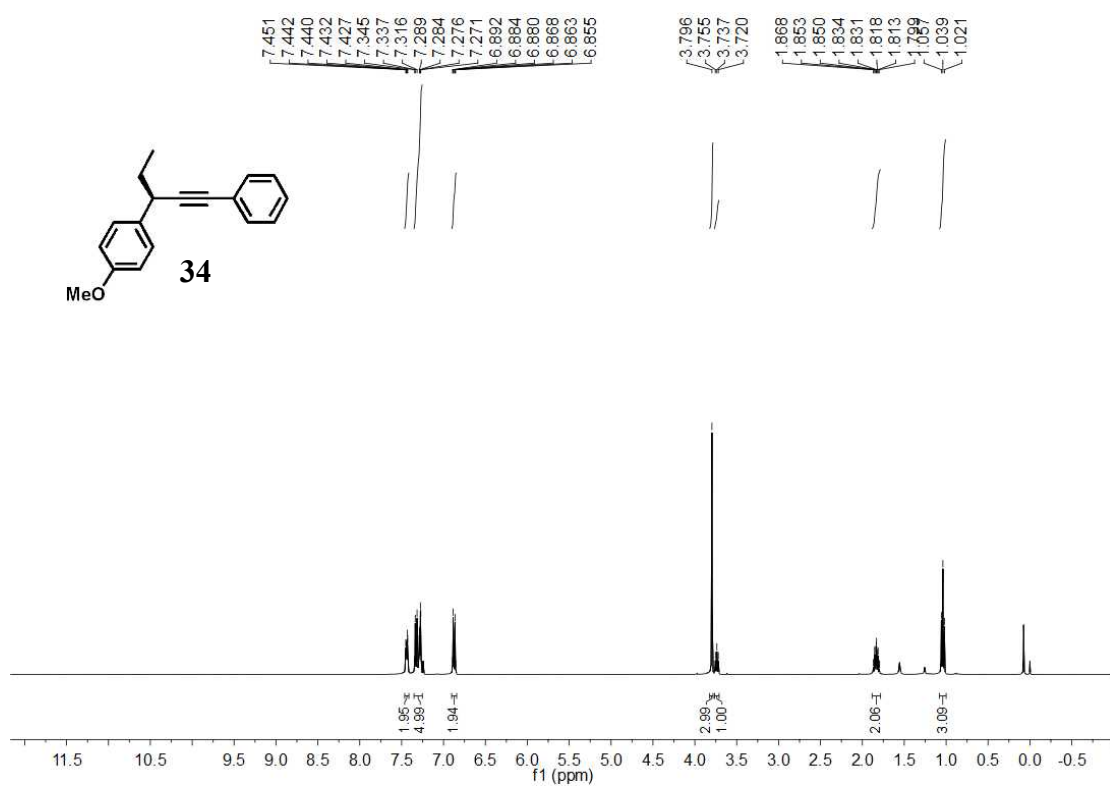


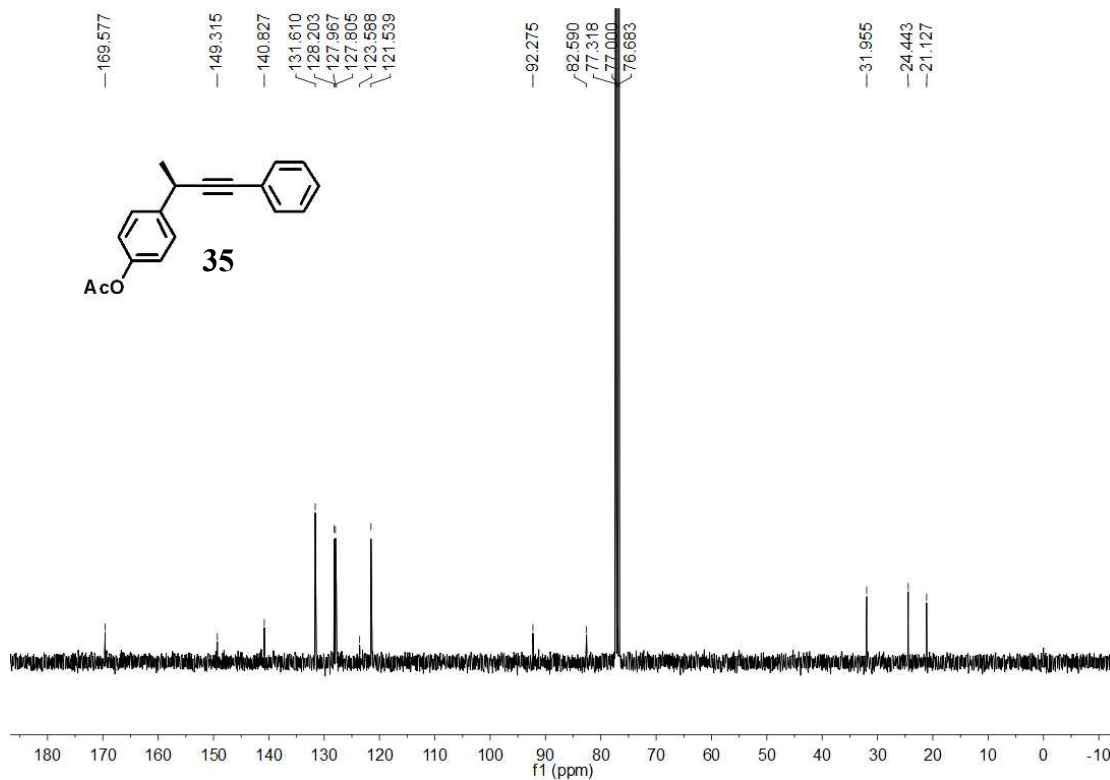
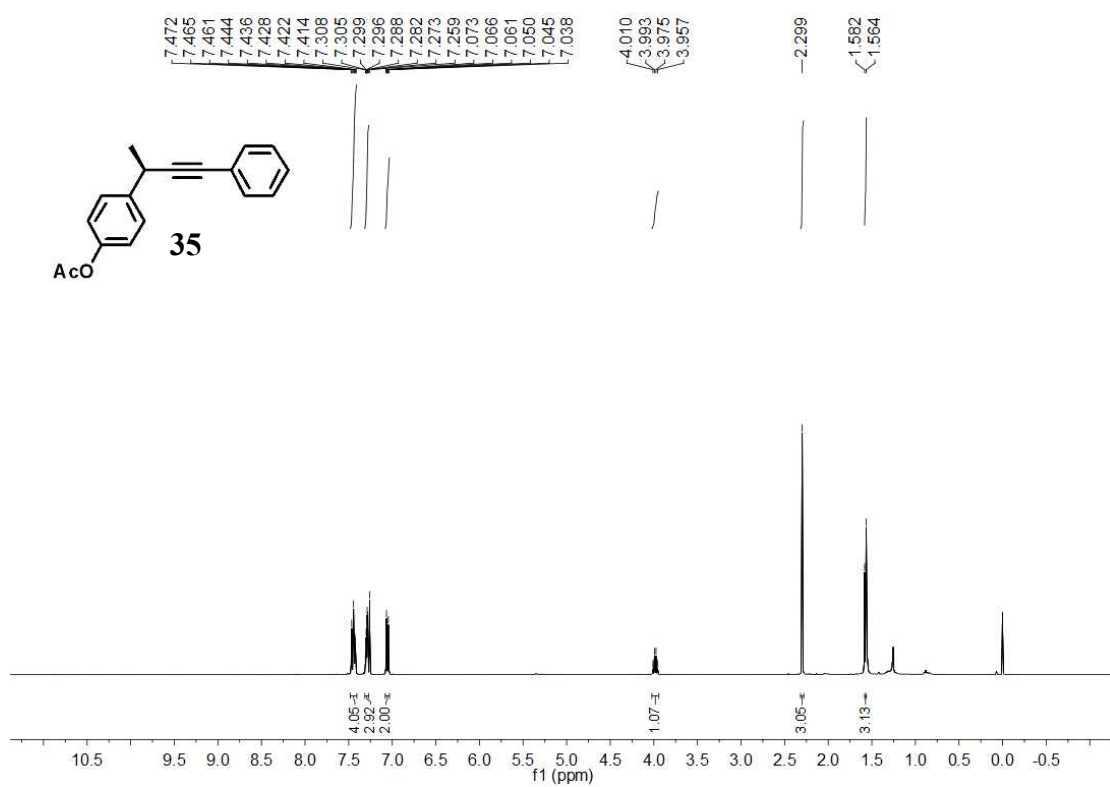


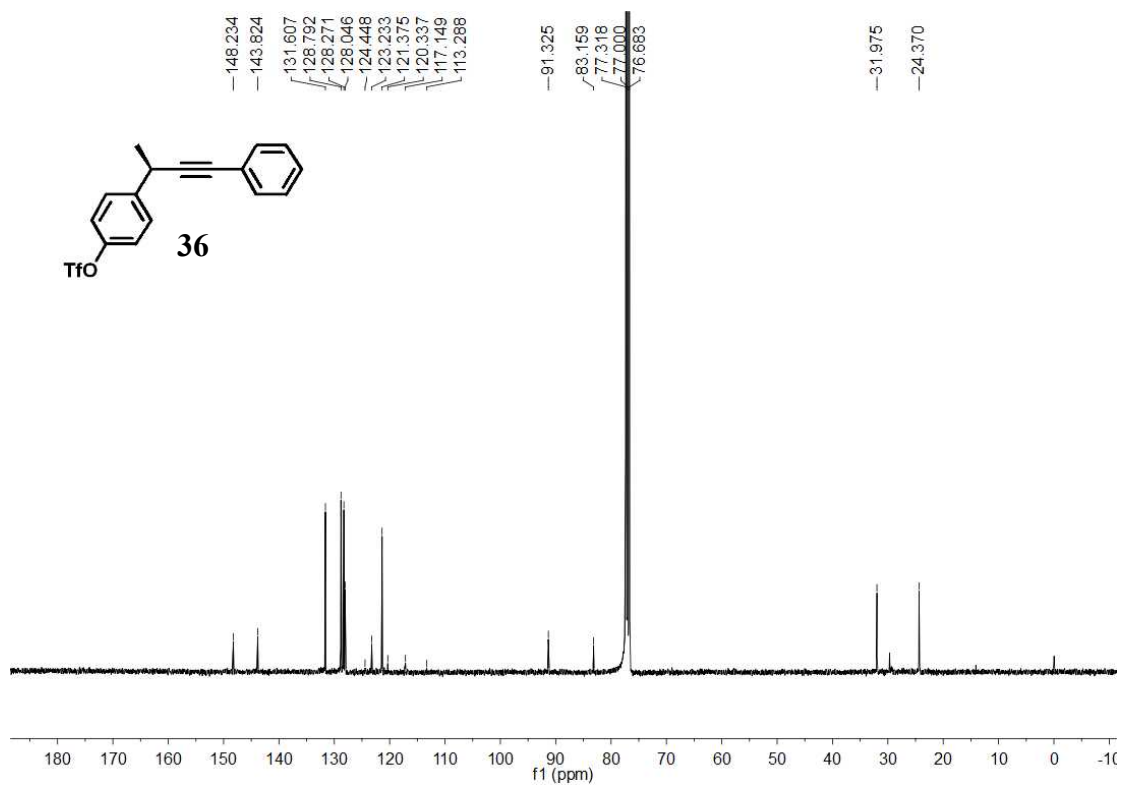
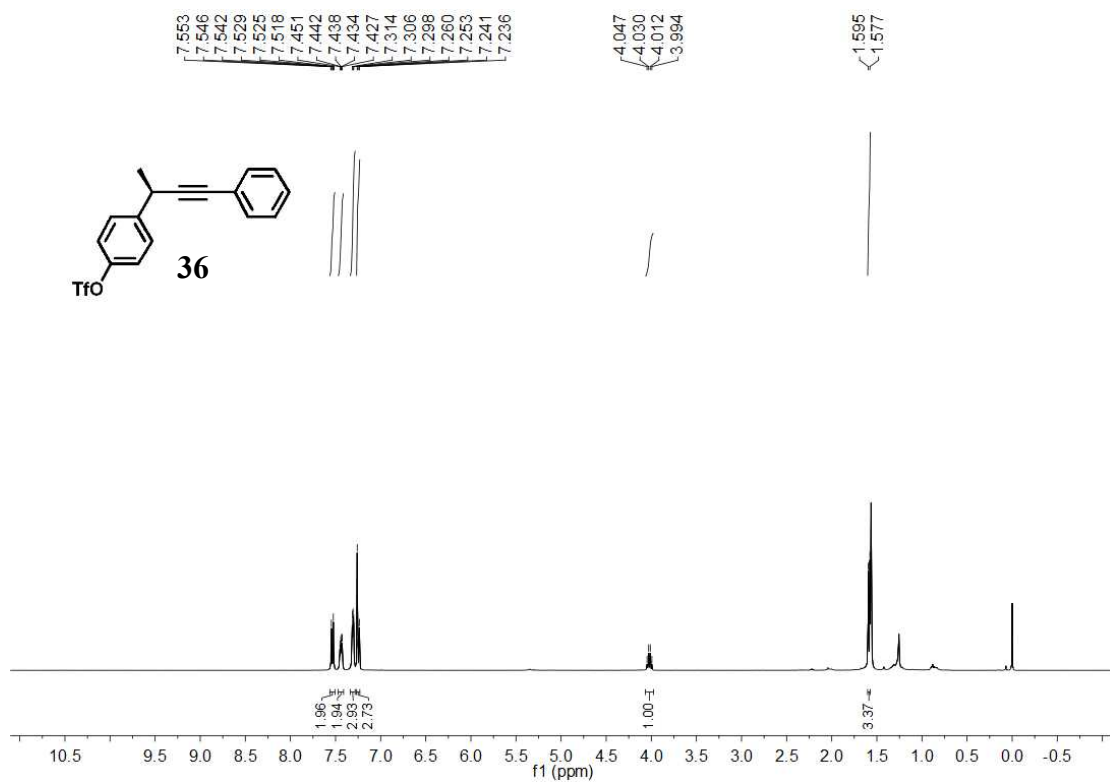


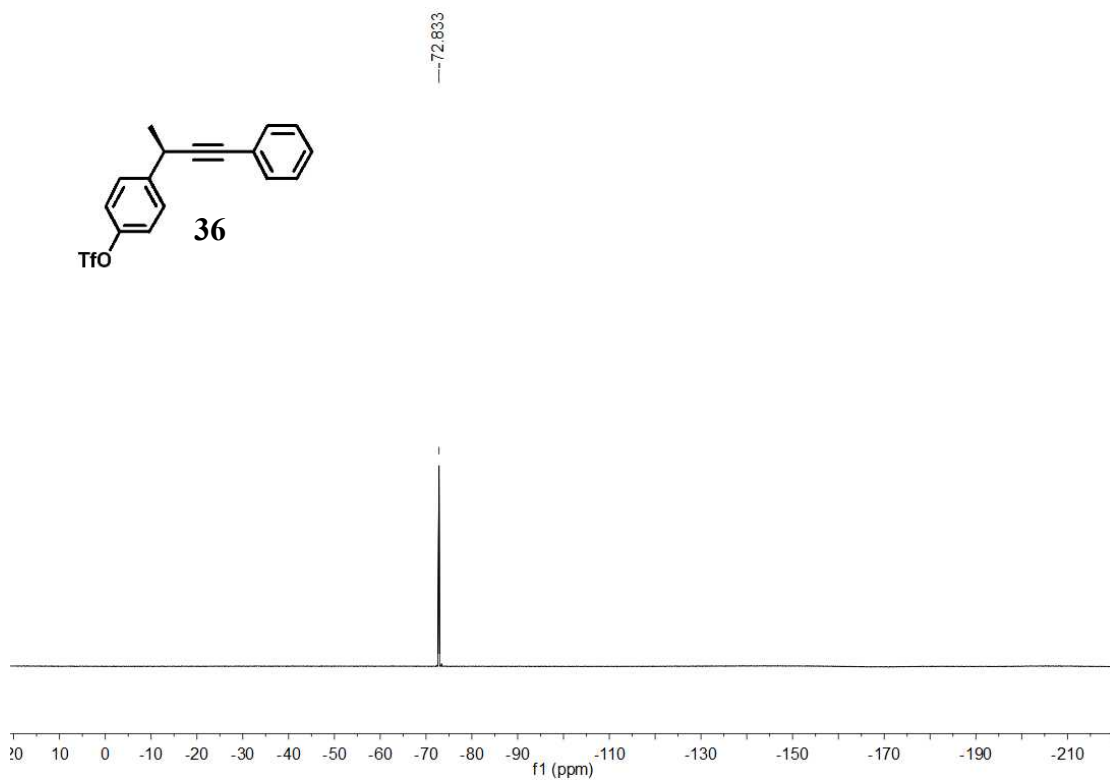


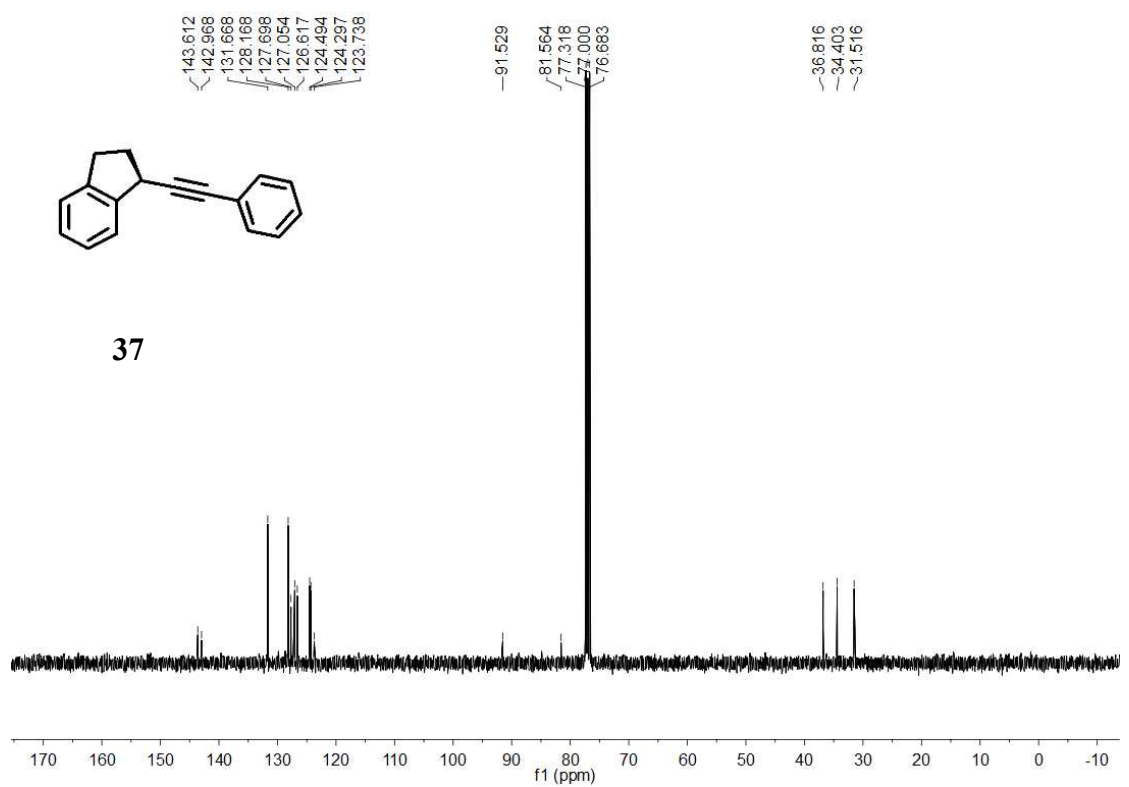
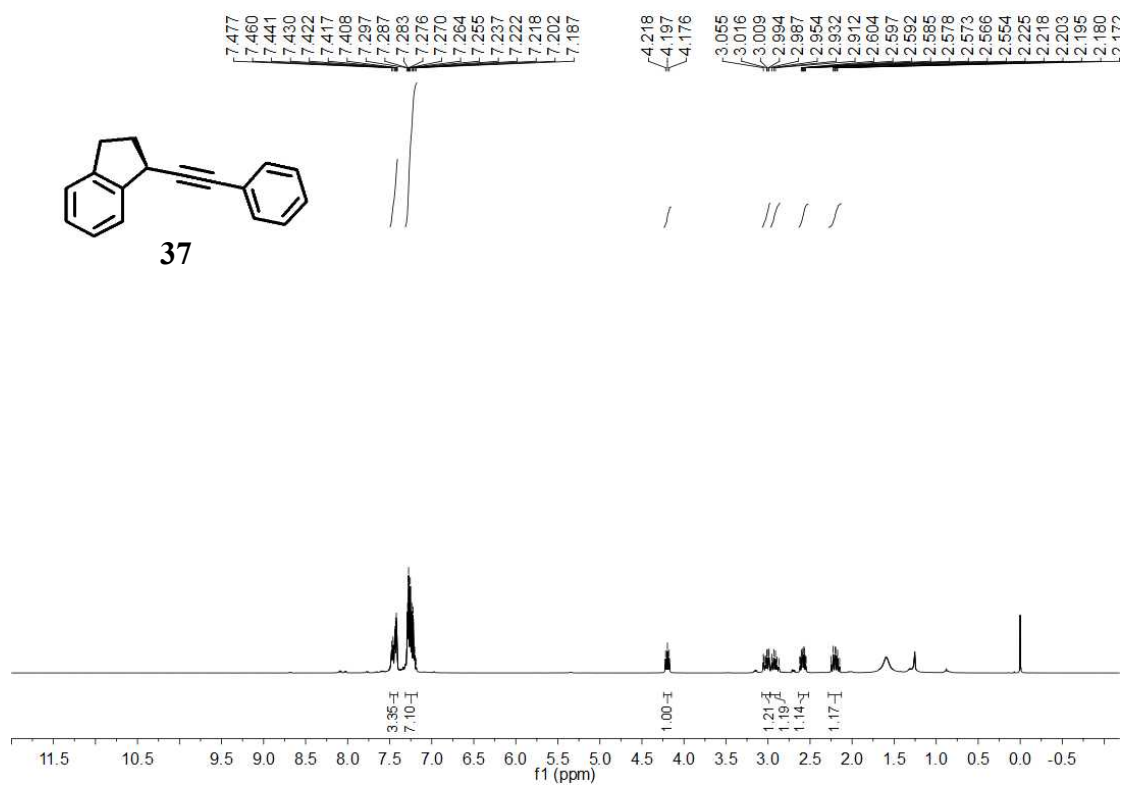


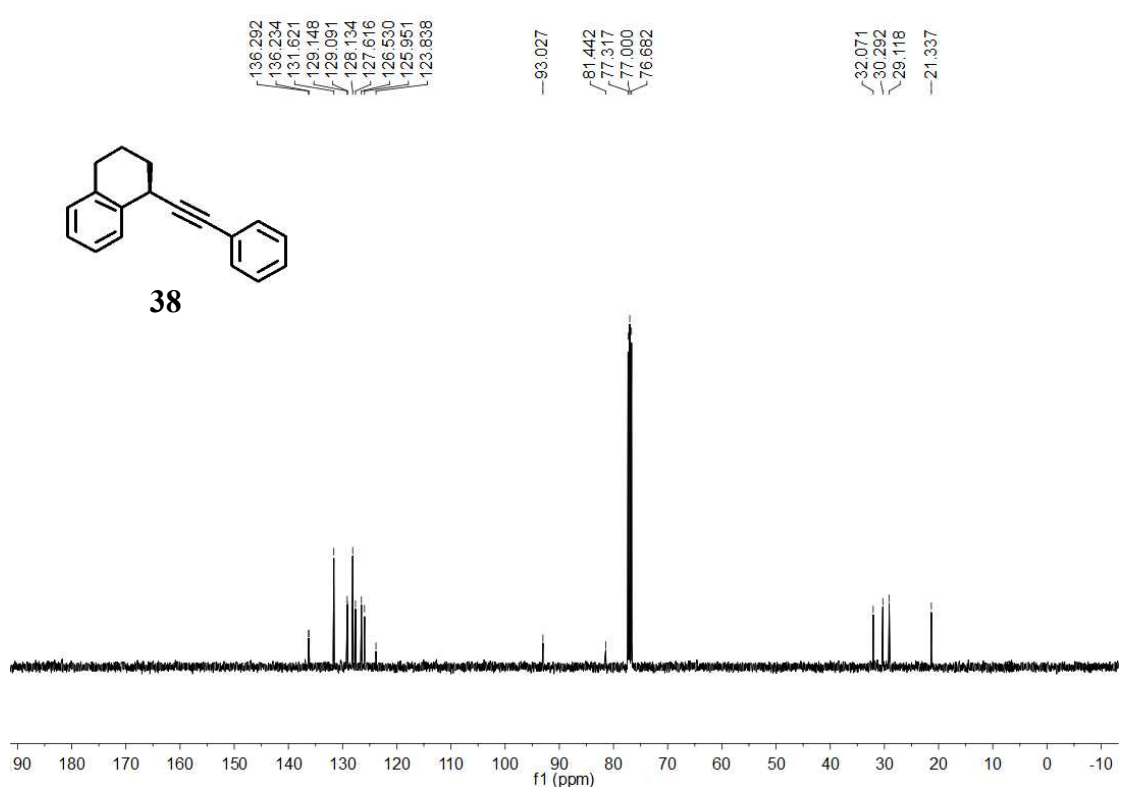
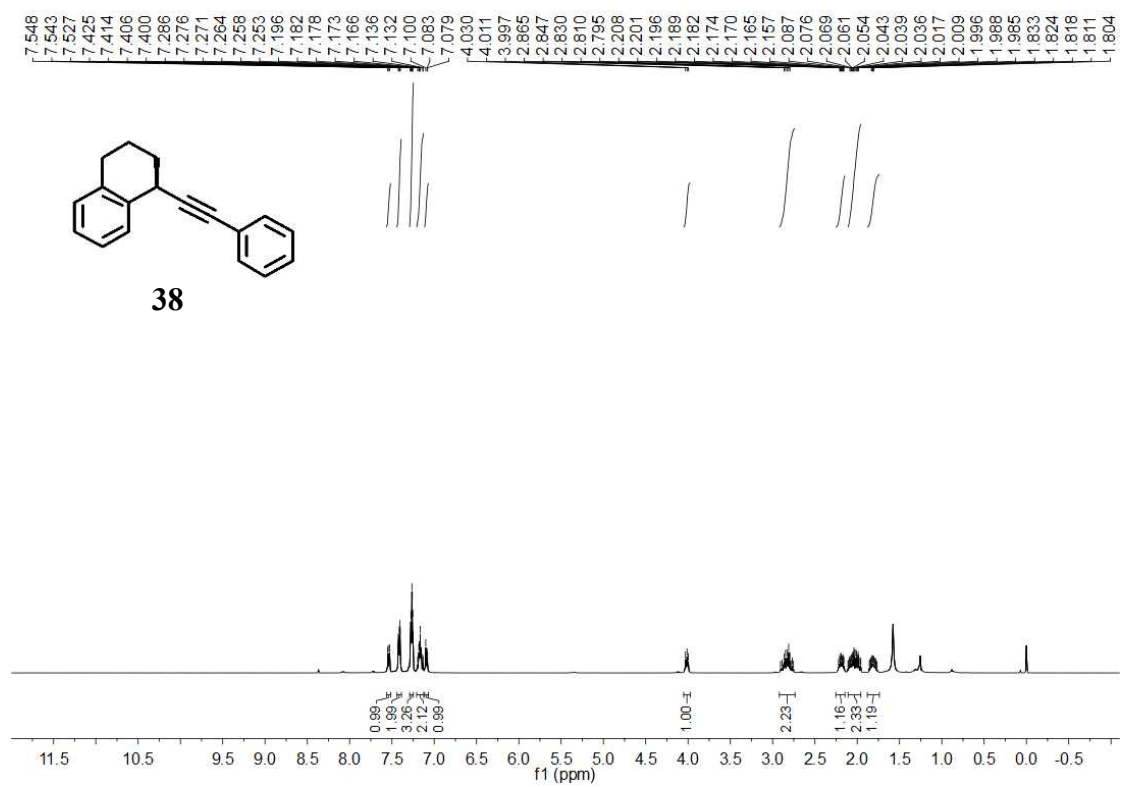


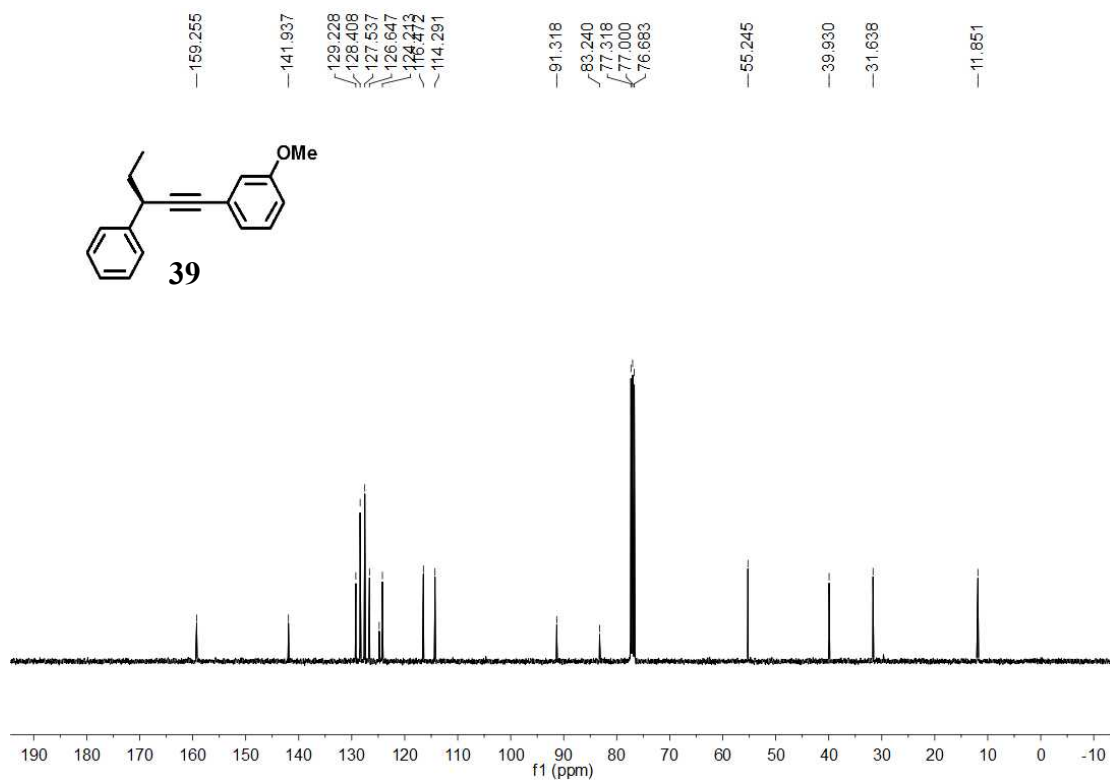
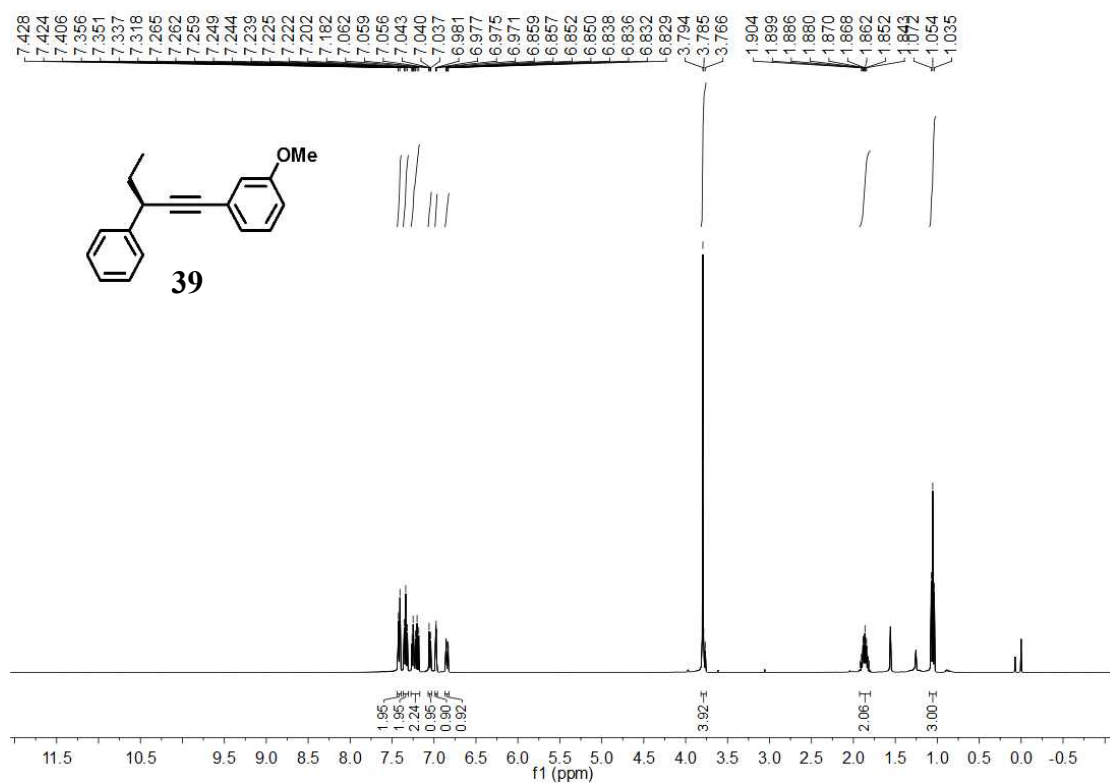


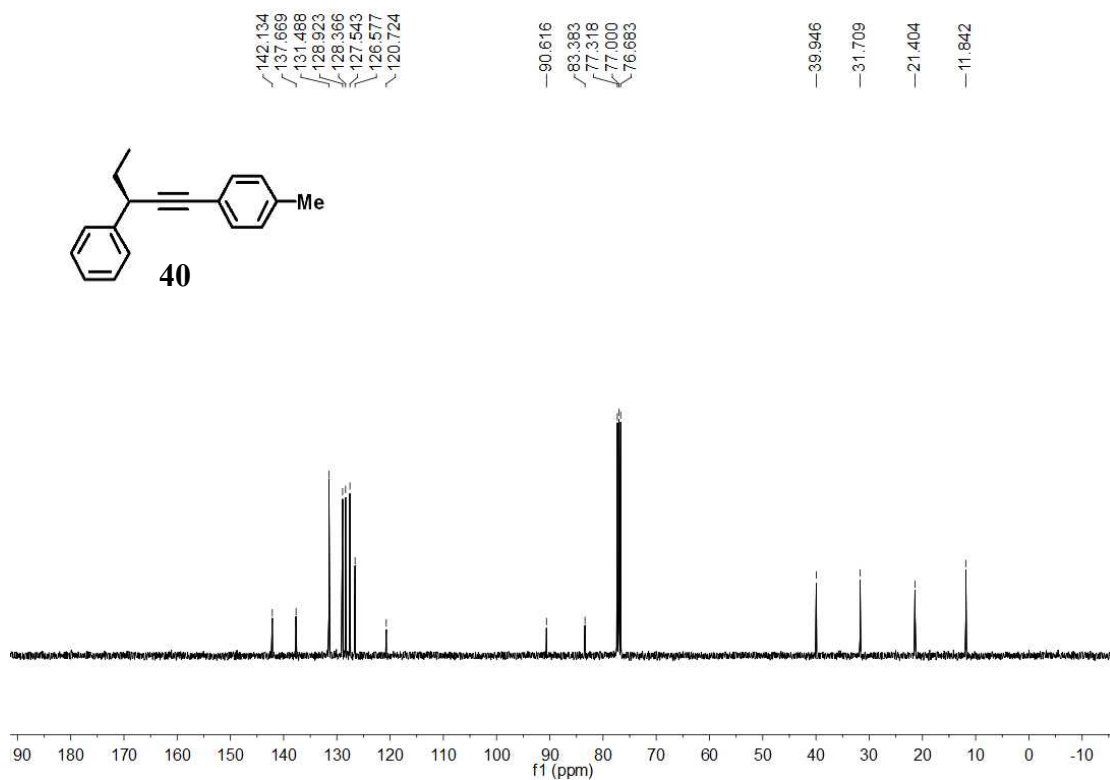
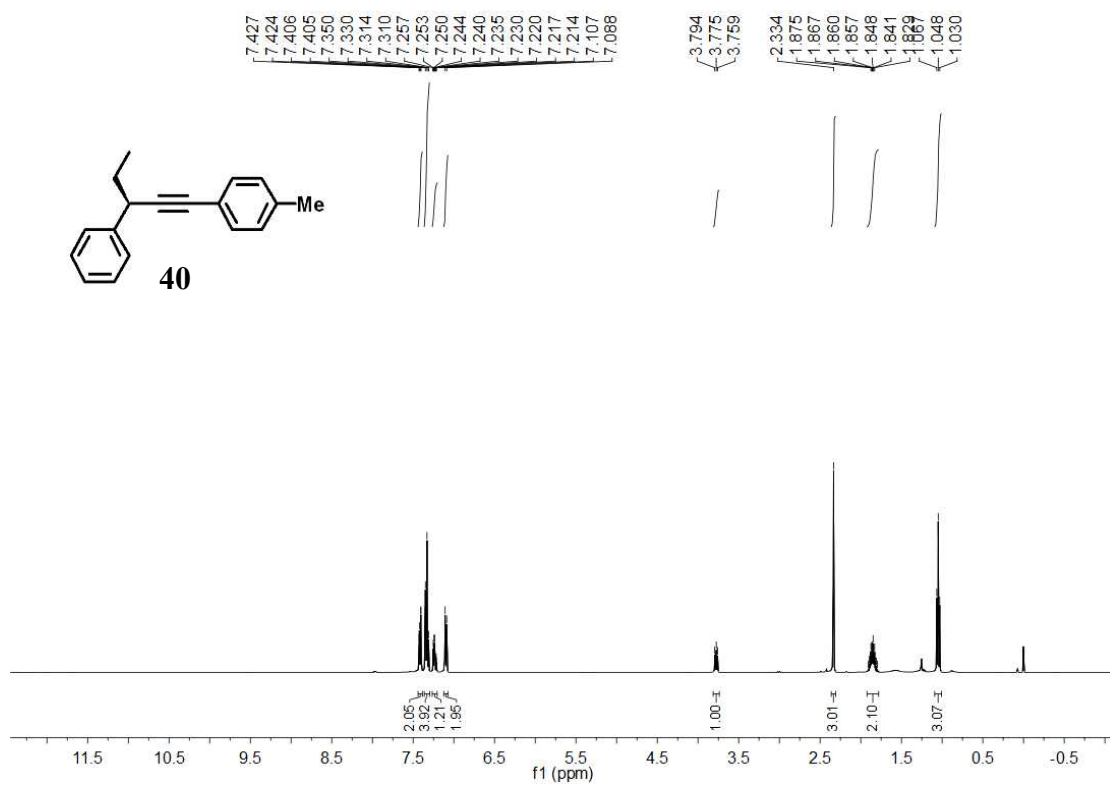


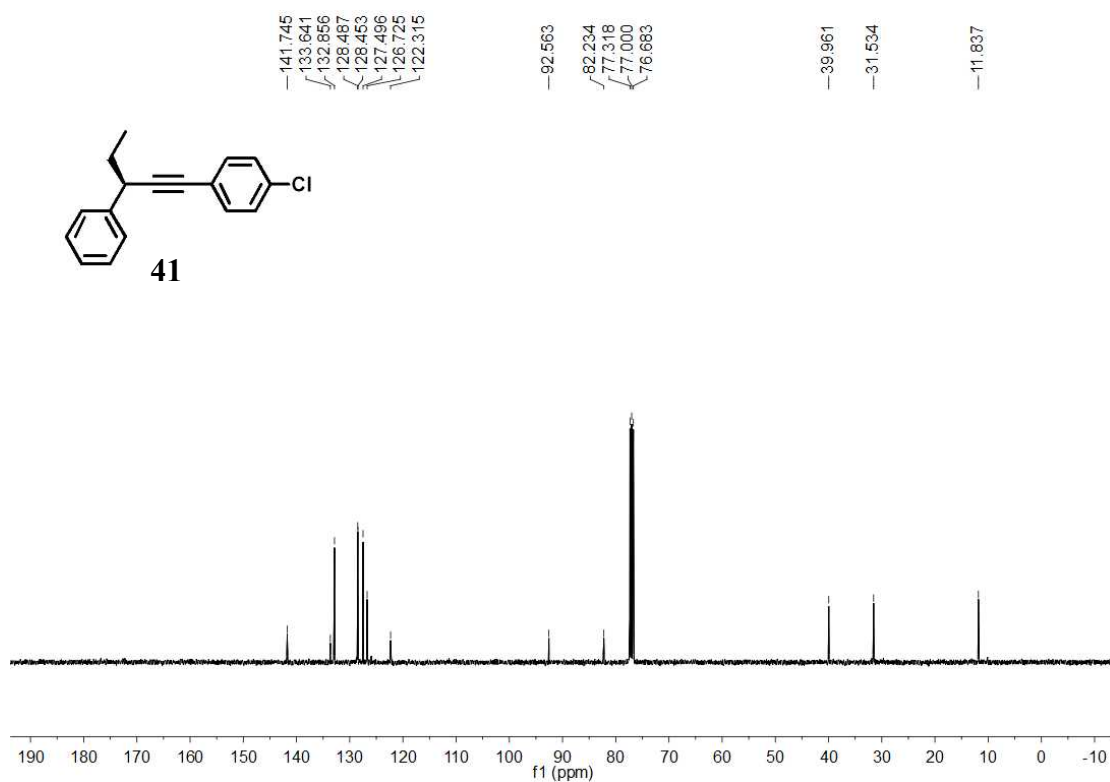
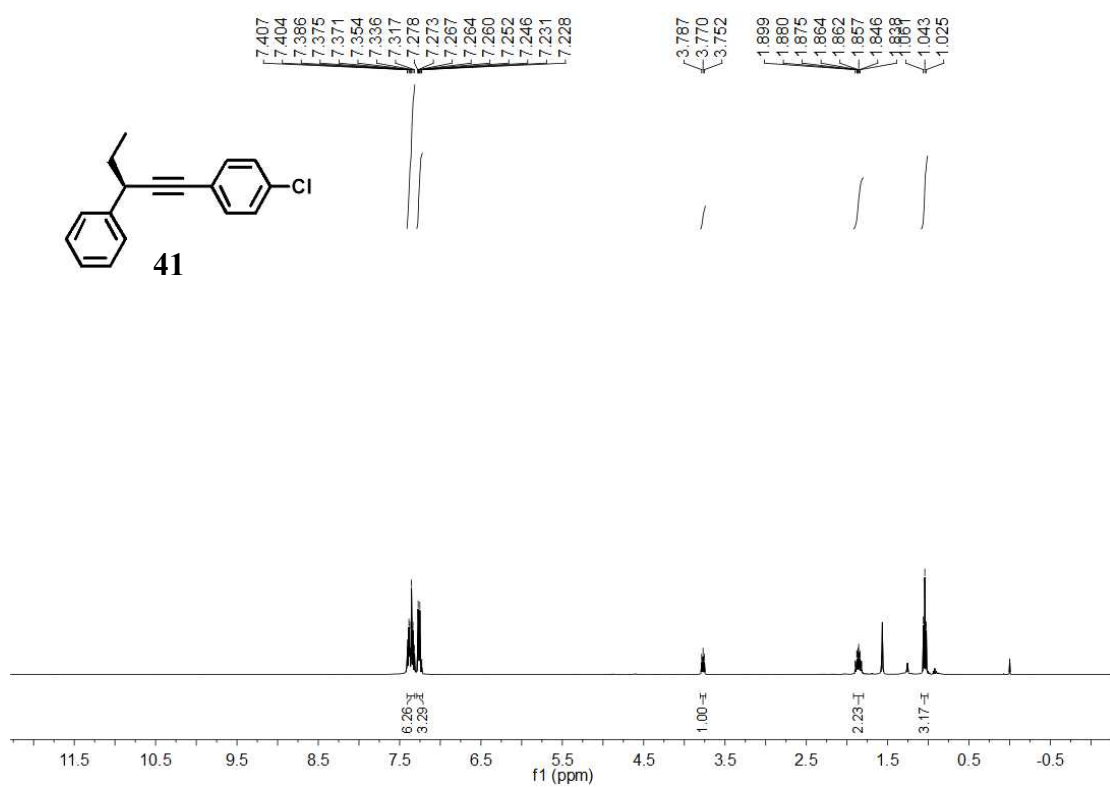


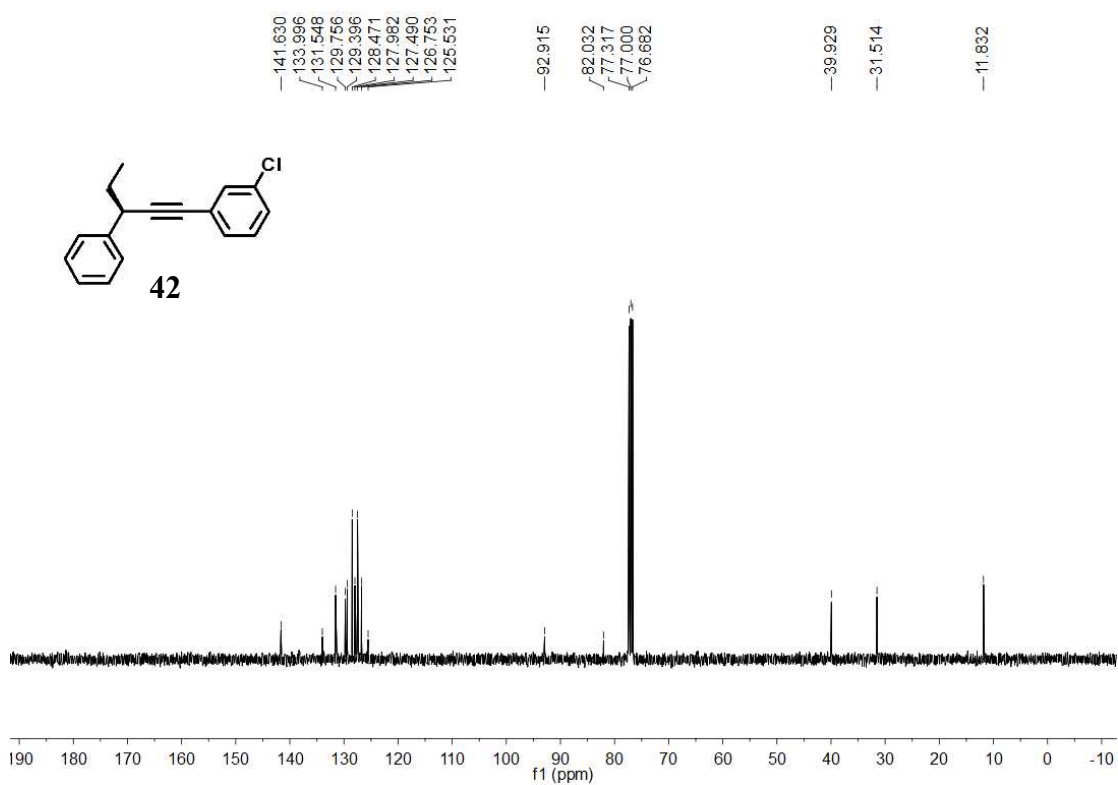
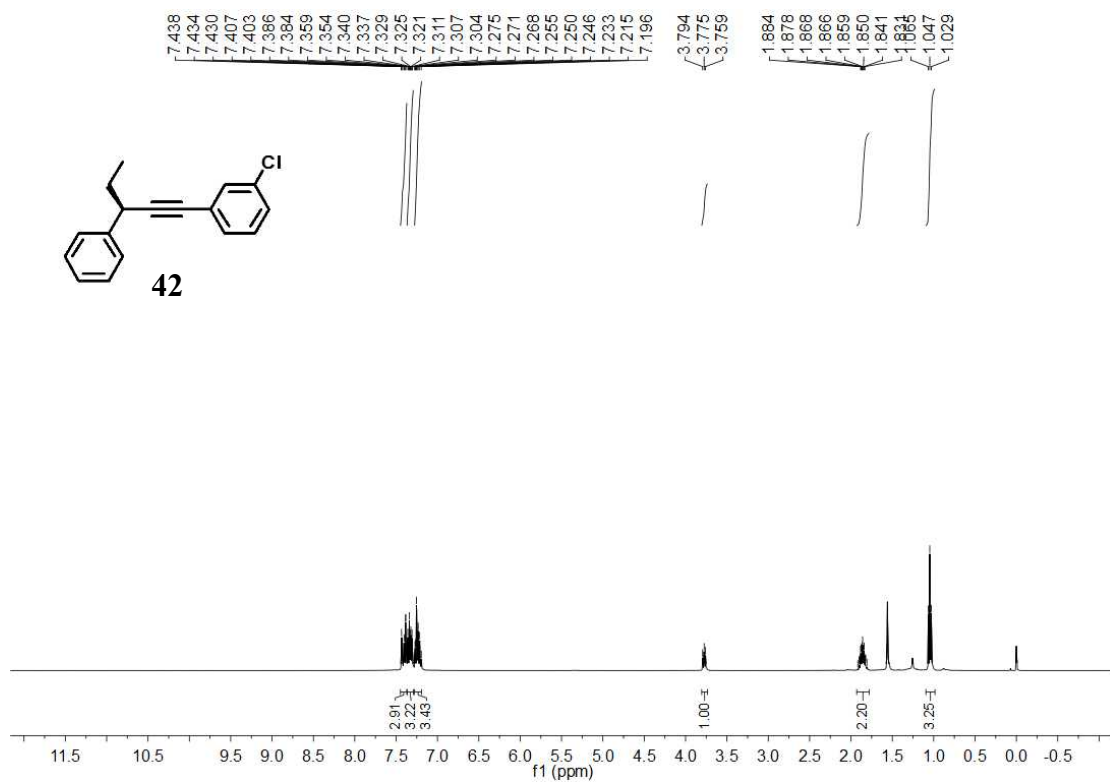


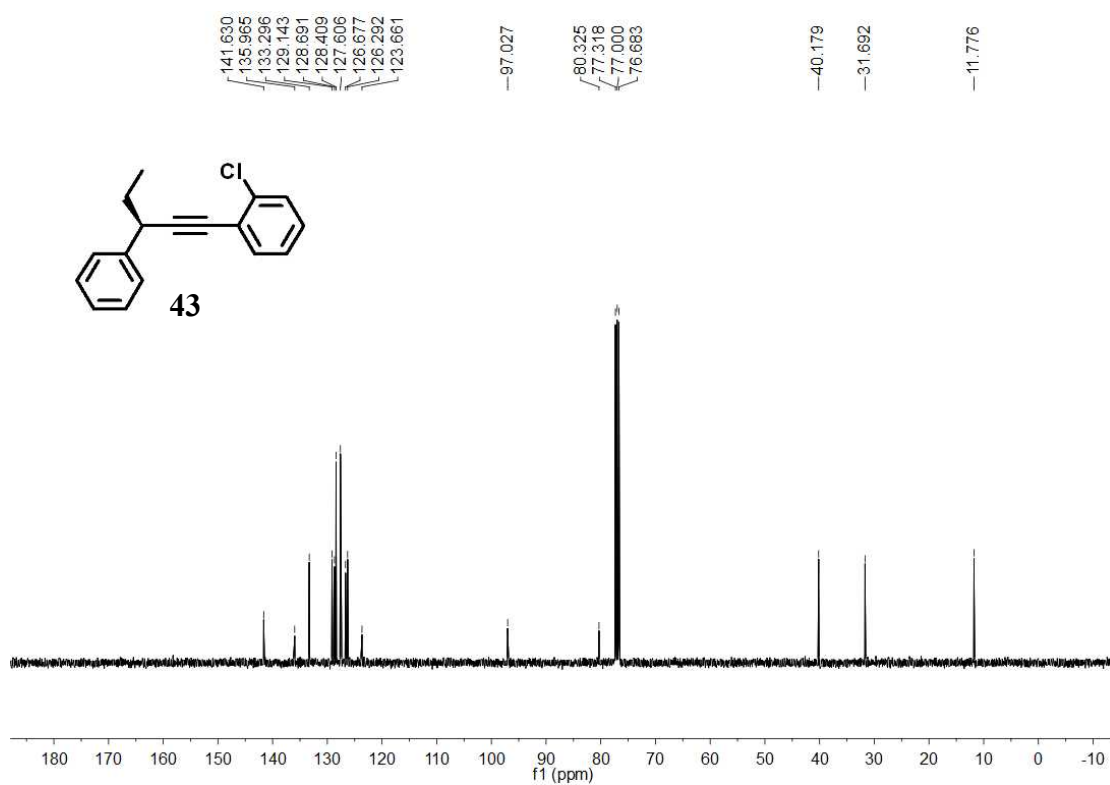
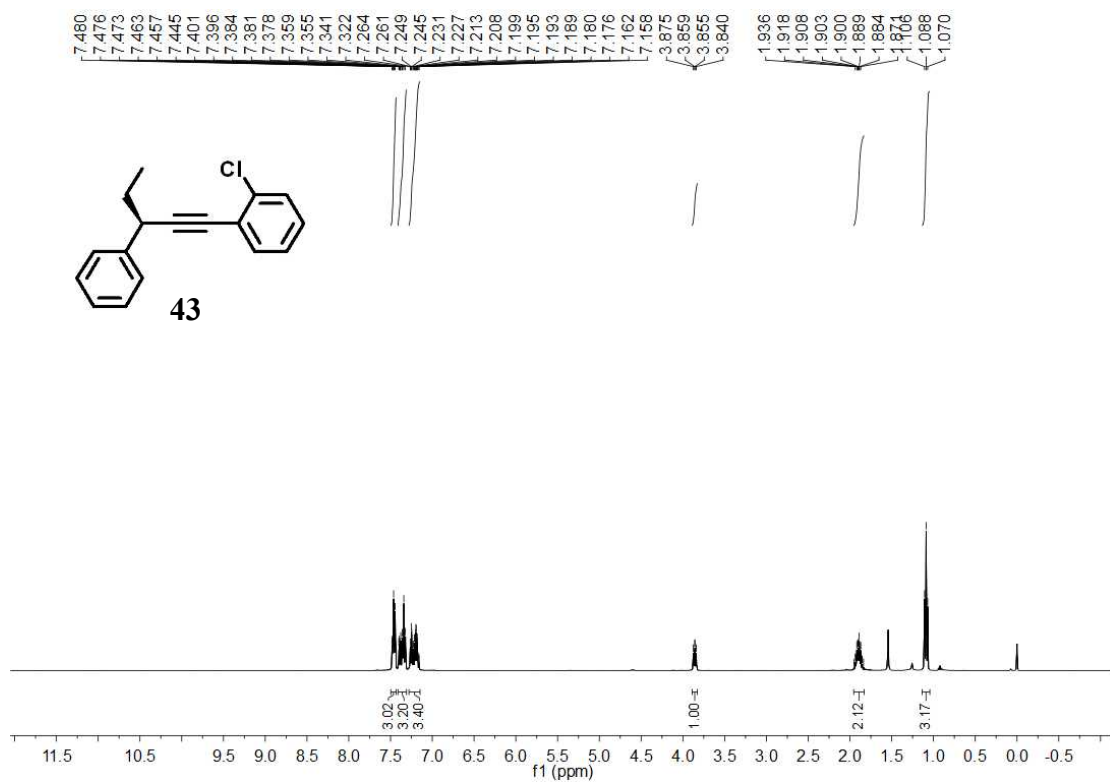


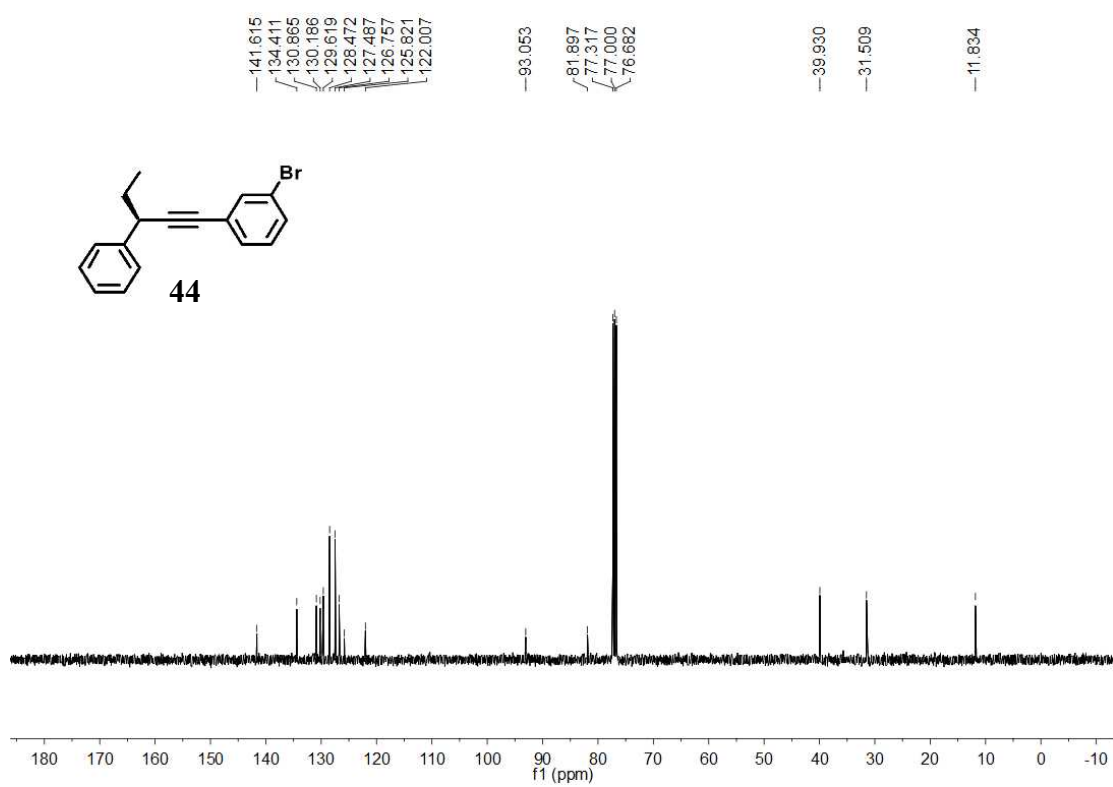
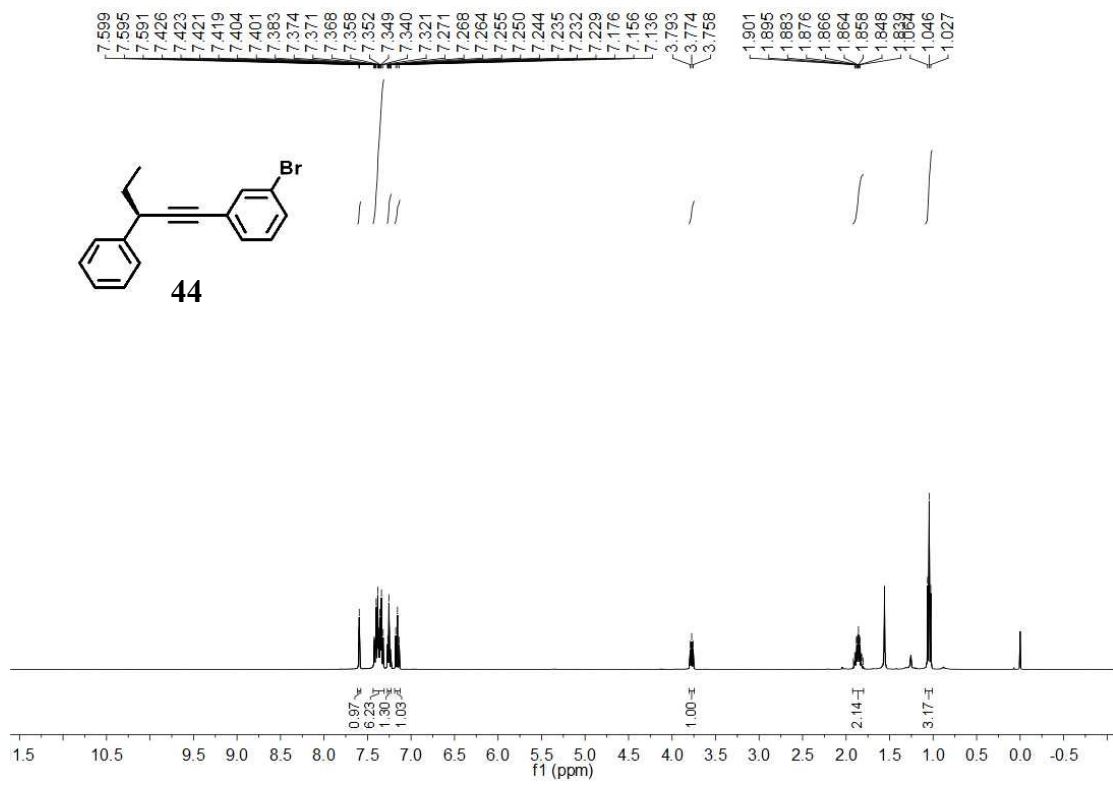


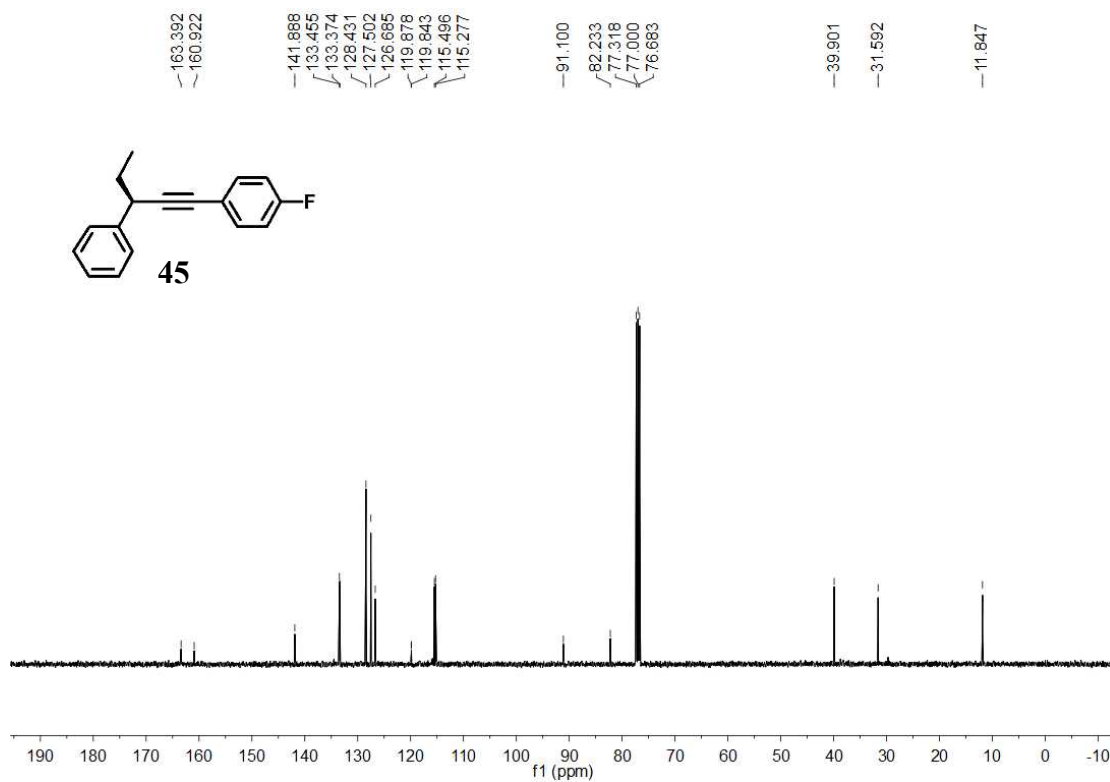
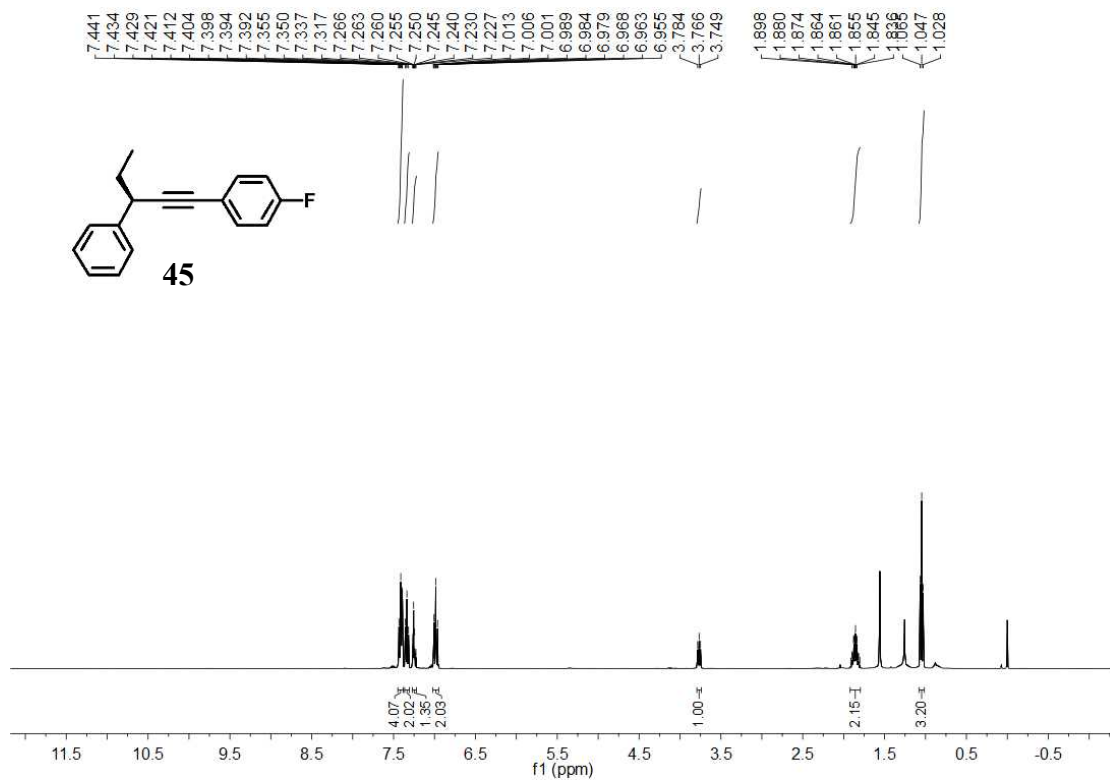


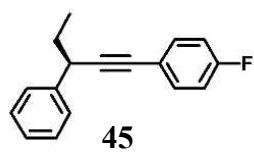




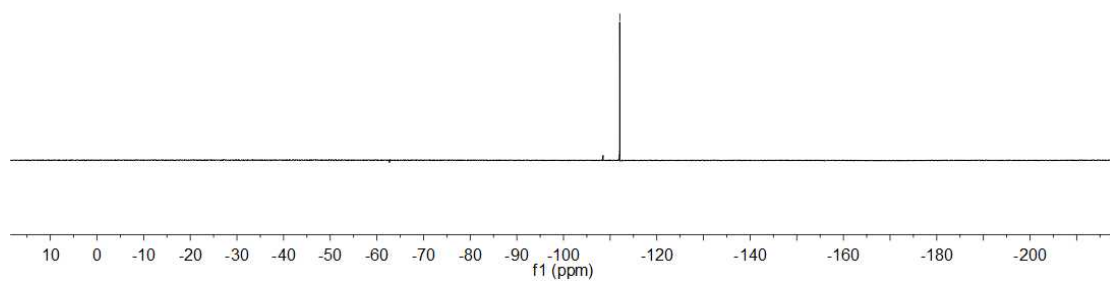


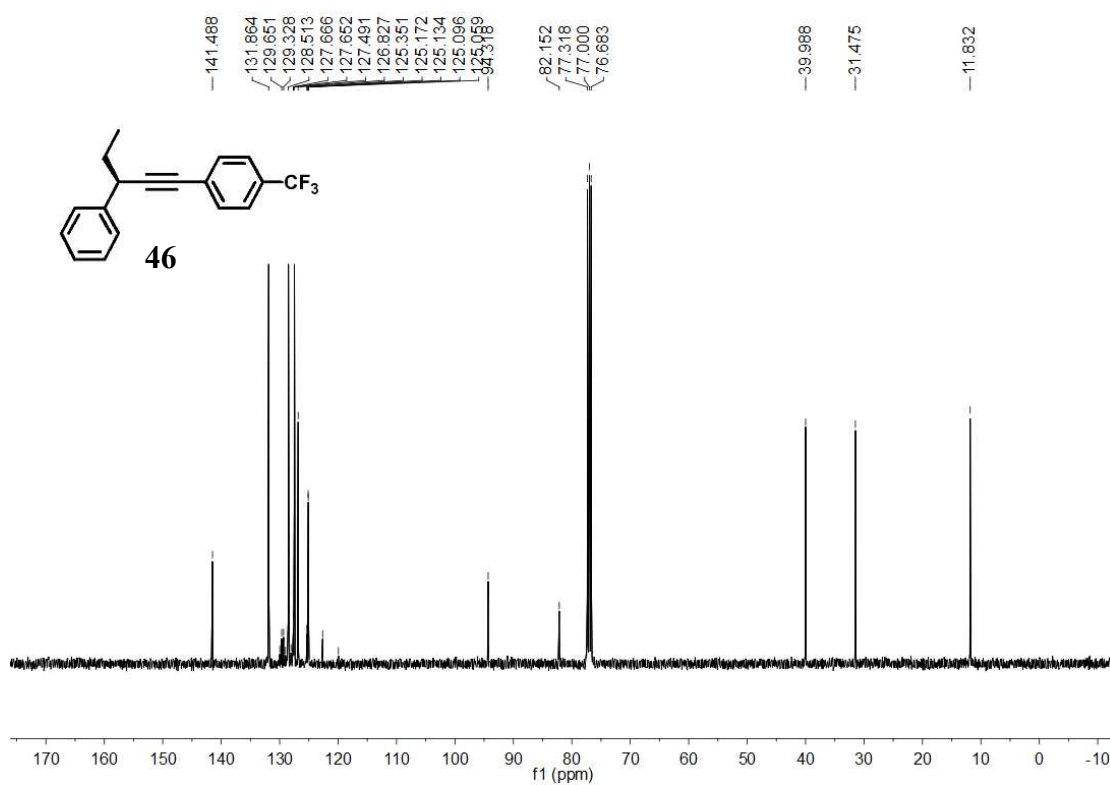
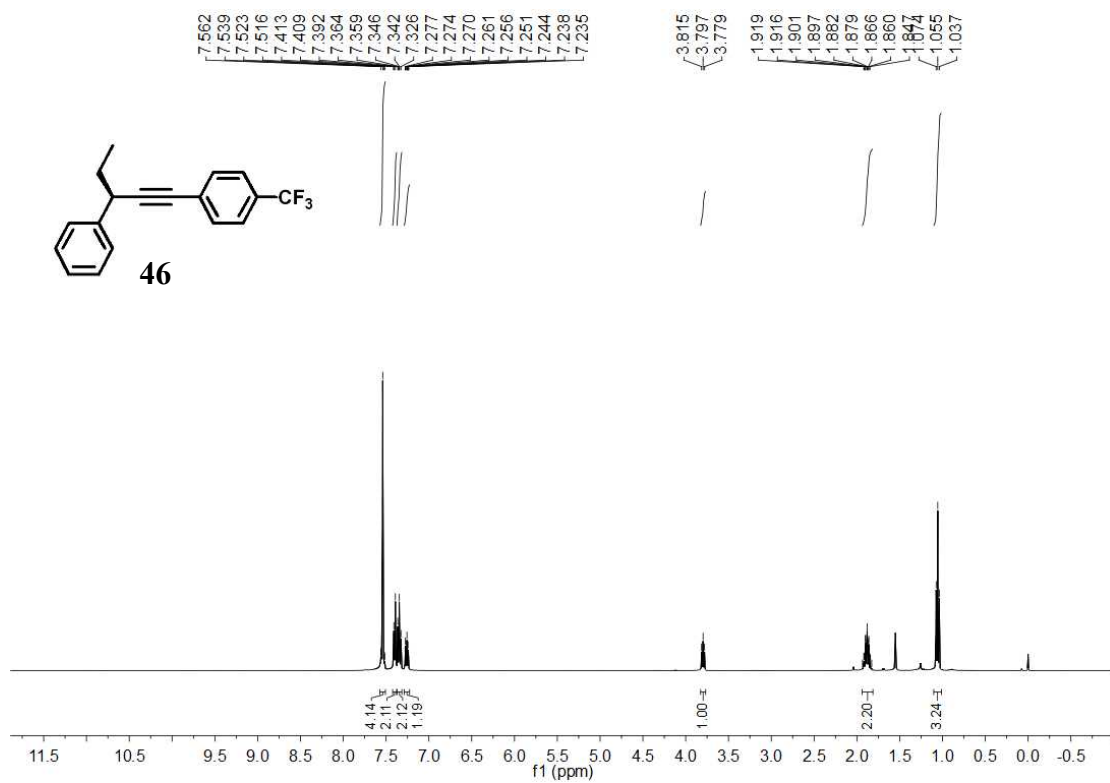


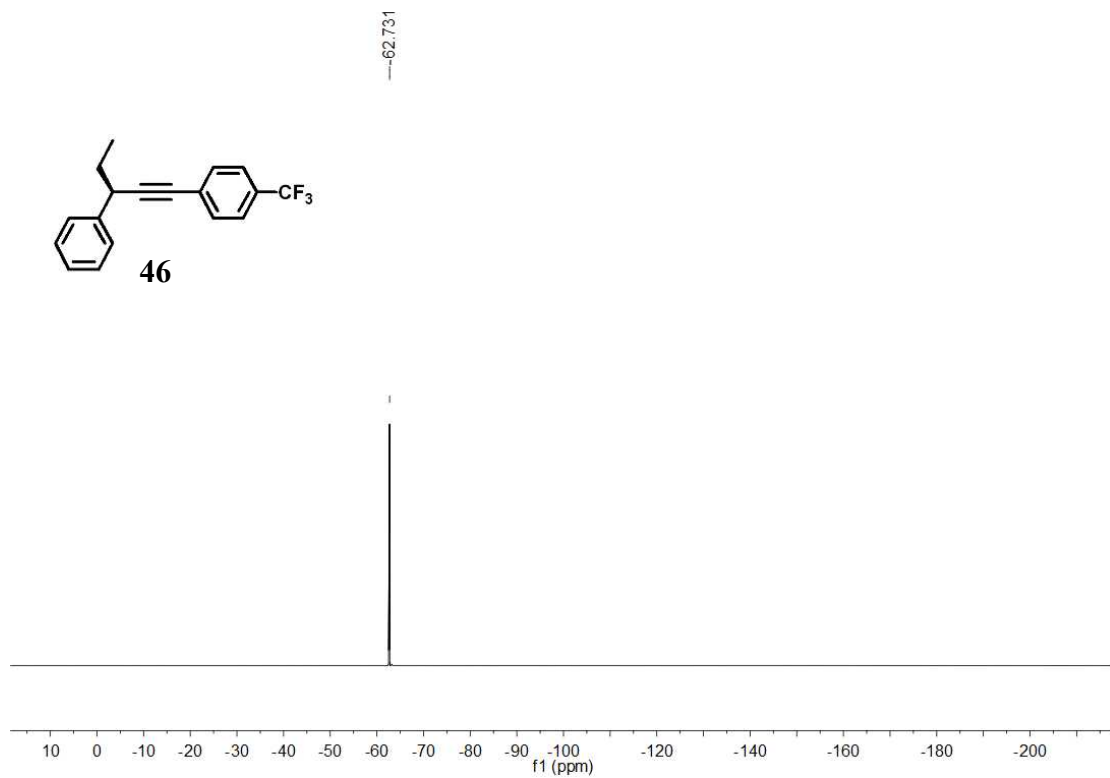
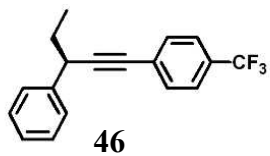


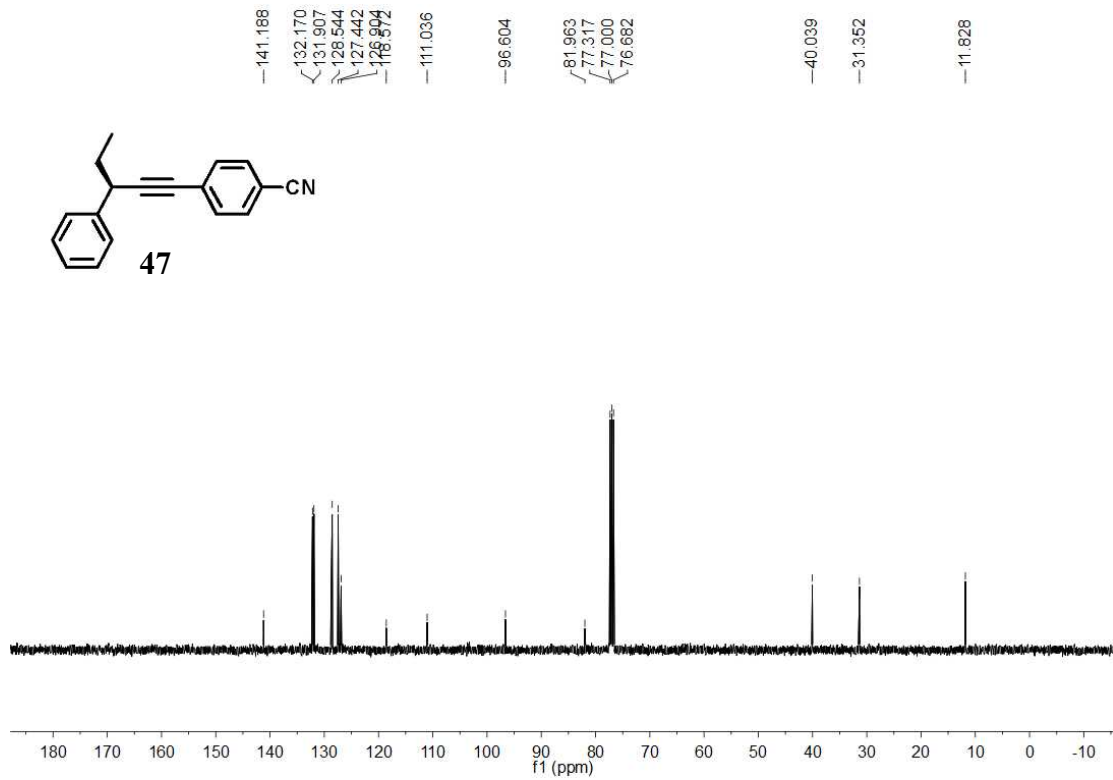
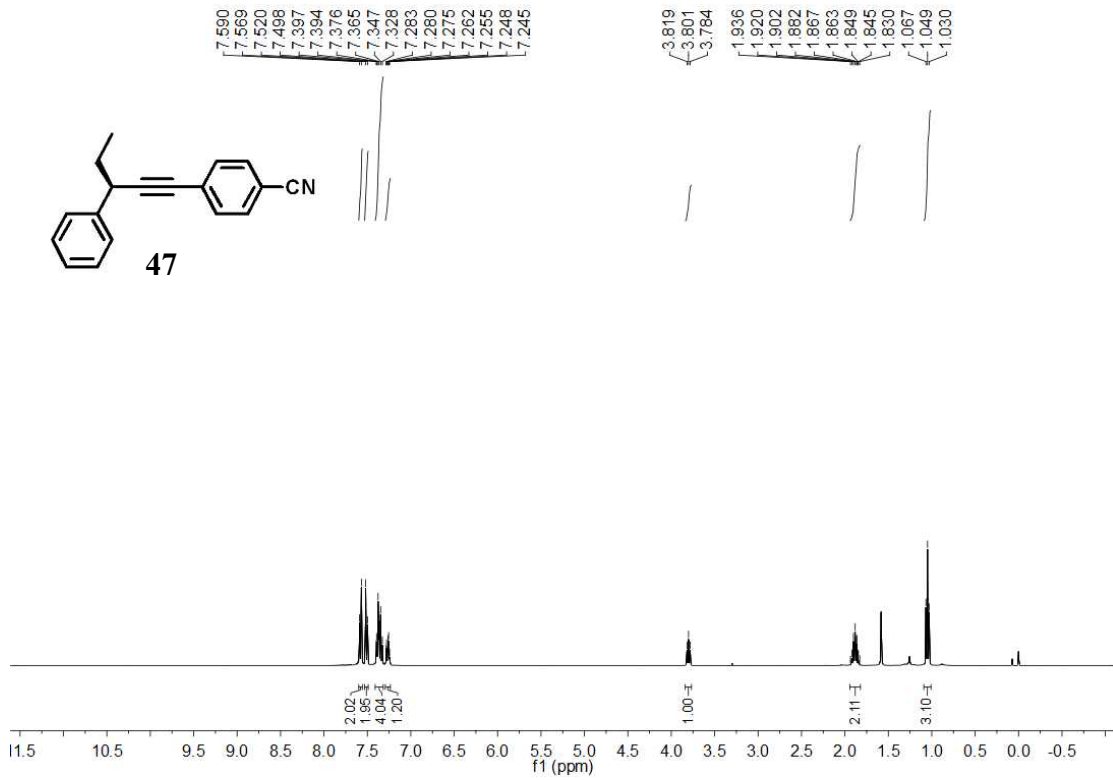


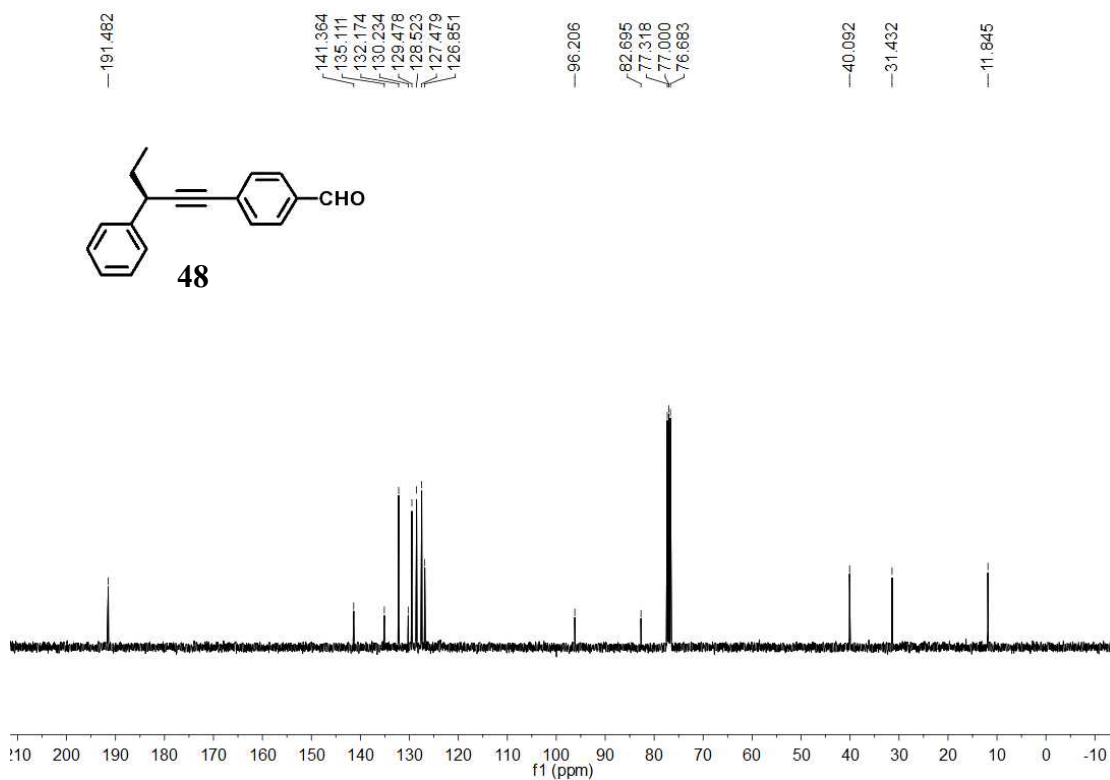
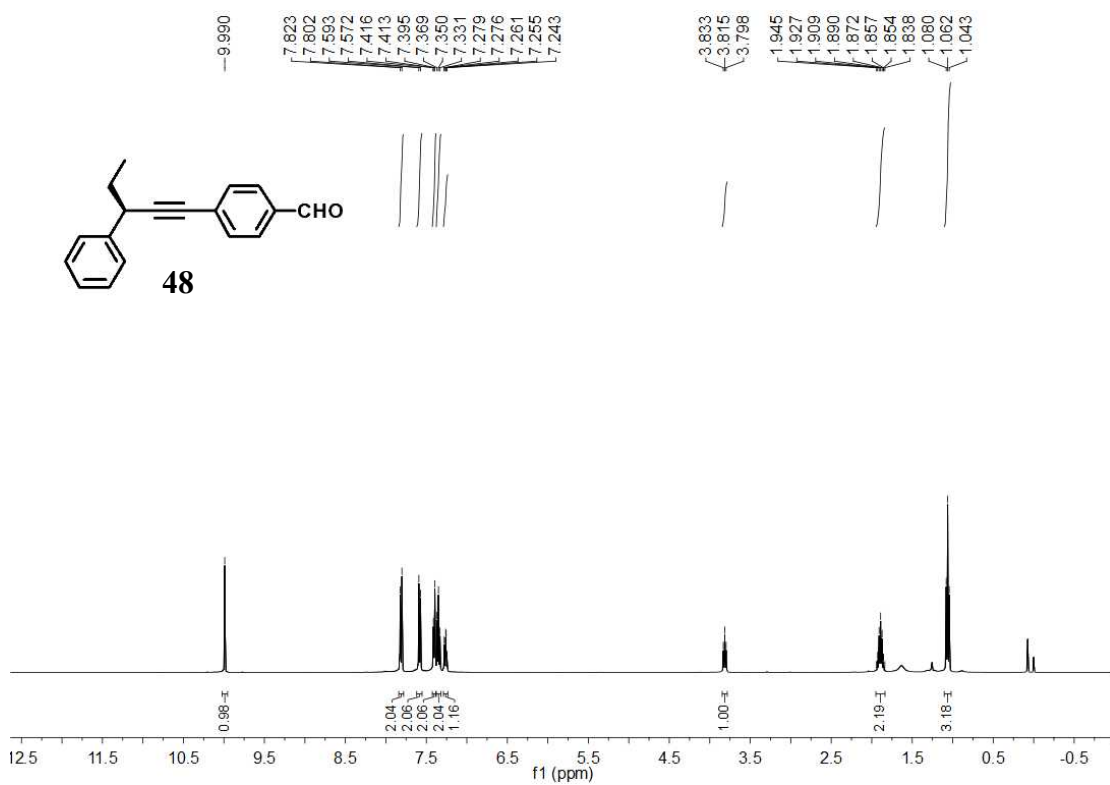
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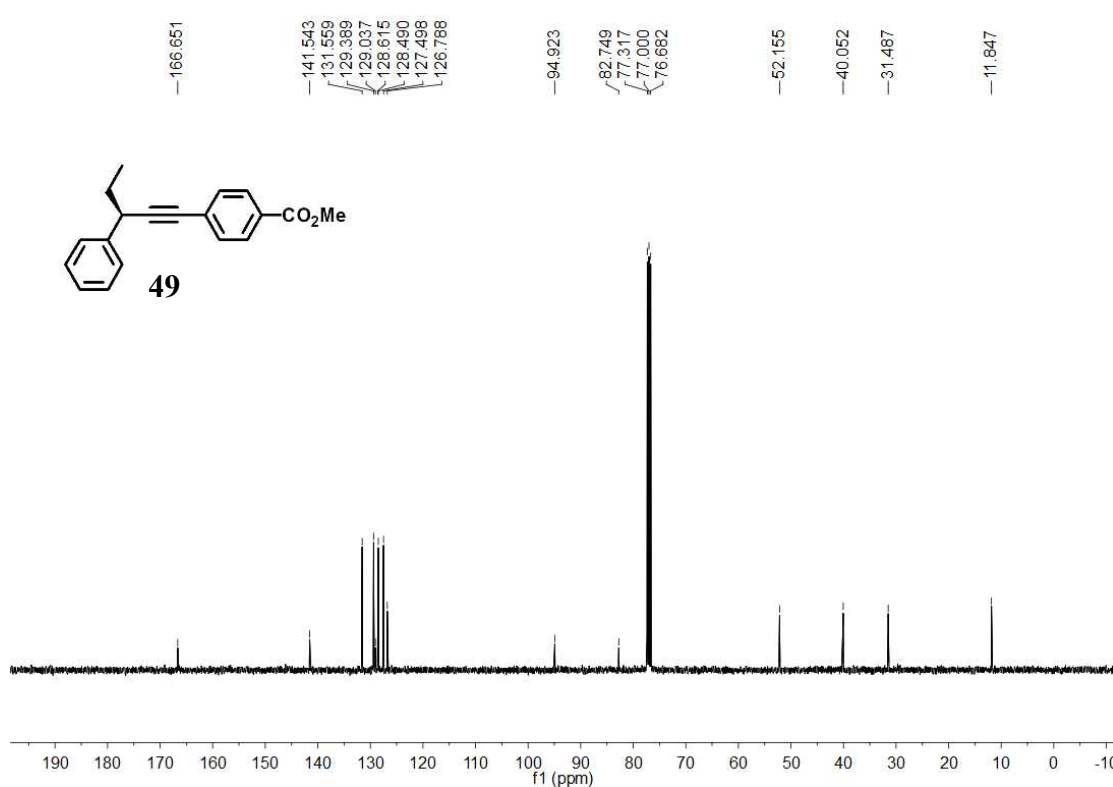
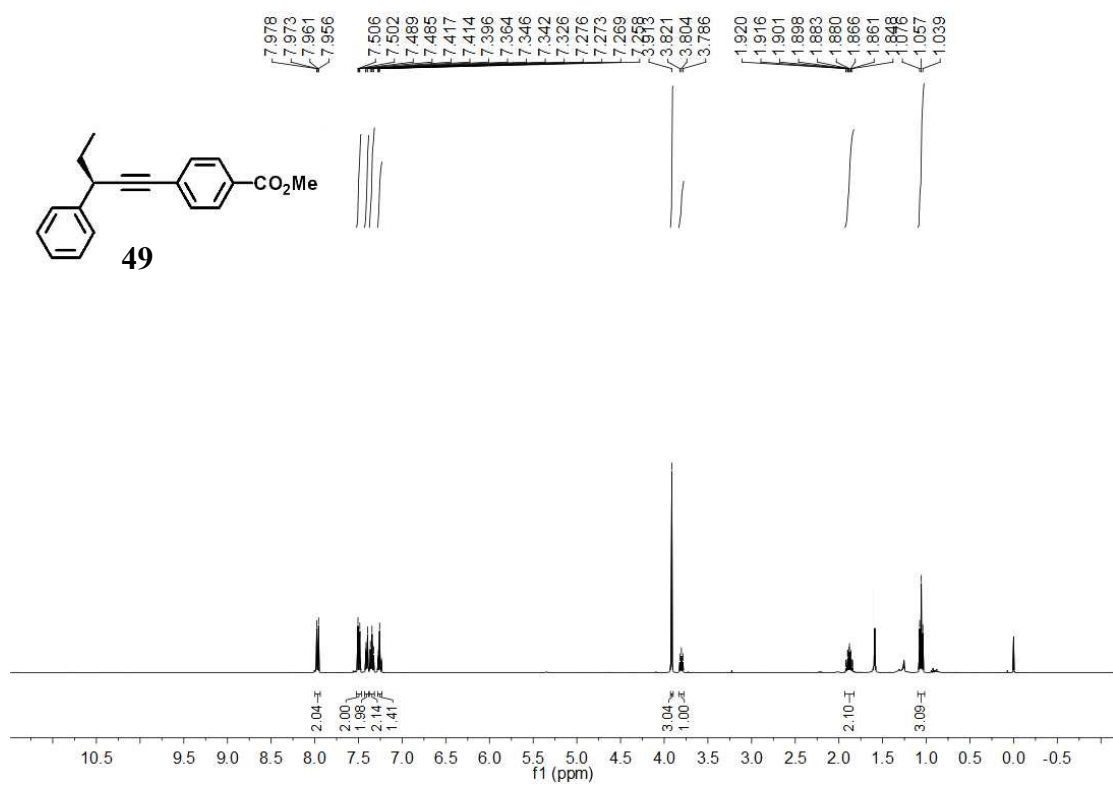


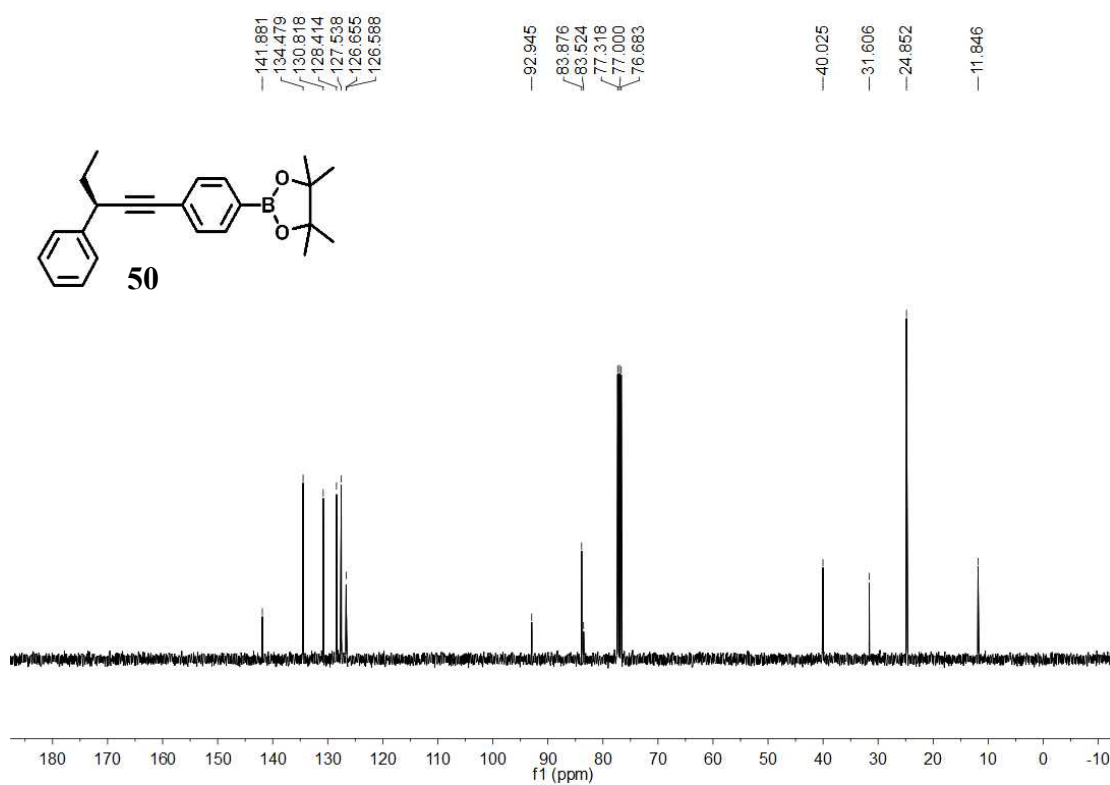
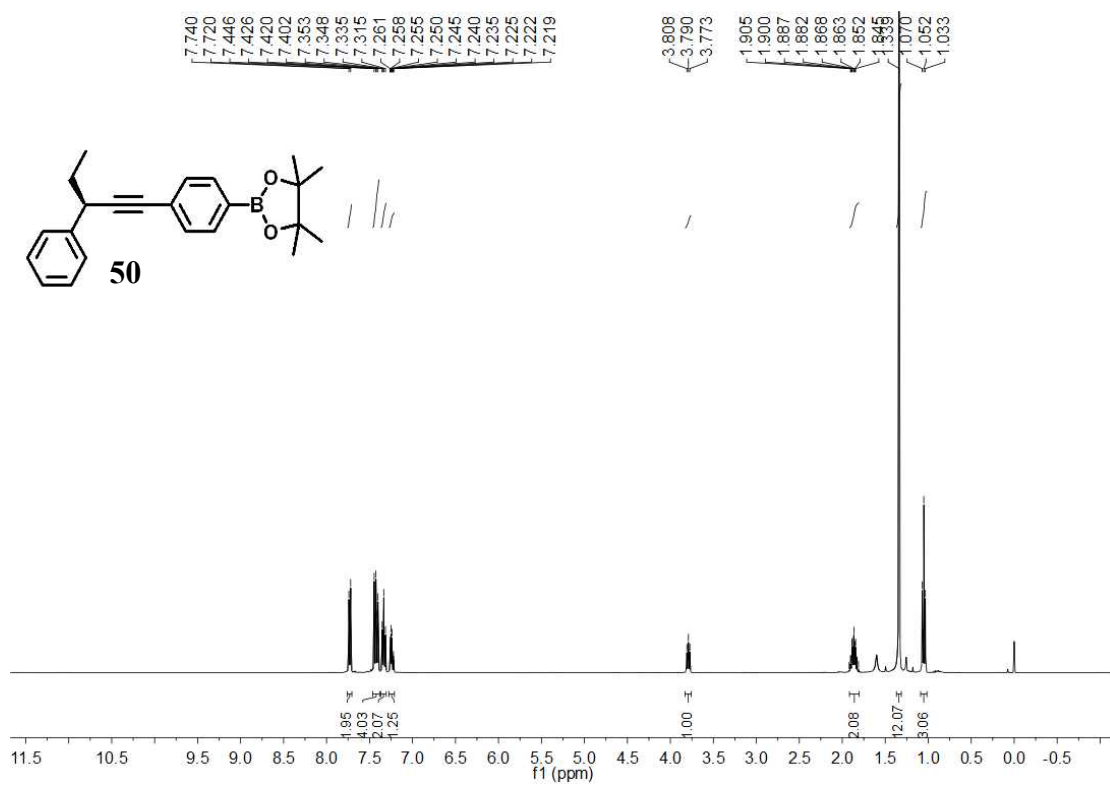


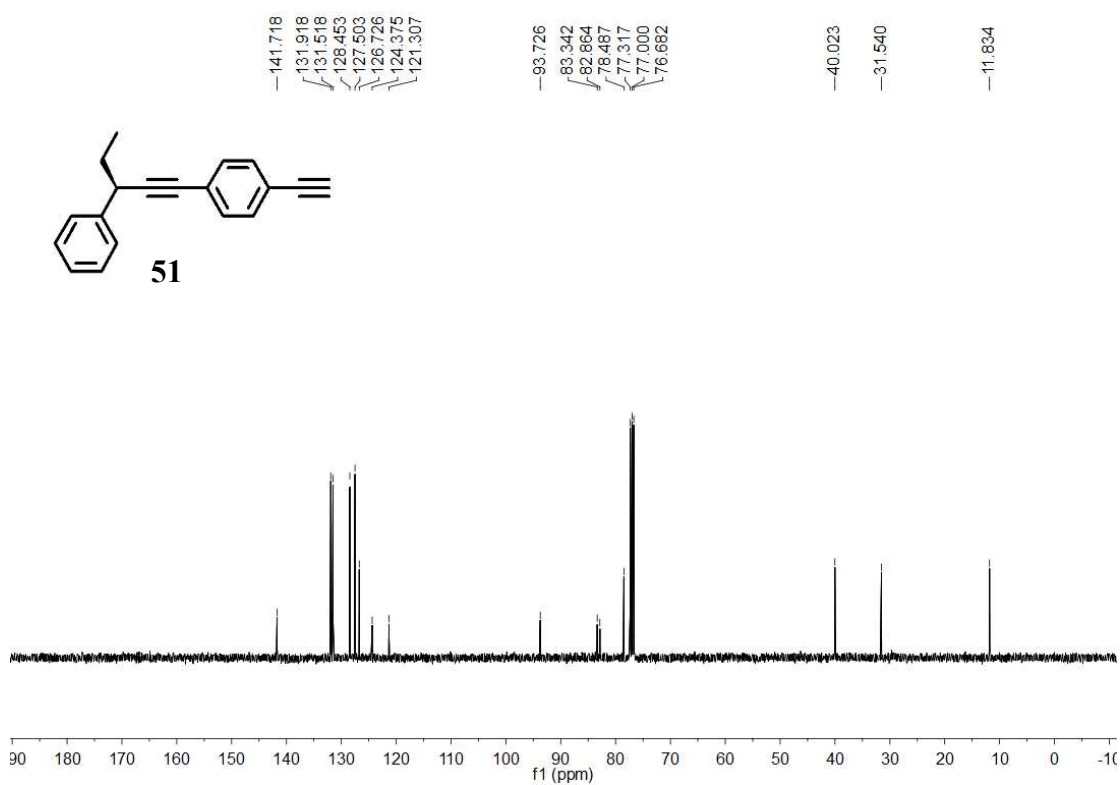
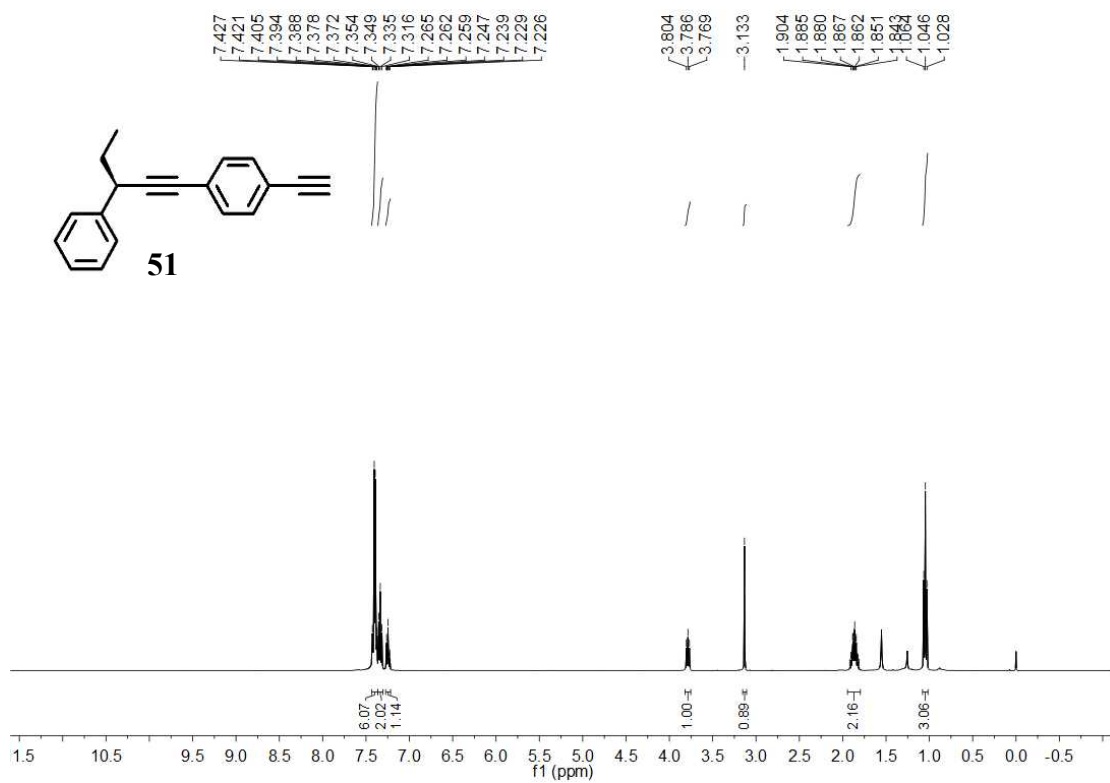


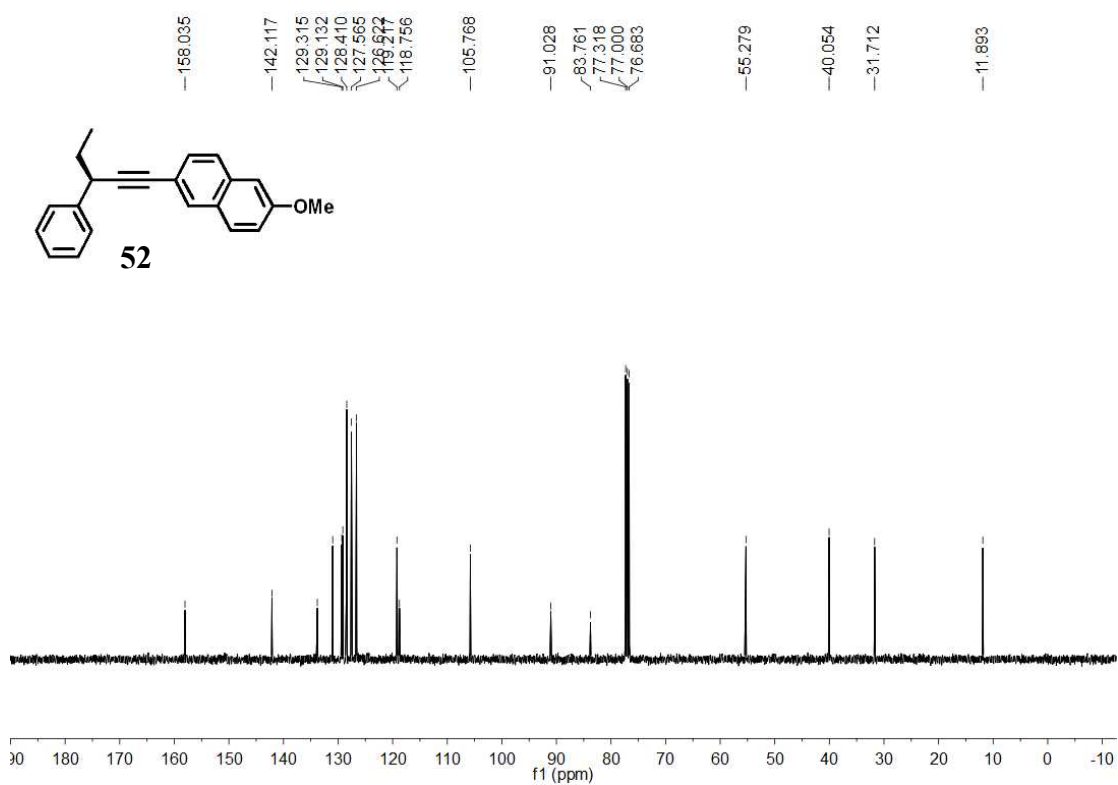
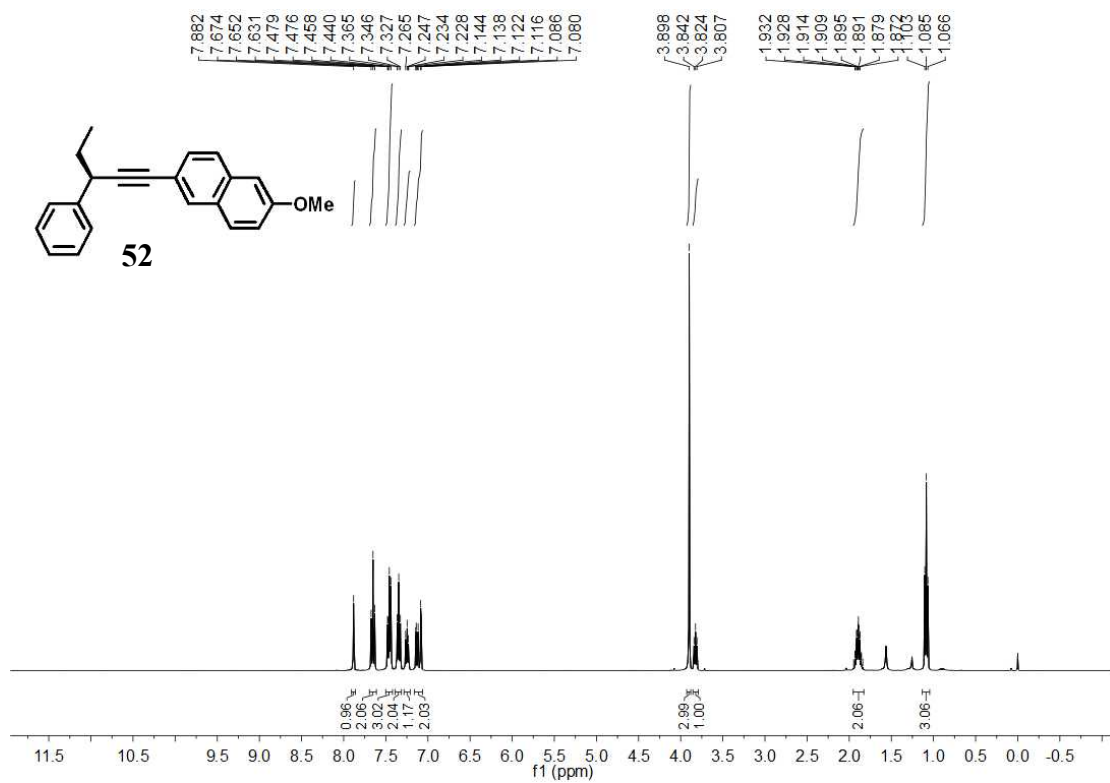


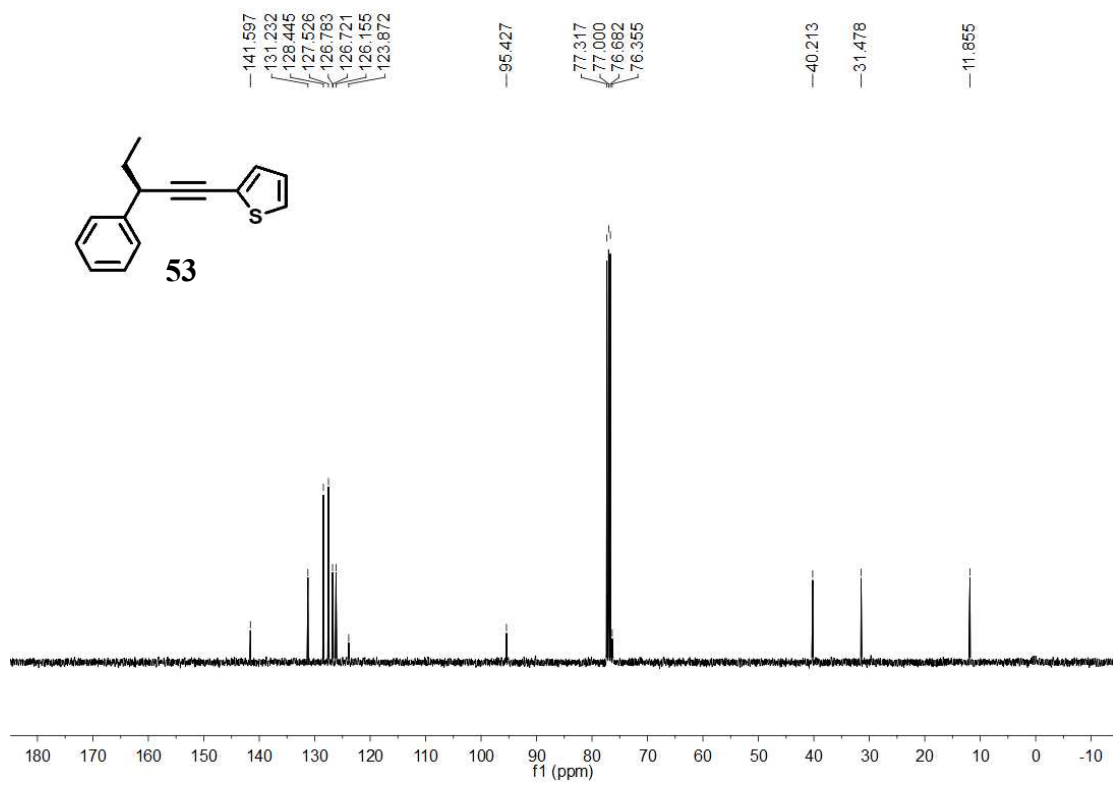
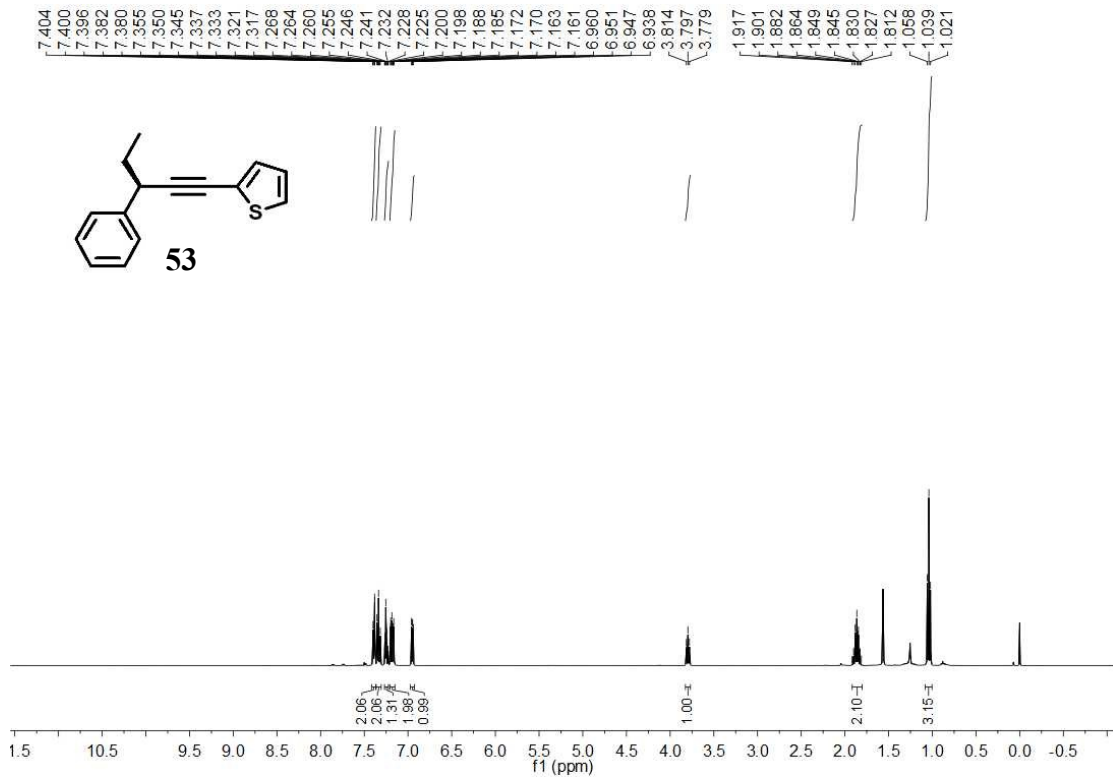


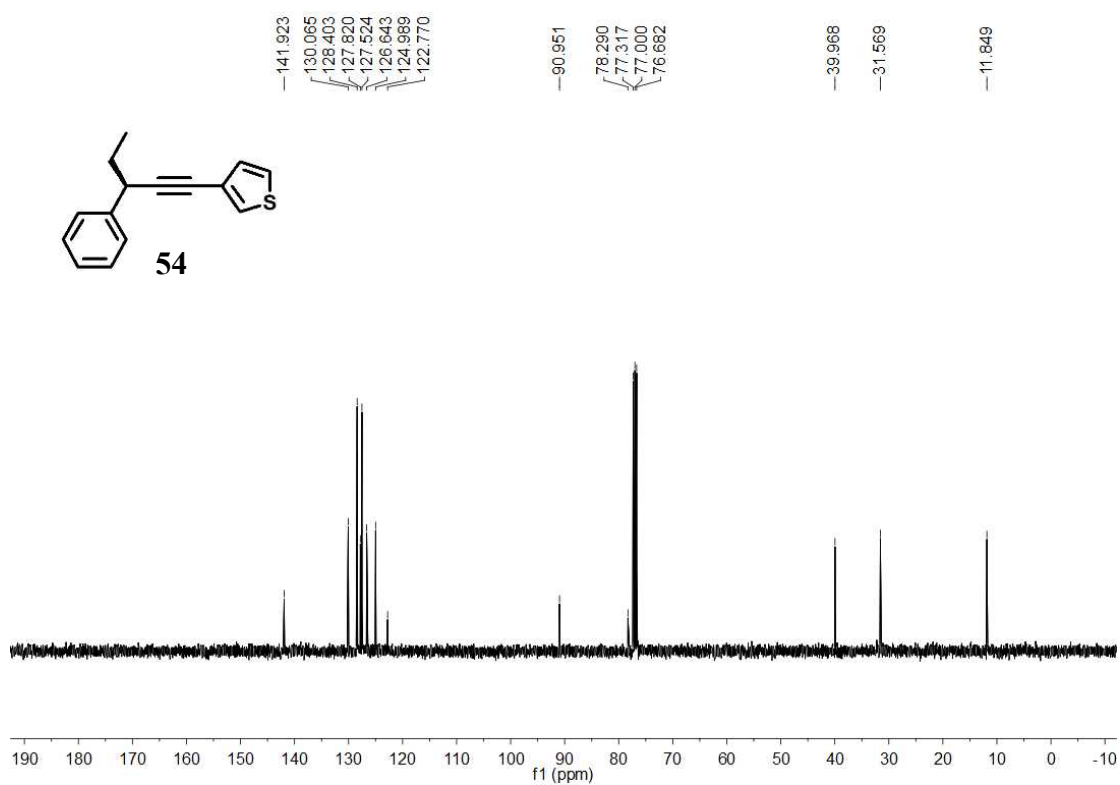
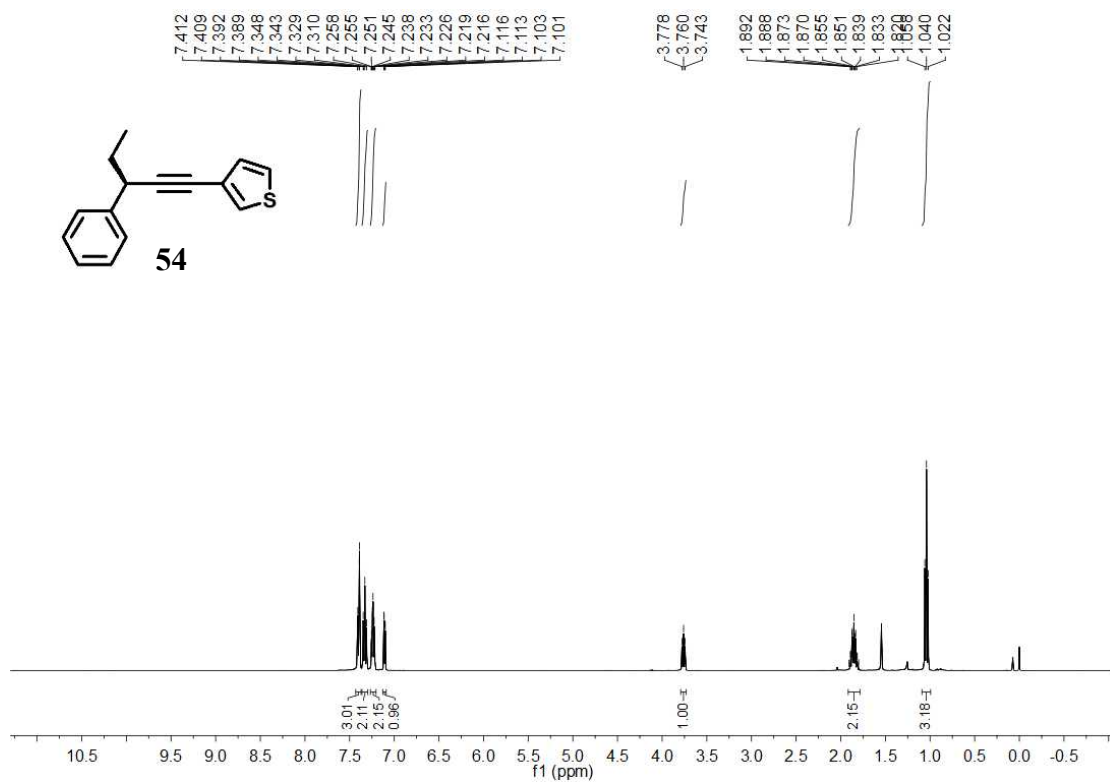


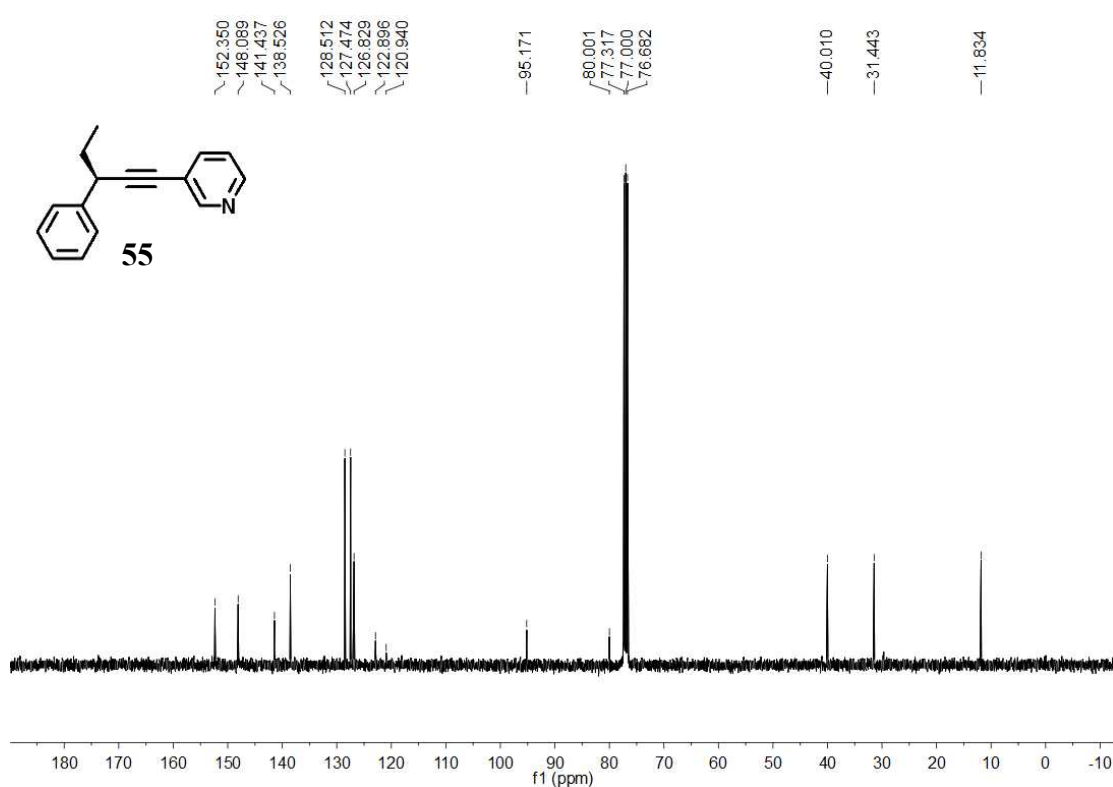
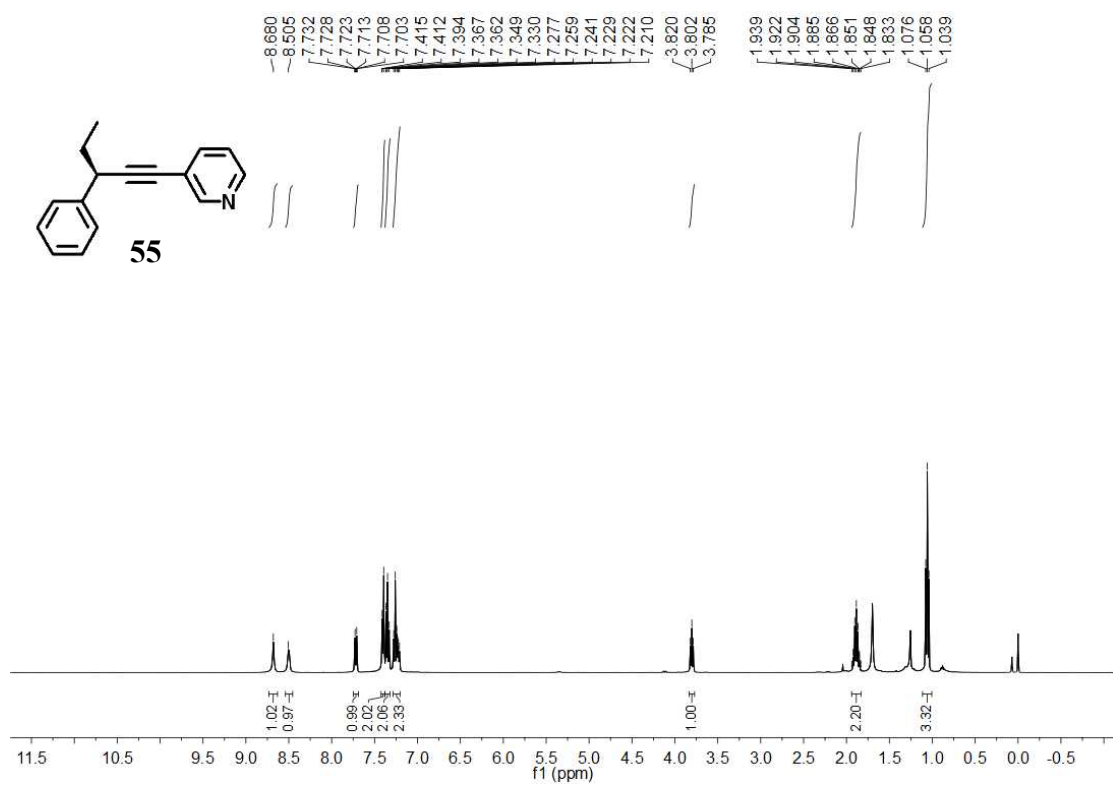


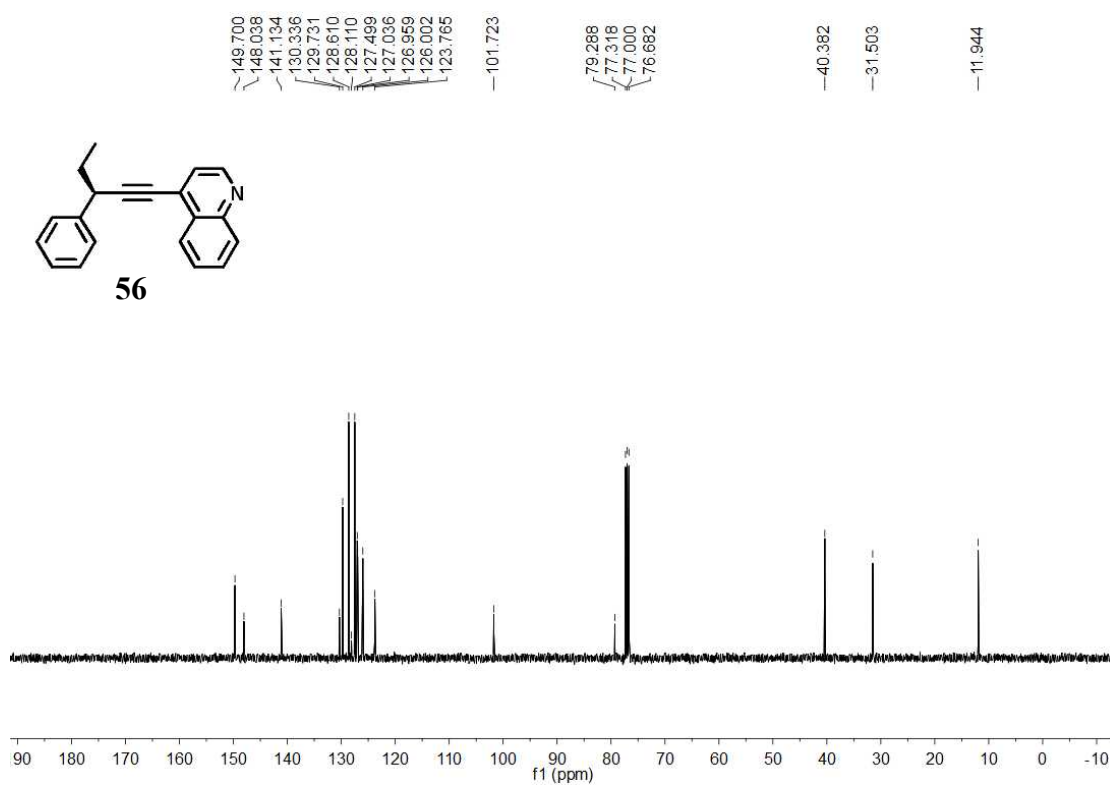
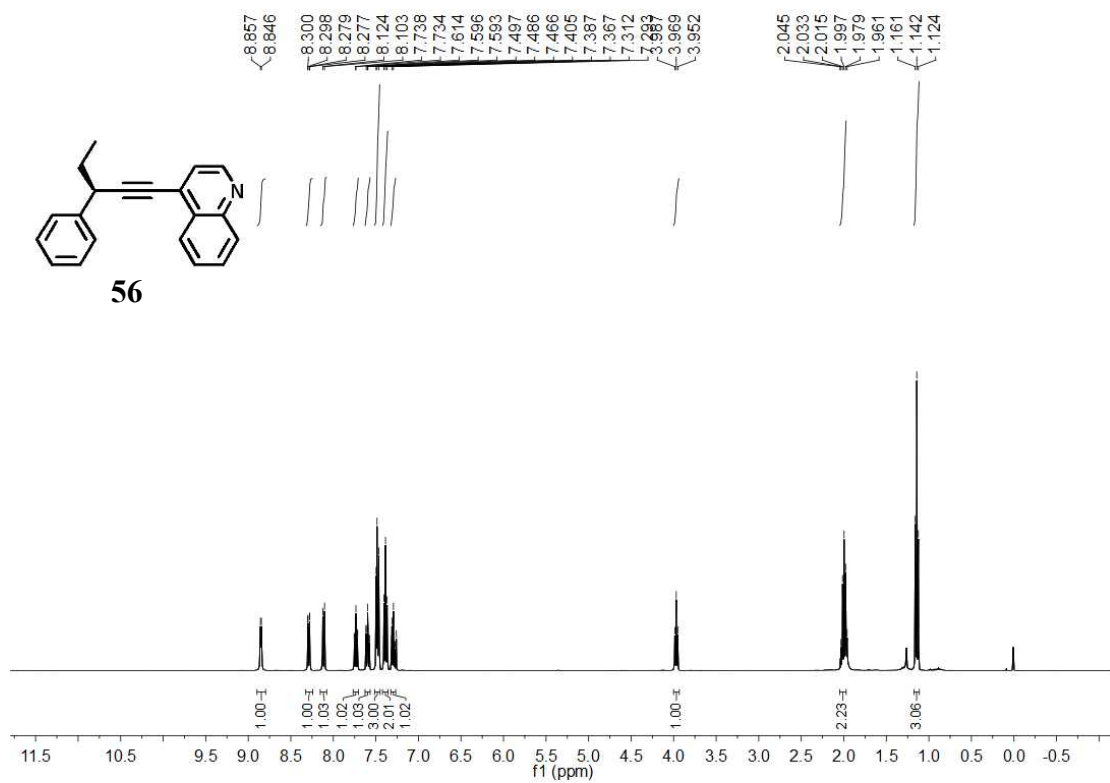


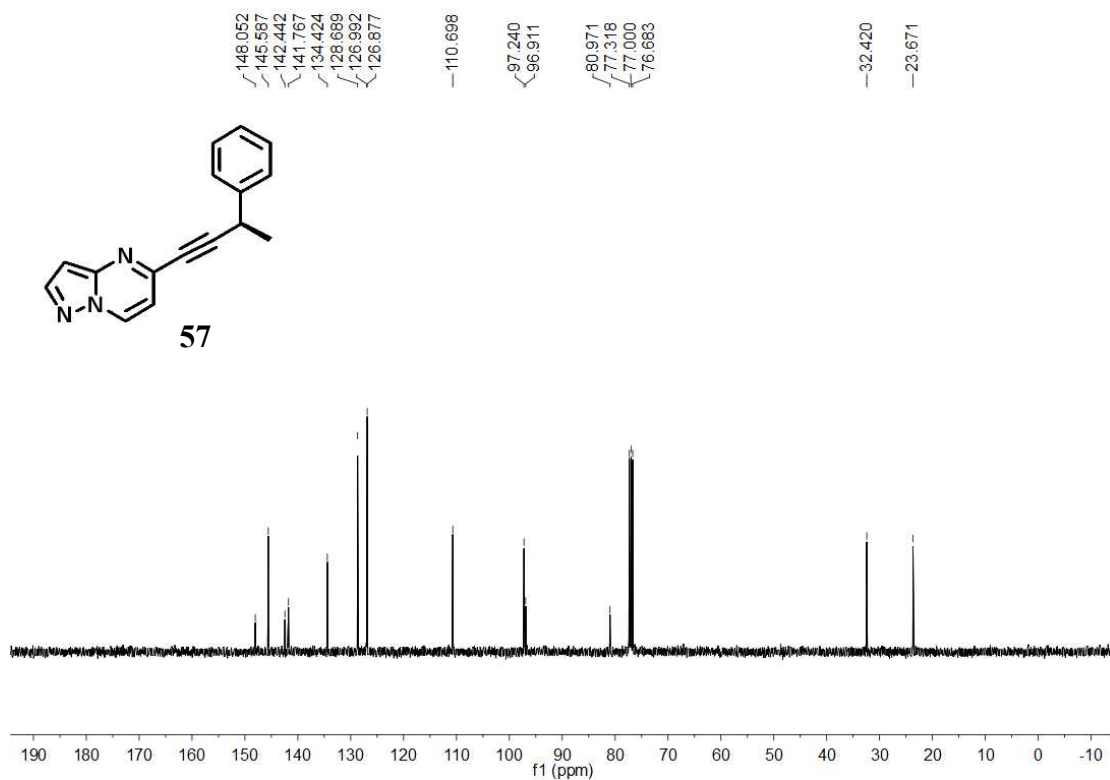
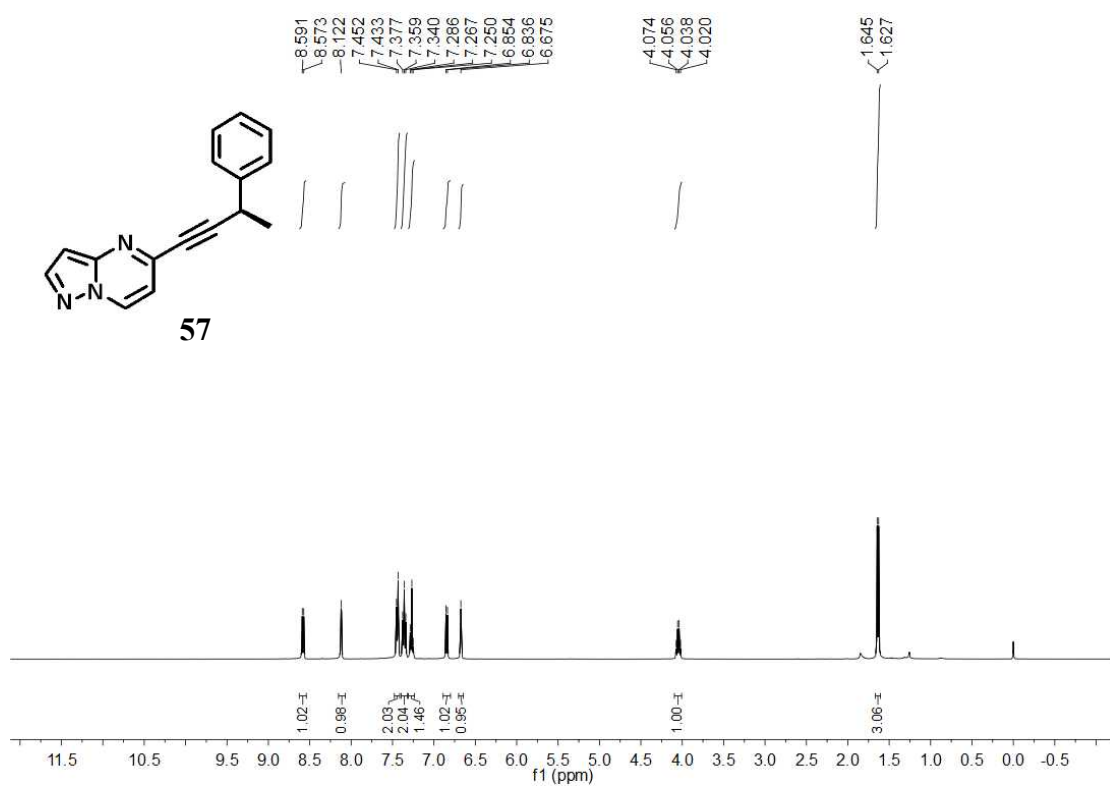


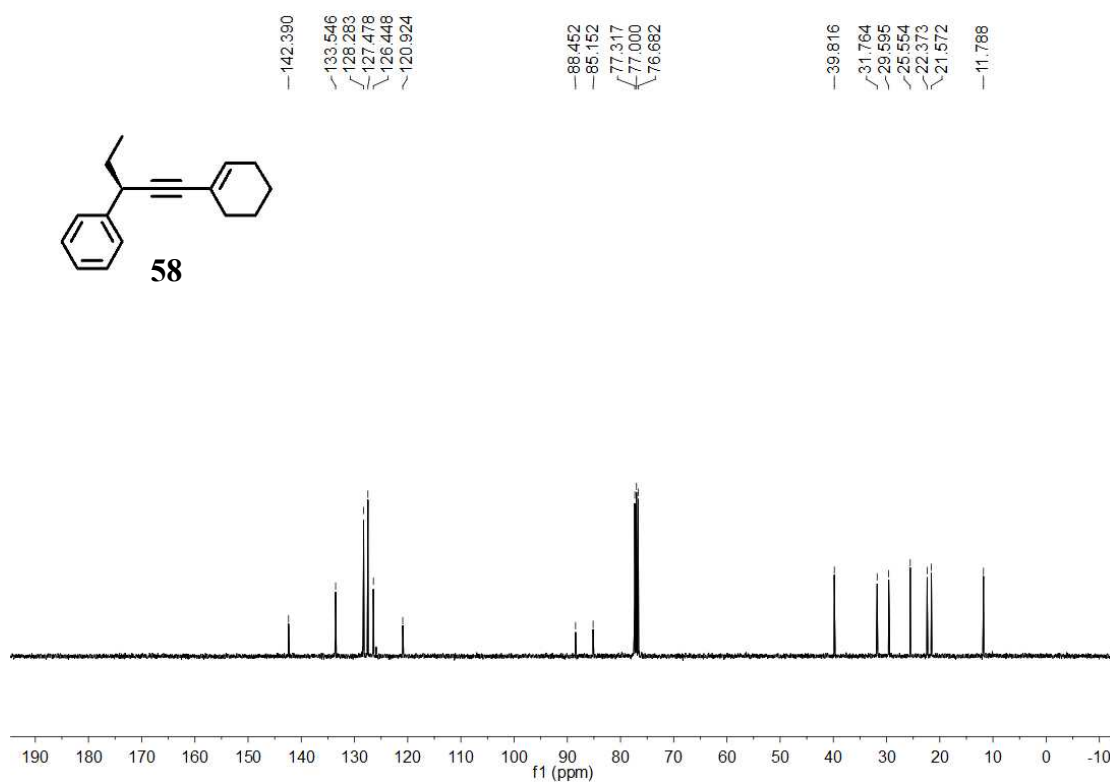
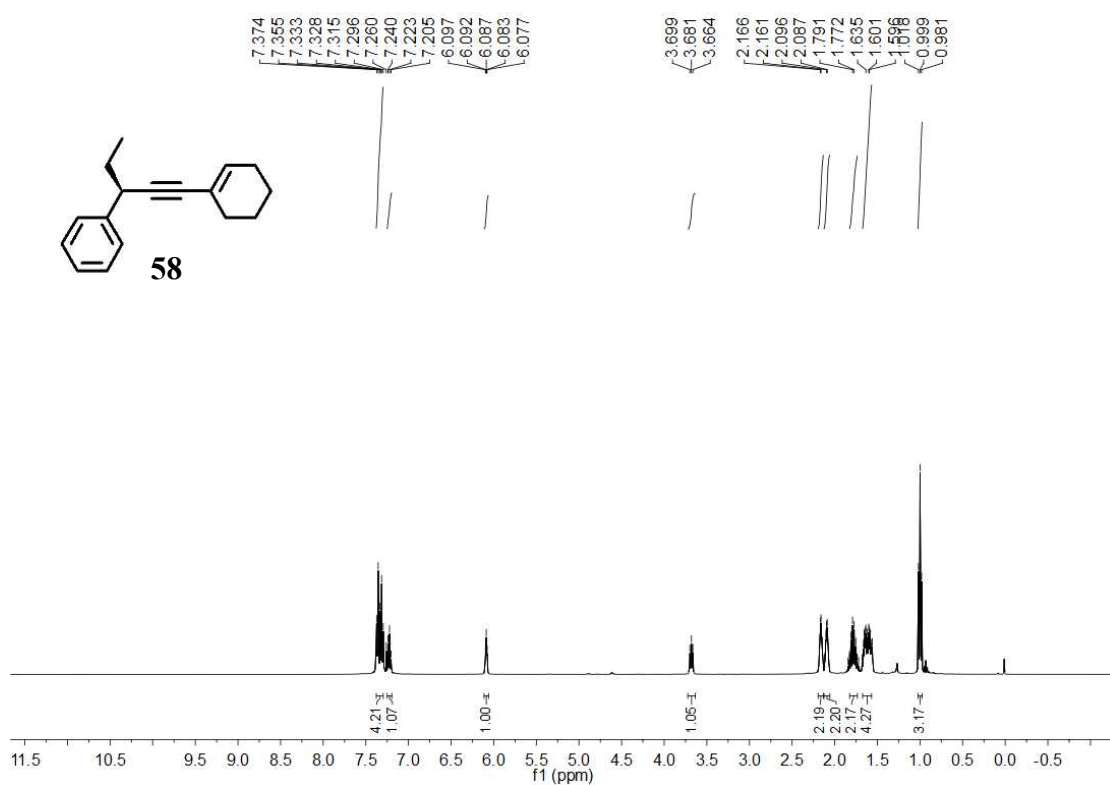


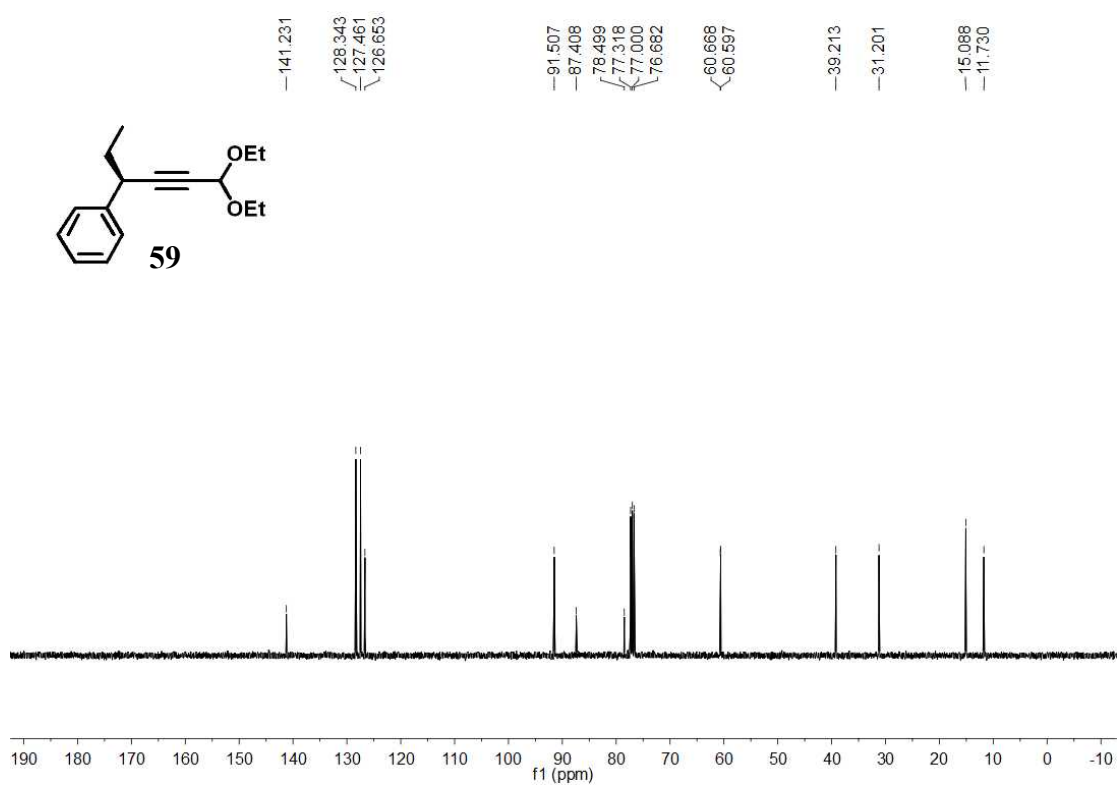
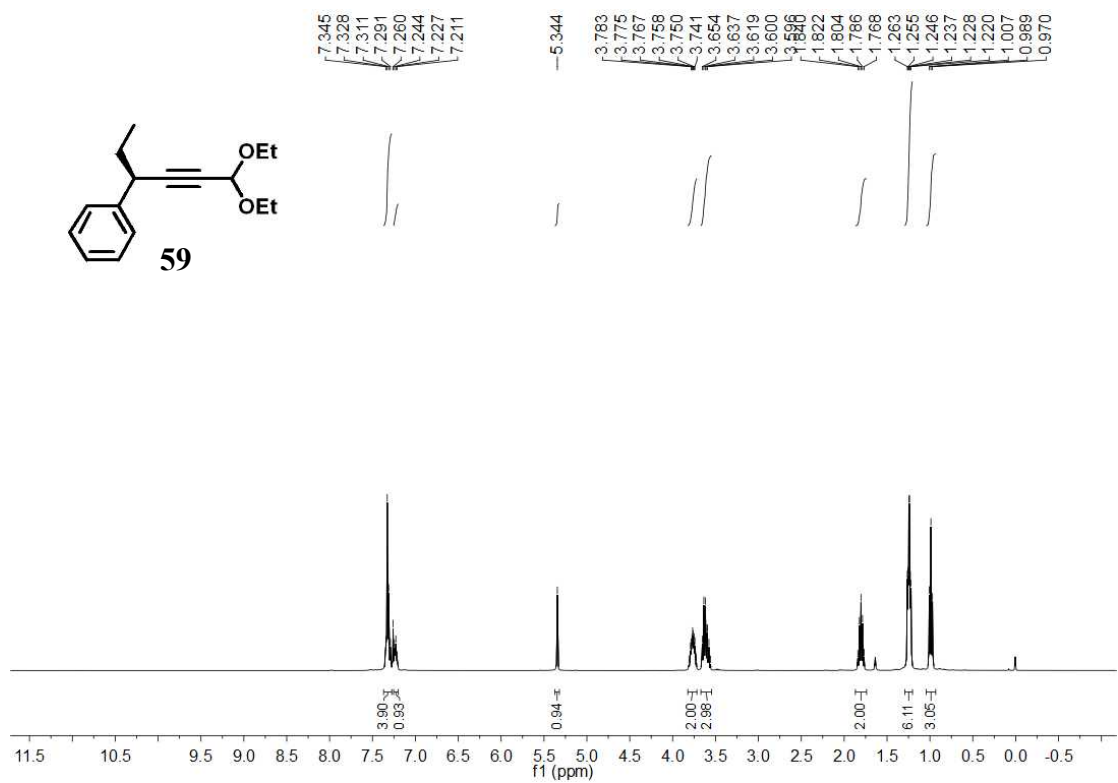


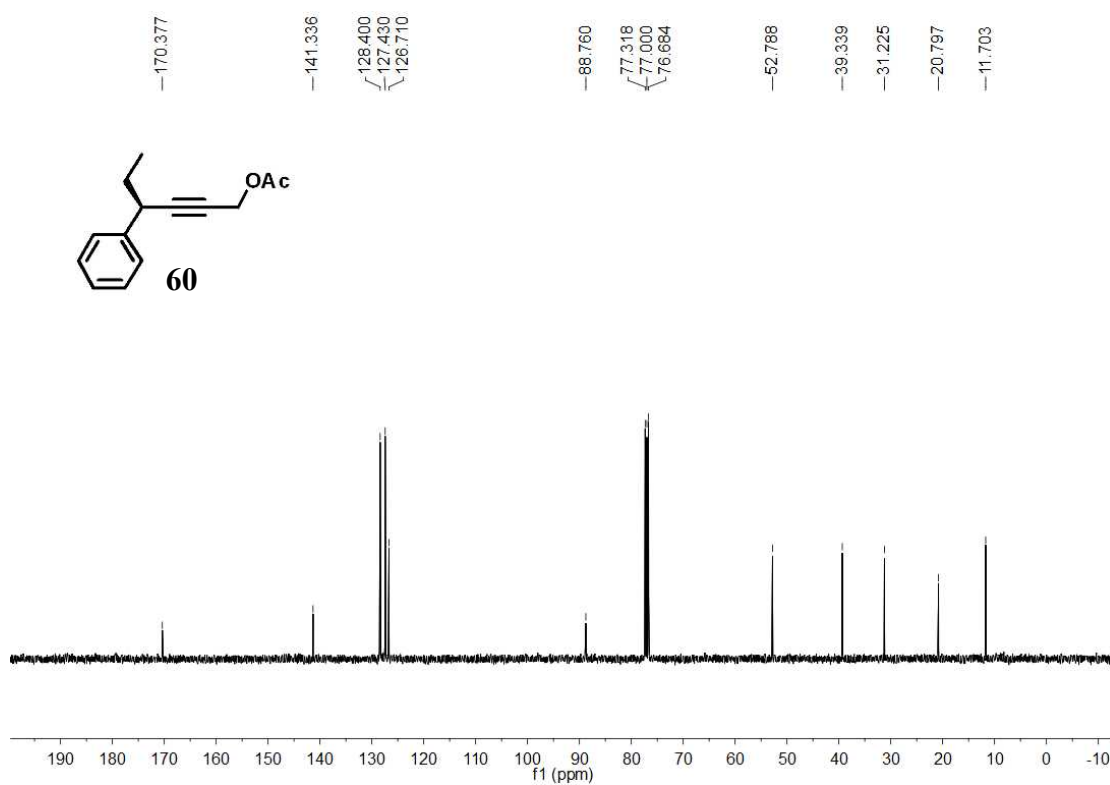
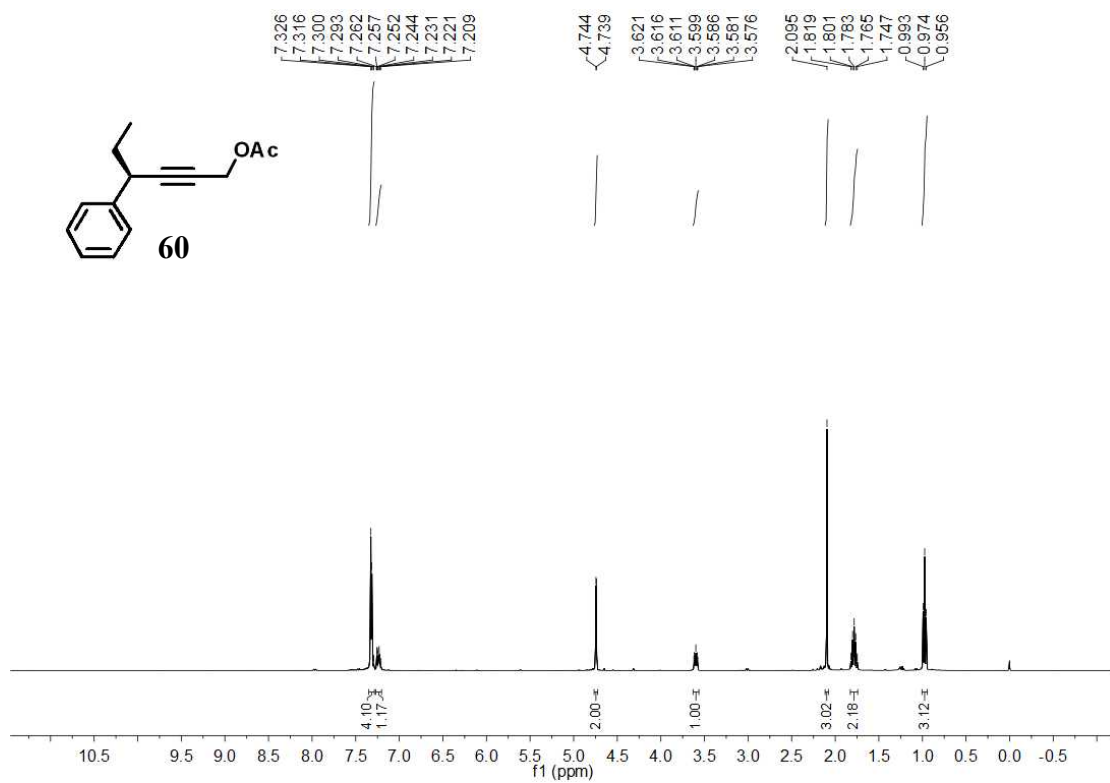


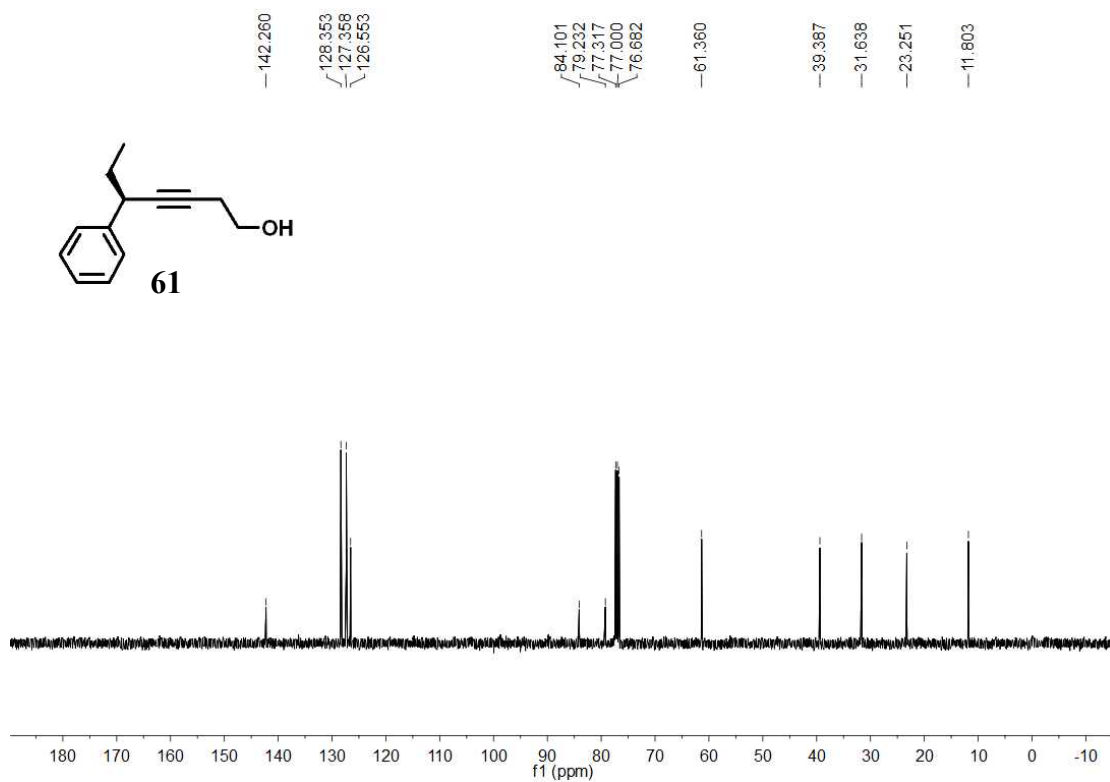
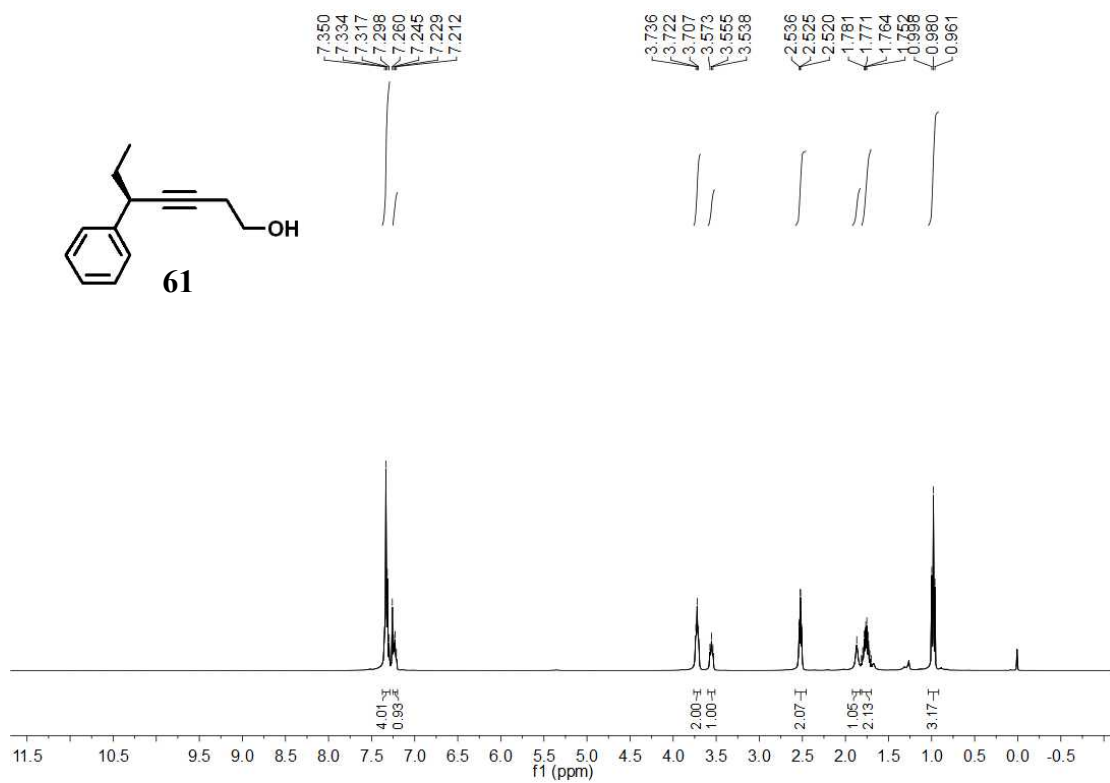


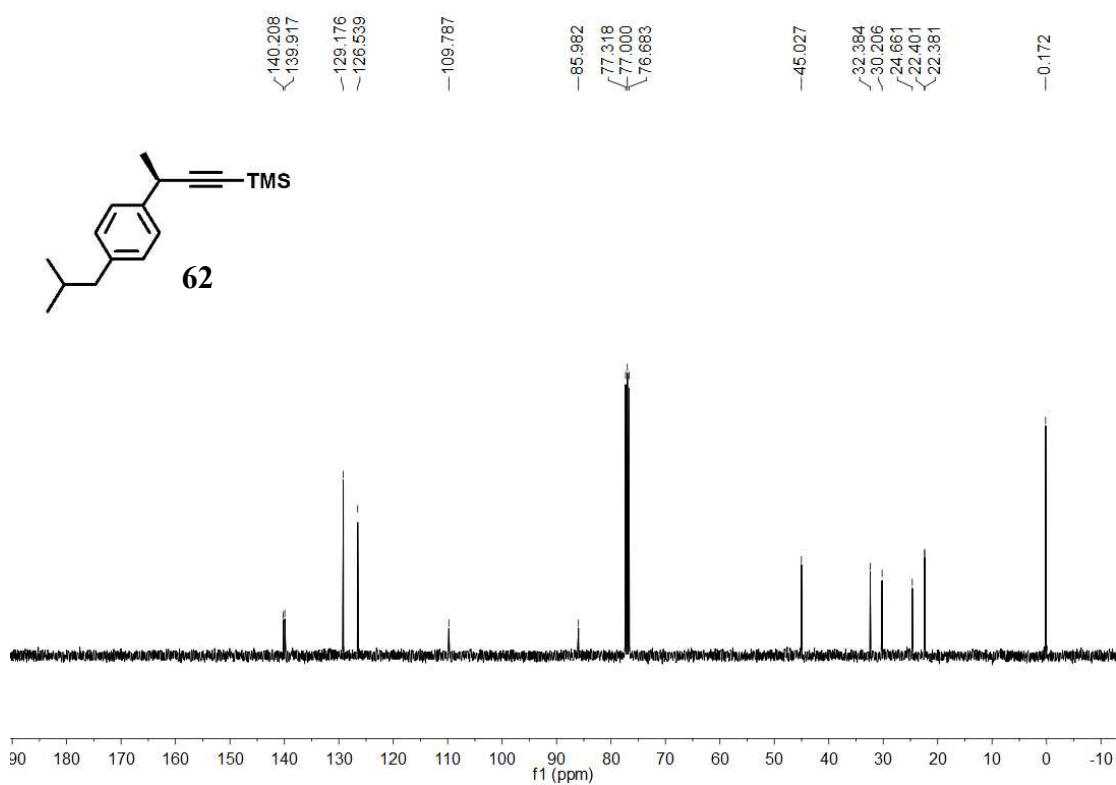
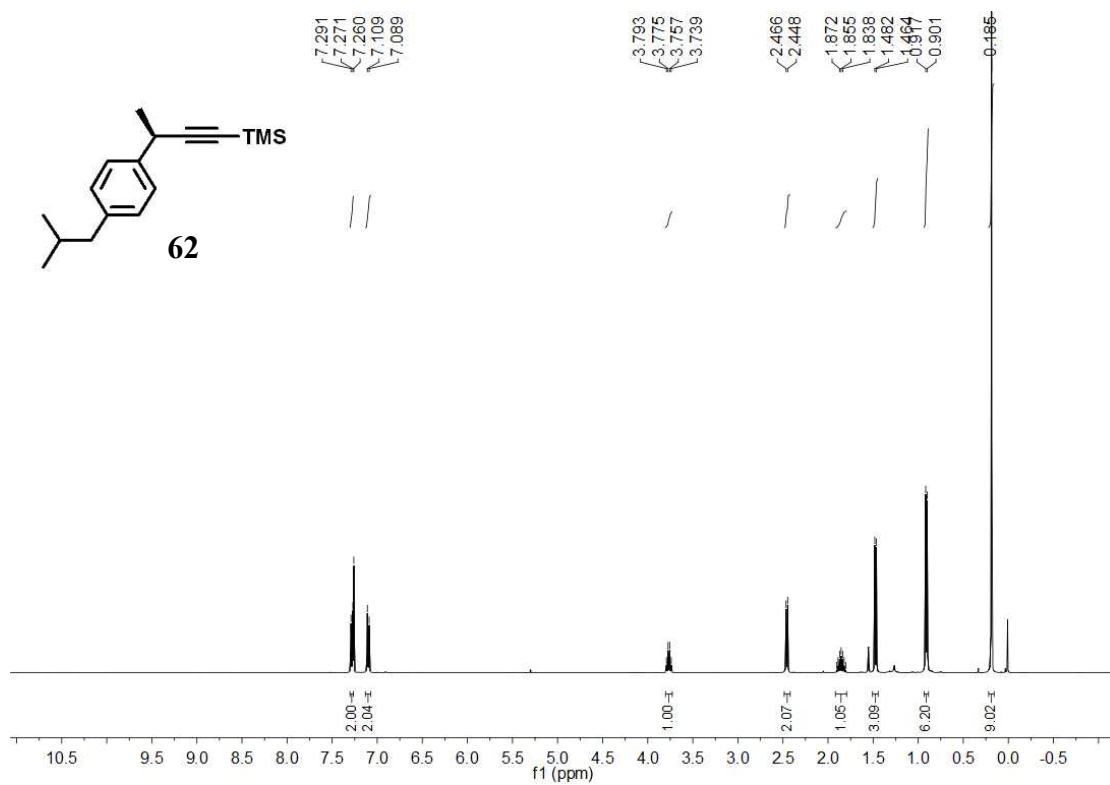


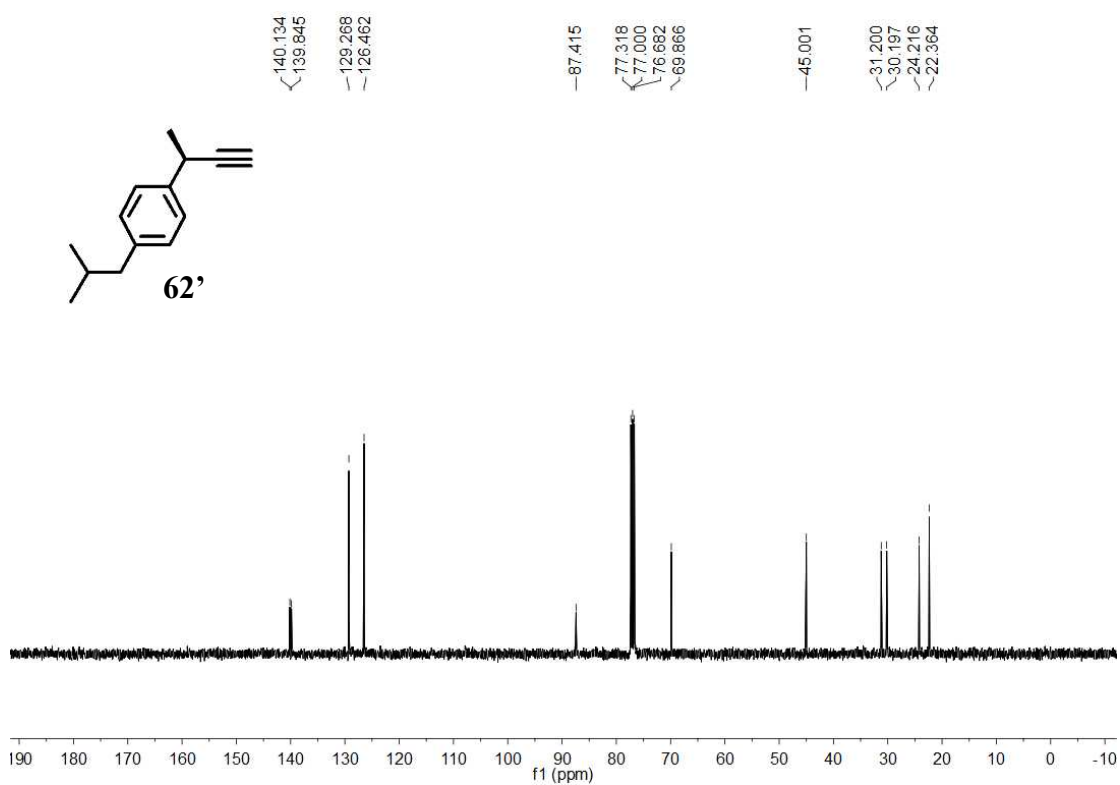
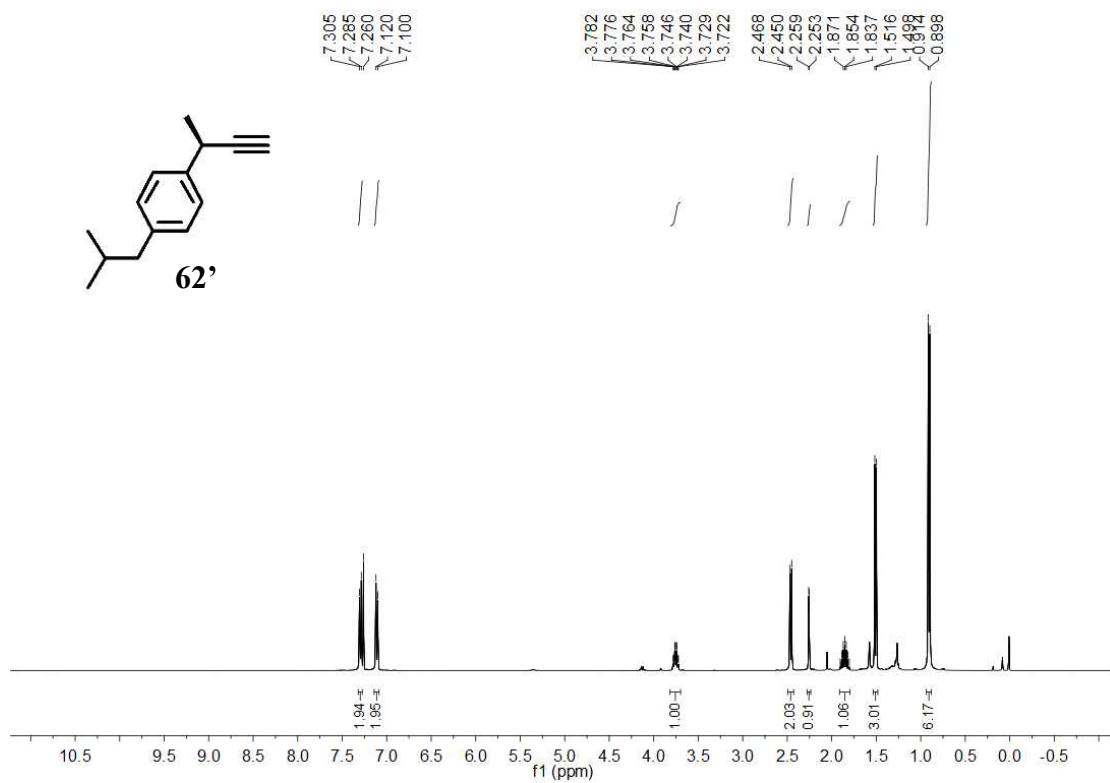


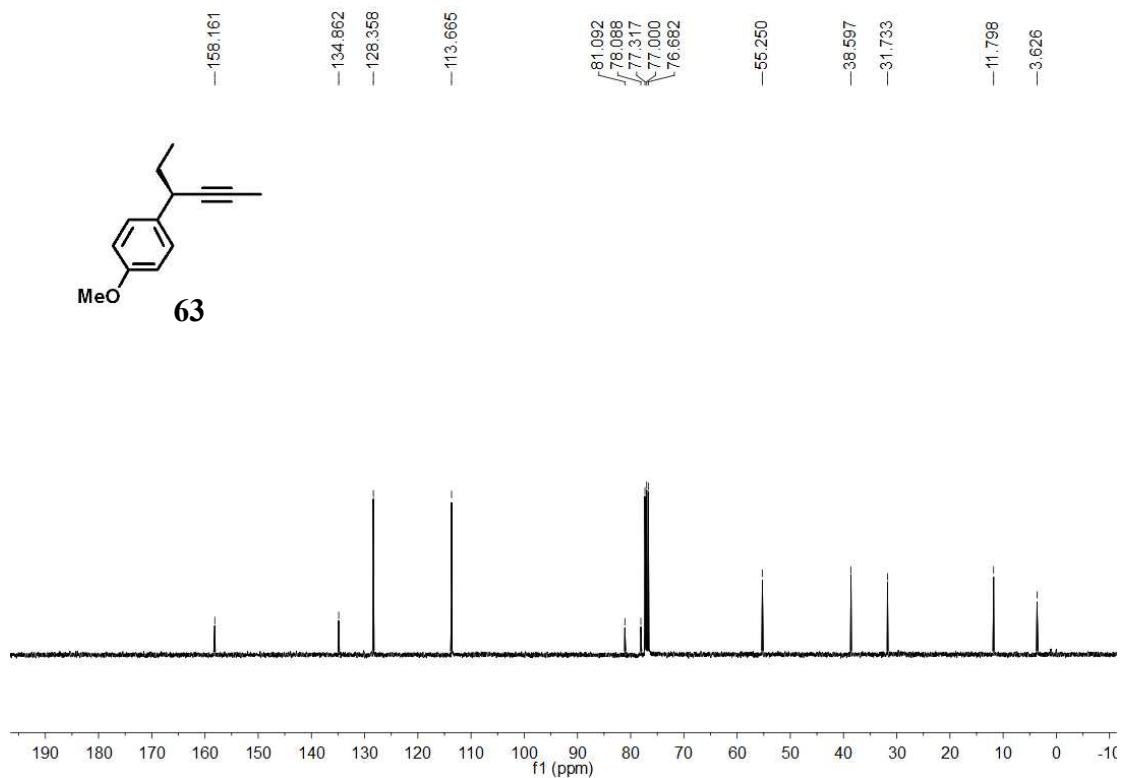
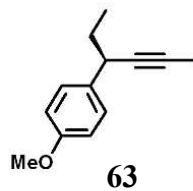
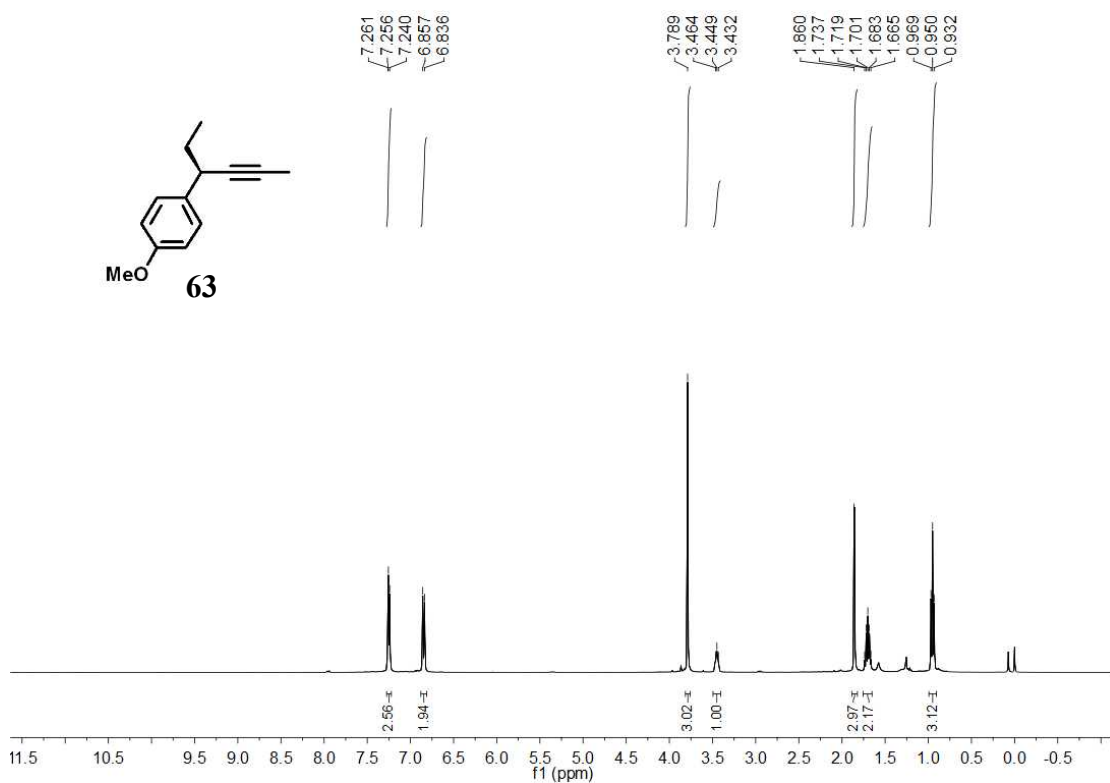
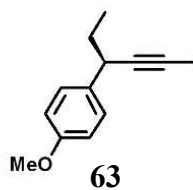


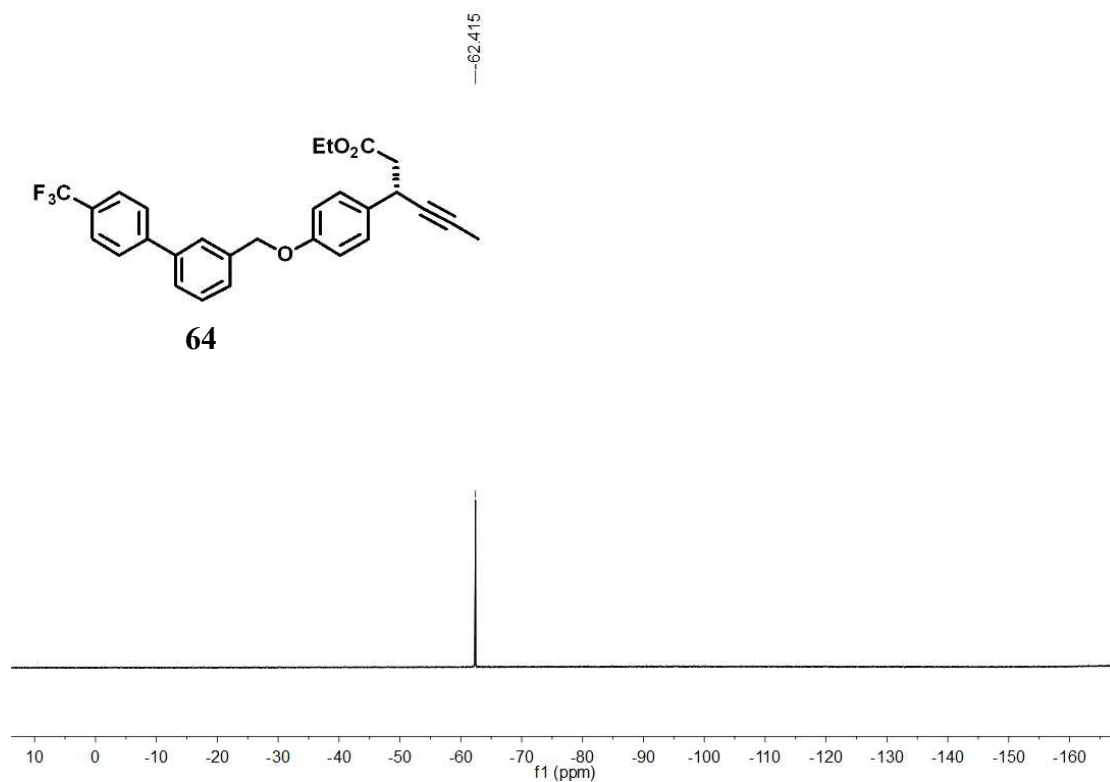
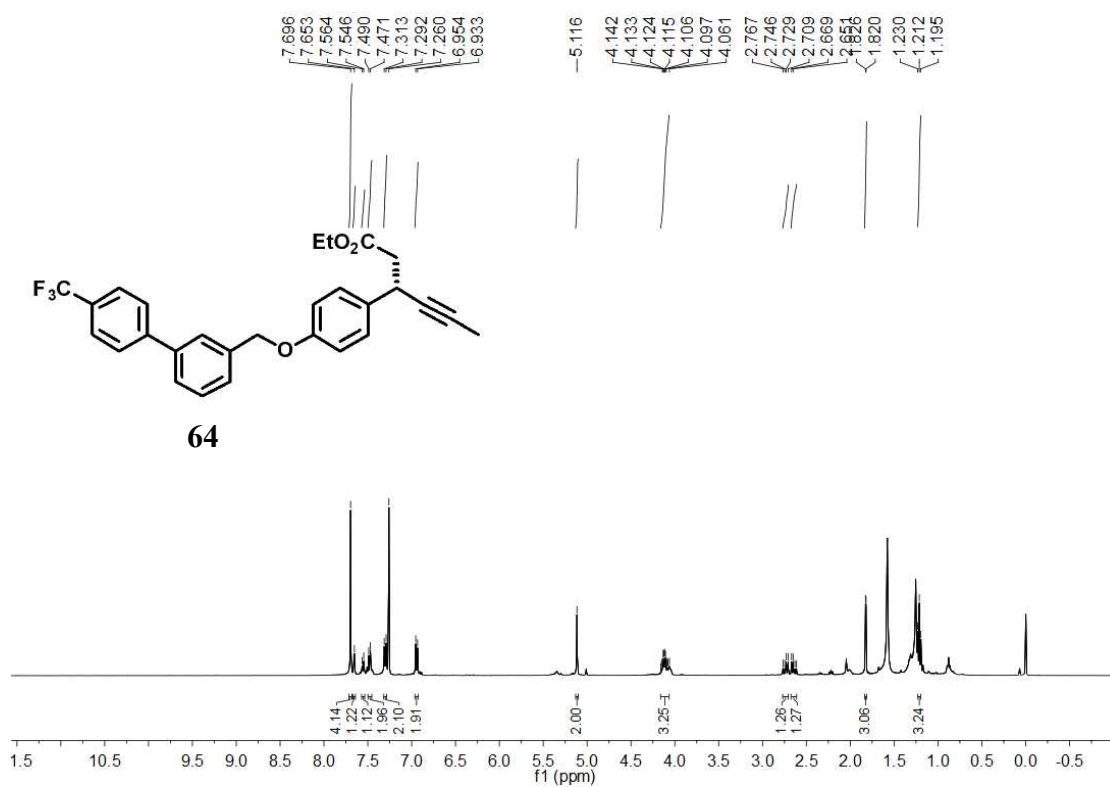


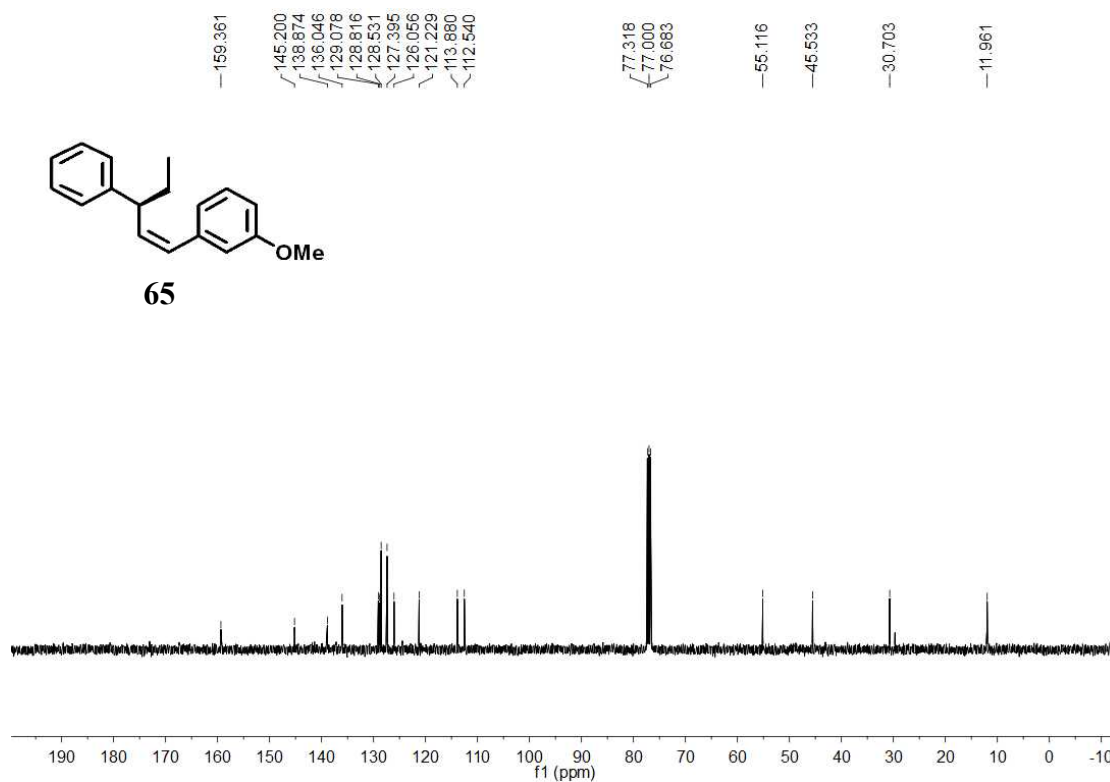
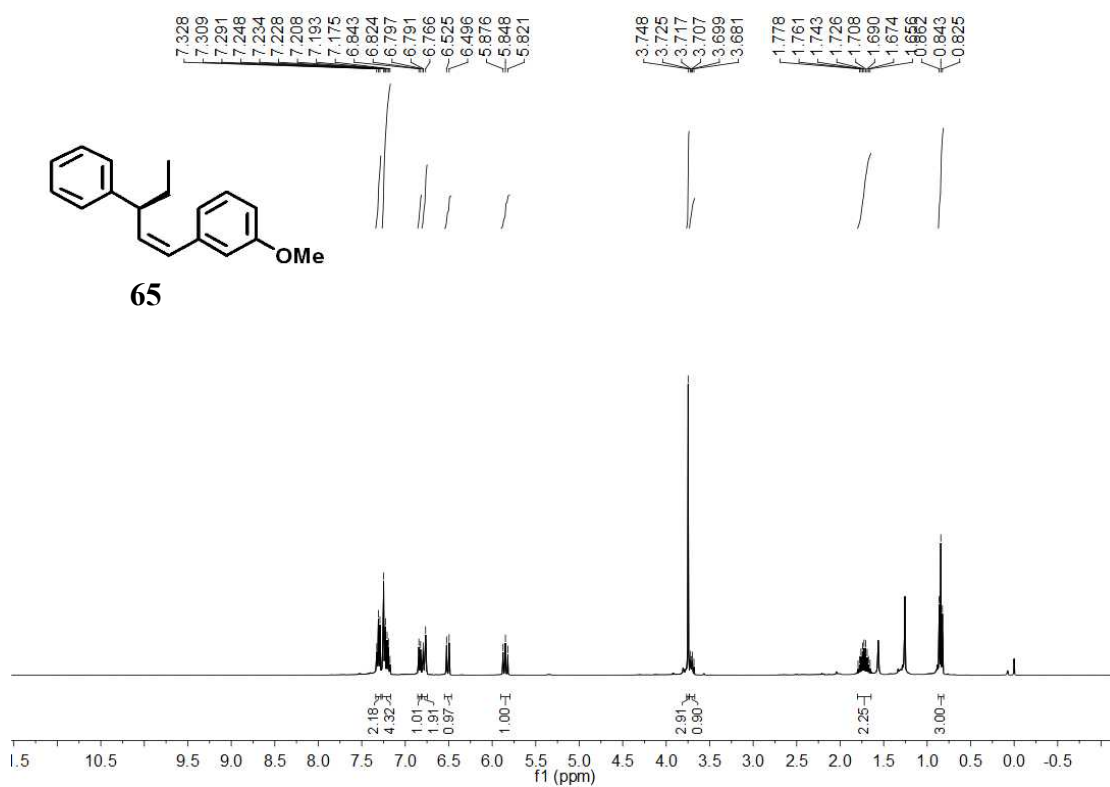


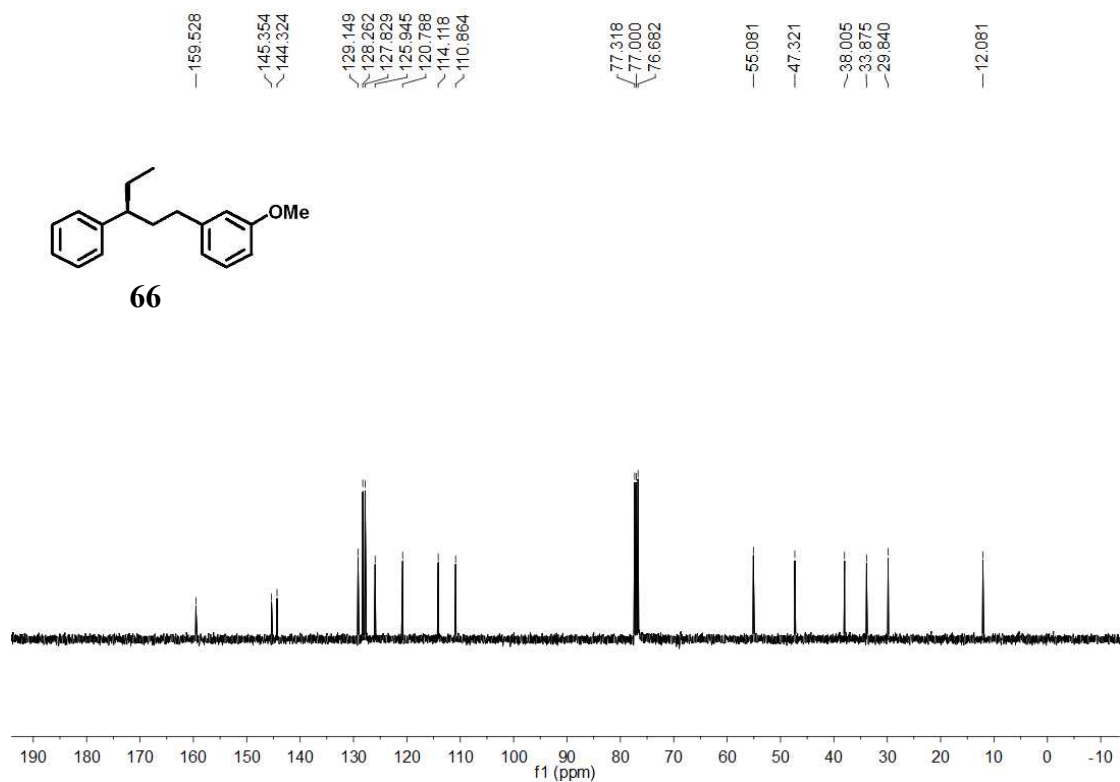
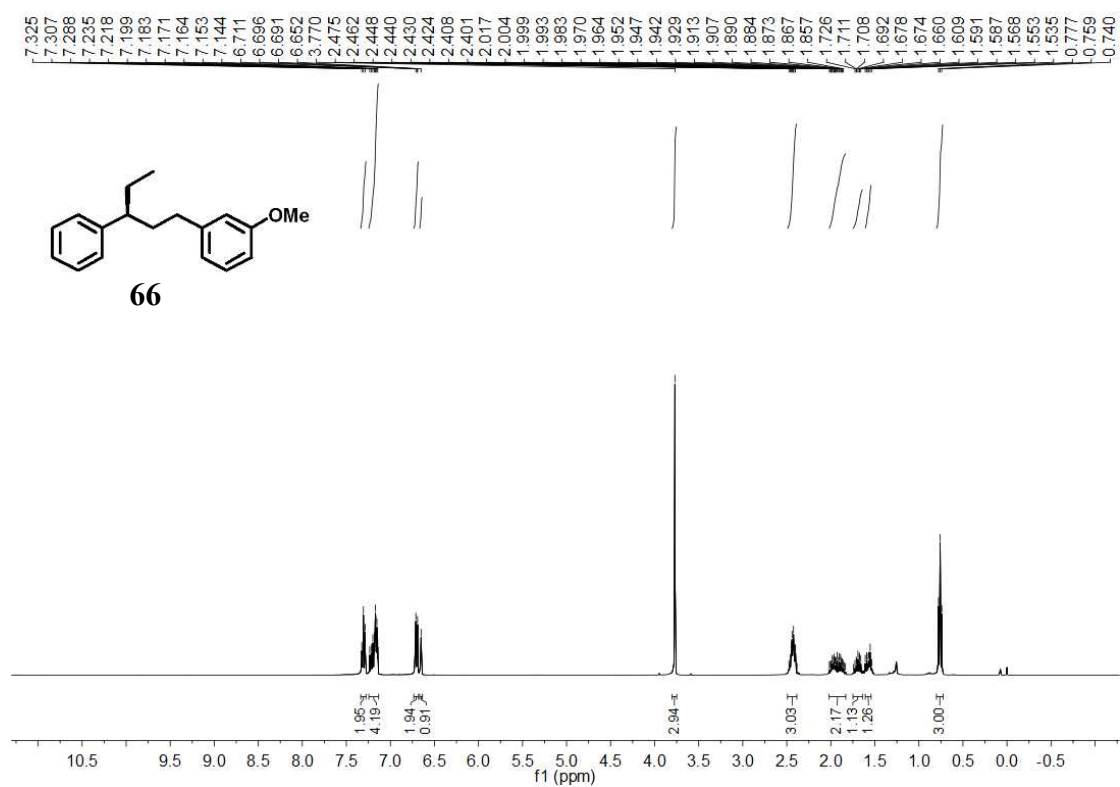


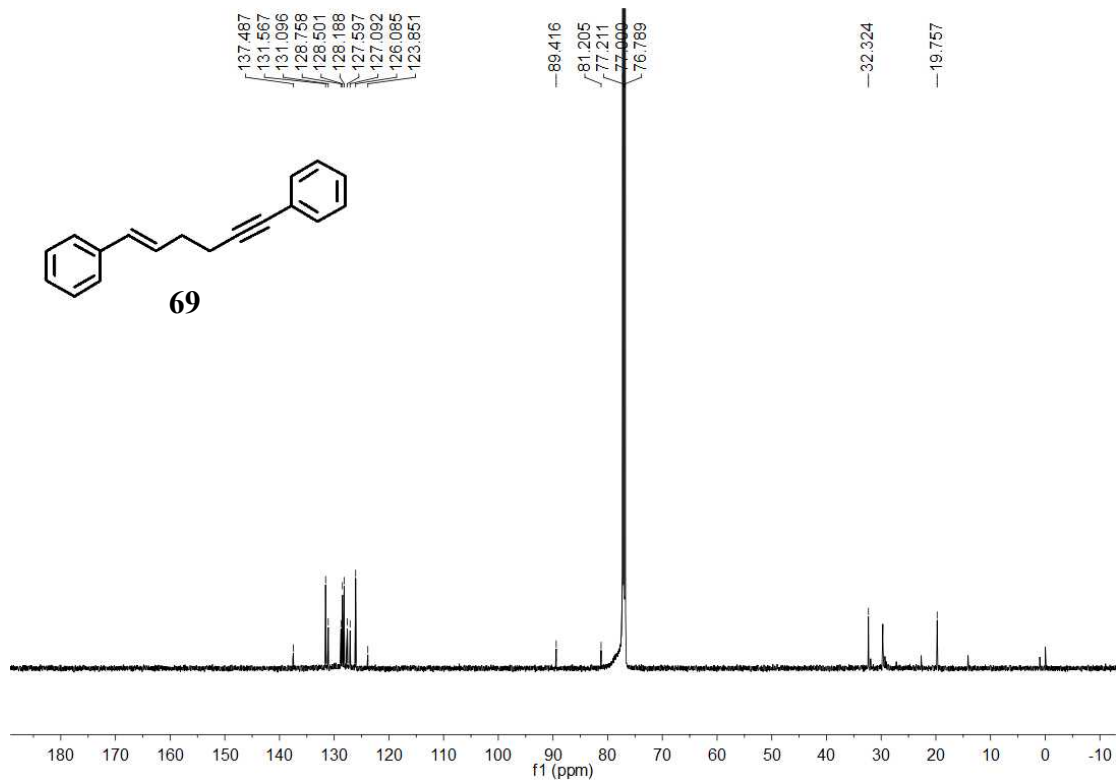
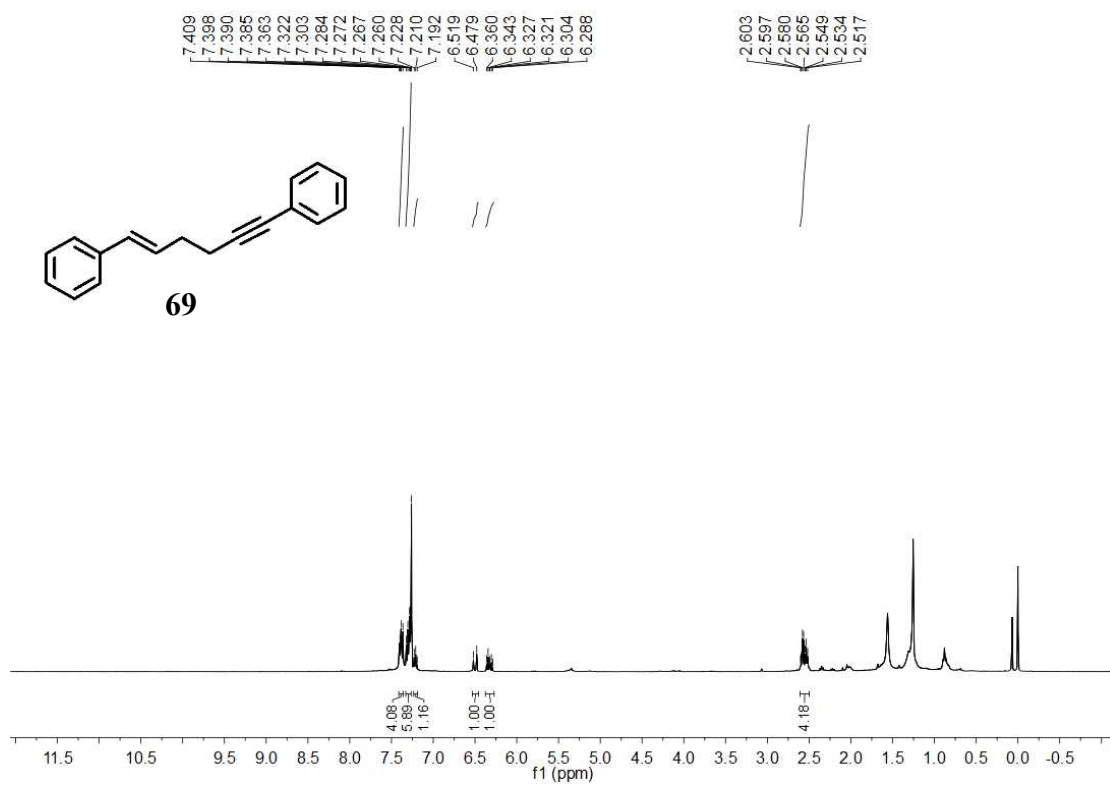


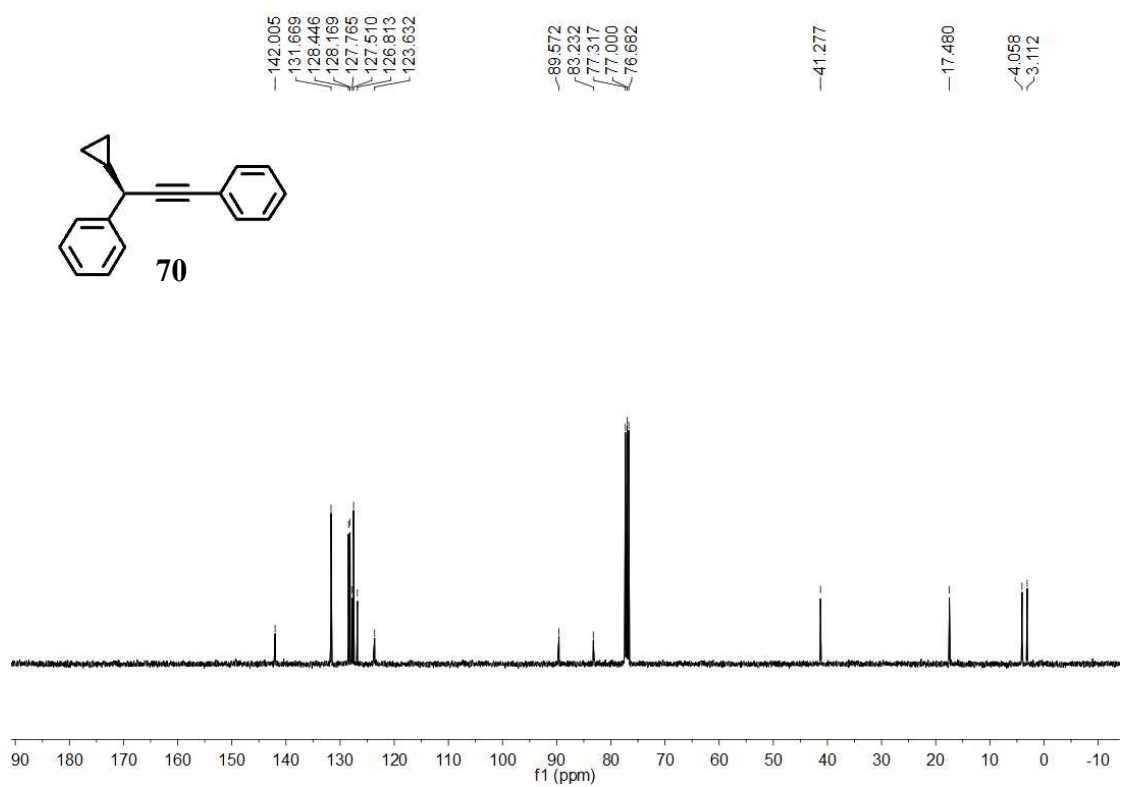
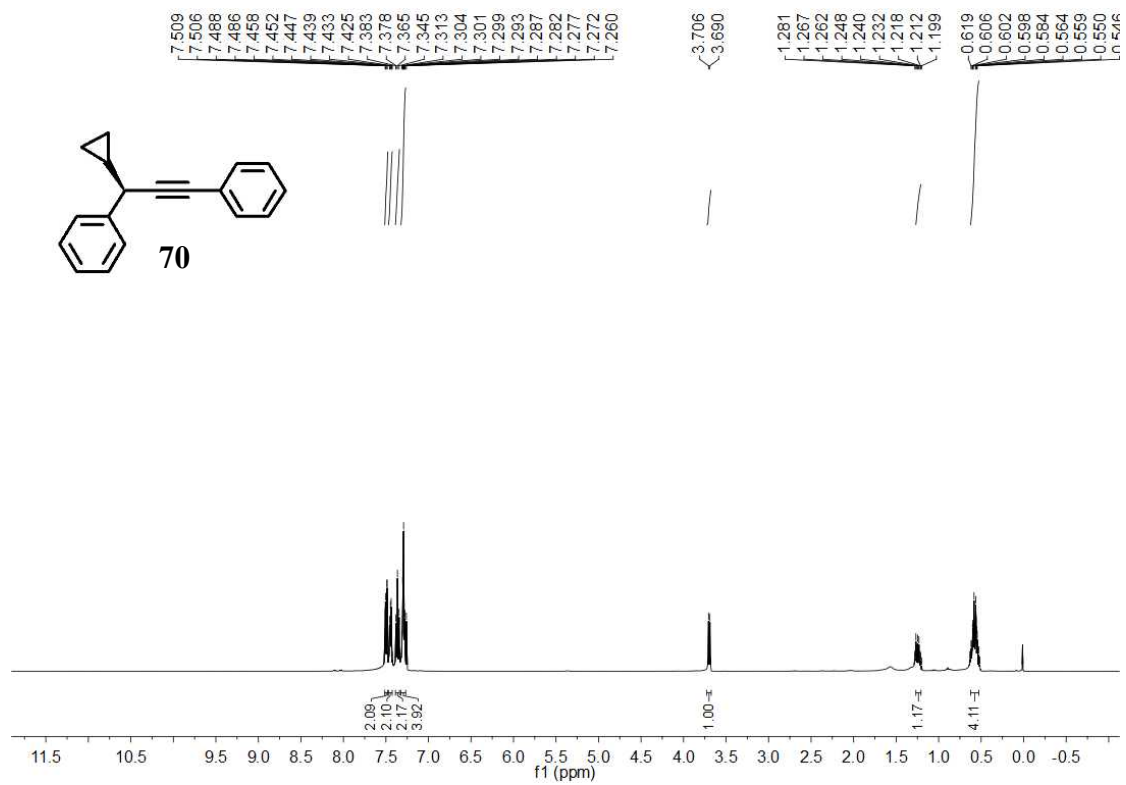




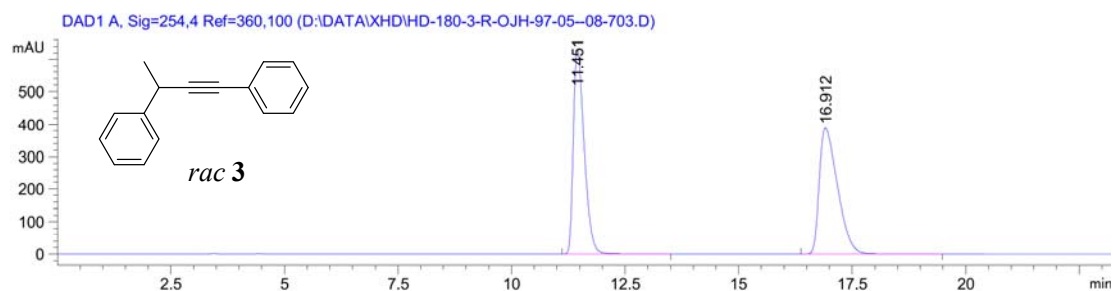








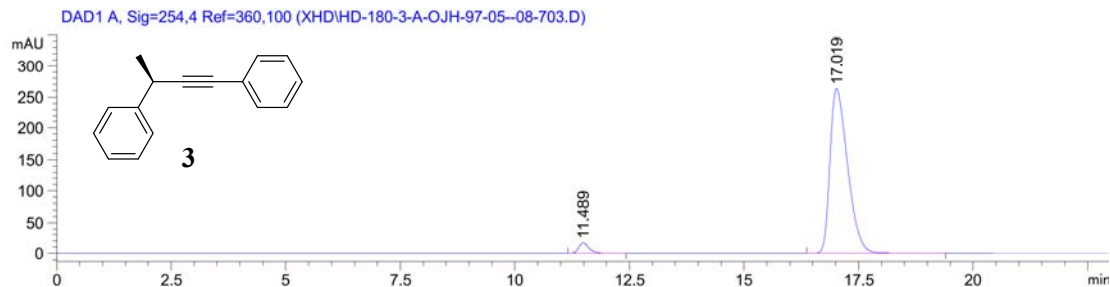
13. HPLC spectra



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.451	BB	0.2531	1.04525e4	631.57770	49.1012
2	16.912	BB	0.4311	1.08352e4	388.87234	50.8988

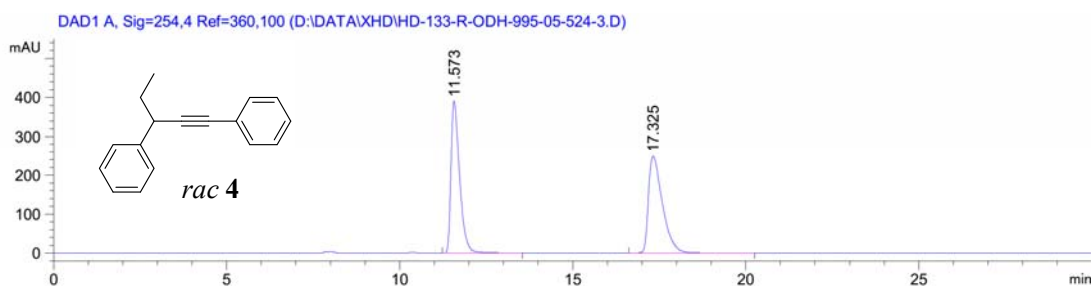
Totals : 2.12877e4 1020.45004



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.489	BB	0.2298	258.19003	16.93769	3.5304
2	17.019	BB	0.4143	7055.04590	263.56769	96.4696

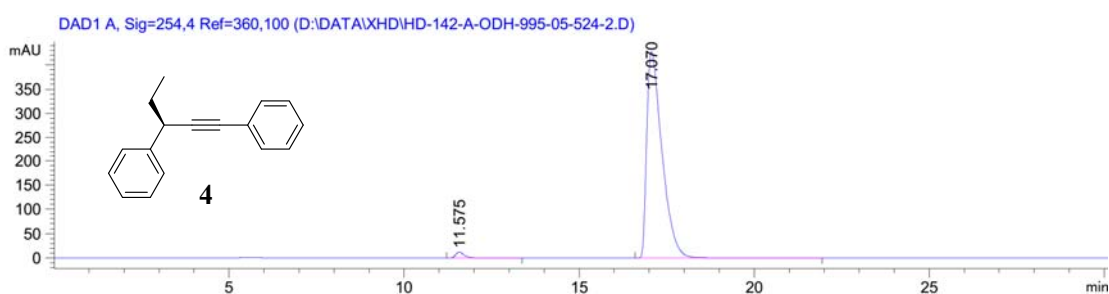
Totals : 7313.23593 280.50538



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.573	BB	0.2621	6775.78369	391.13312	49.3059
2	17.325	BB	0.4231	6966.55273	248.47195	50.6941

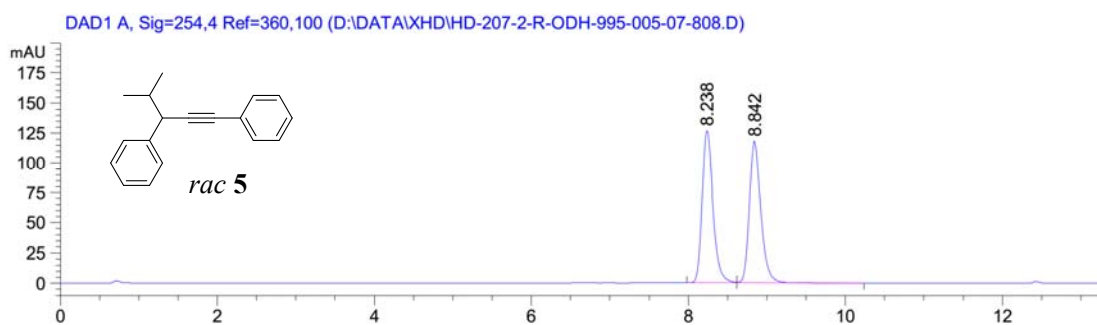
Totals : 1.37423e4 639.60507



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.575	BB	0.2630	210.00005	12.31266	1.5981
2	17.070	BB	0.4575	1.29307e4	426.81030	98.4019

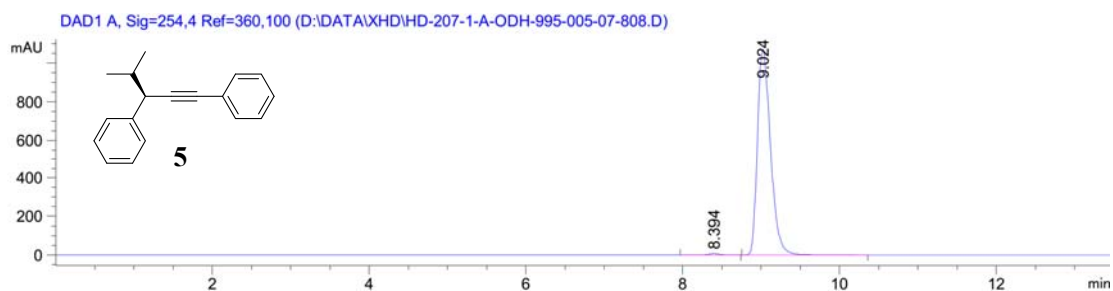
Totals : 1.31407e4 439.12296



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.238	BV	0.1473	1208.14026	126.69585	49.7011
2	8.842	VB	0.1613	1222.67090	117.64048	50.2989

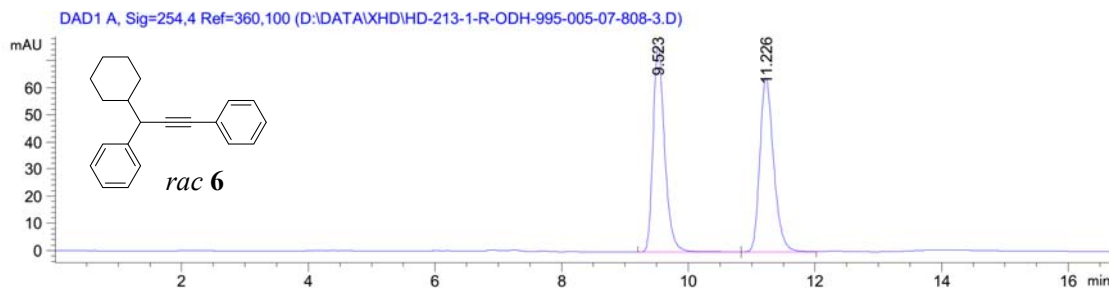
Totals : 2430.81116 244.33633



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.394	BB	0.1561	70.83384	7.11729	0.5663
2	9.024	BV	0.1841	1.24367e4	1066.27051	99.4337

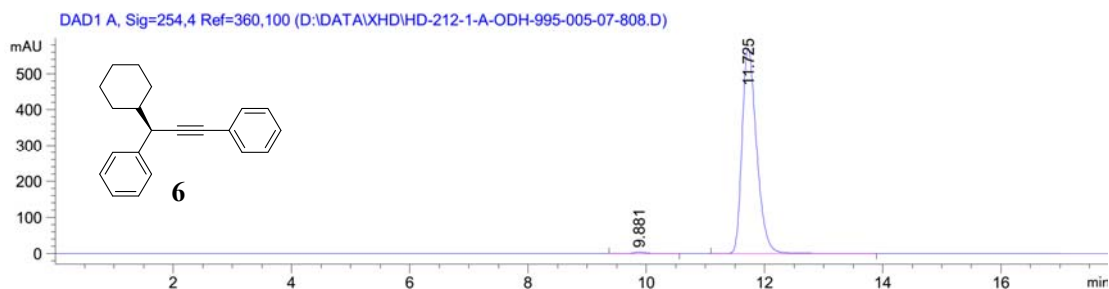
Totals : 1.25076e4 1073.38780



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.523	BB	0.1974	966.05292	75.45396	49.9102
2	11.226	BV	0.2325	969.52899	64.07447	50.0898

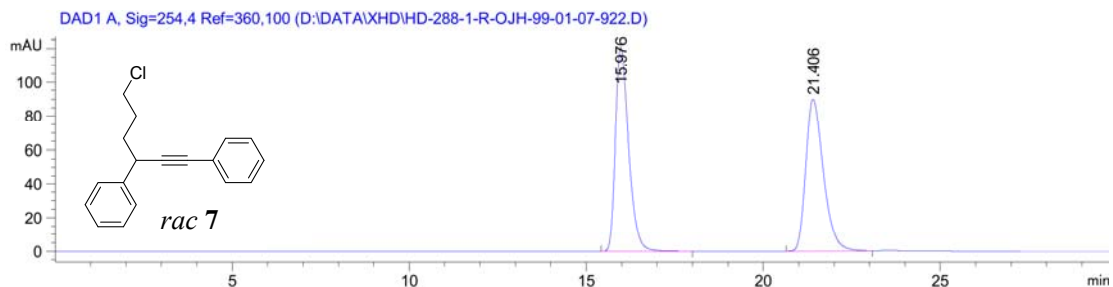
Totals : 1935.58191 139.52843



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.881	BB	0.2068	57.89194	4.25309	0.5897
2	11.725	BV	0.2637	9758.67871	569.99646	99.4103

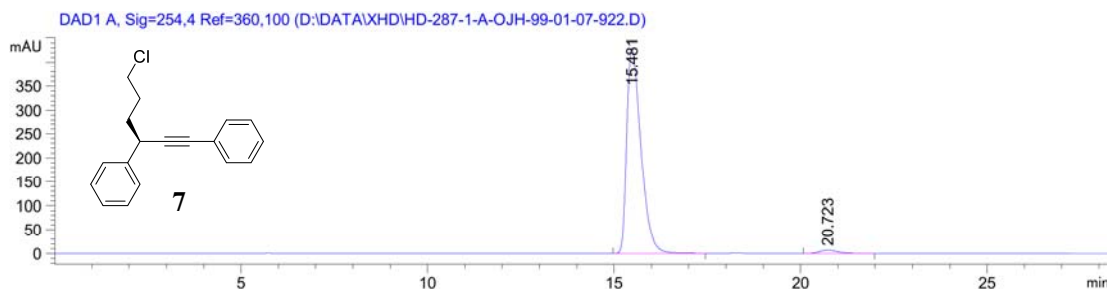
Totals : 9816.57065 574.24955



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.976	BB	0.3964	3135.25586	120.96627	50.0913
2	21.406	BB	0.5311	3123.82520	89.97477	49.9087

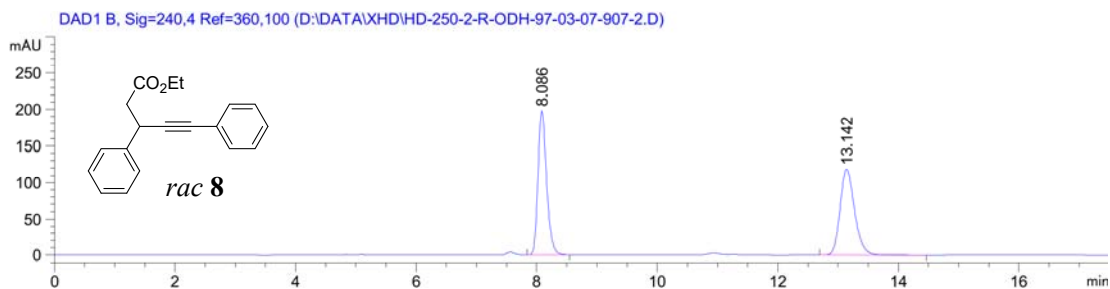
Totals : 6259.08105 210.94104



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.481	BB	0.4015	1.13086e4	429.03671	97.8884
2	20.723	BB	0.5189	243.94160	7.09948	2.1116

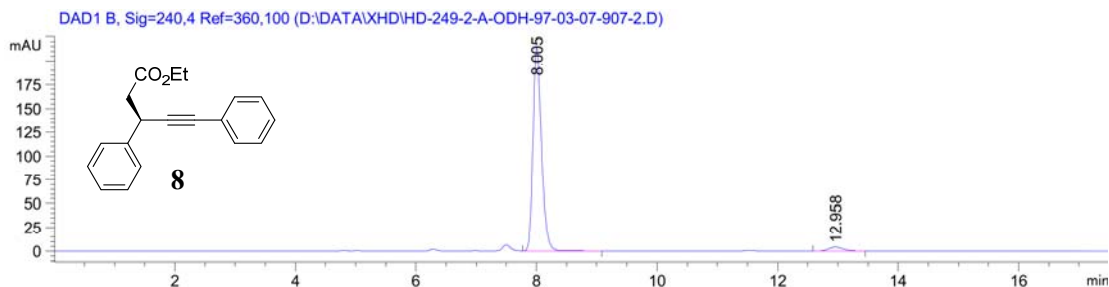
Totals : 1.15526e4 436.13619



Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.086	BB	0.1517	1962.60669	197.88177	49.8281
2	13.142	BB	0.2564	1976.15051	118.62304	50.1719

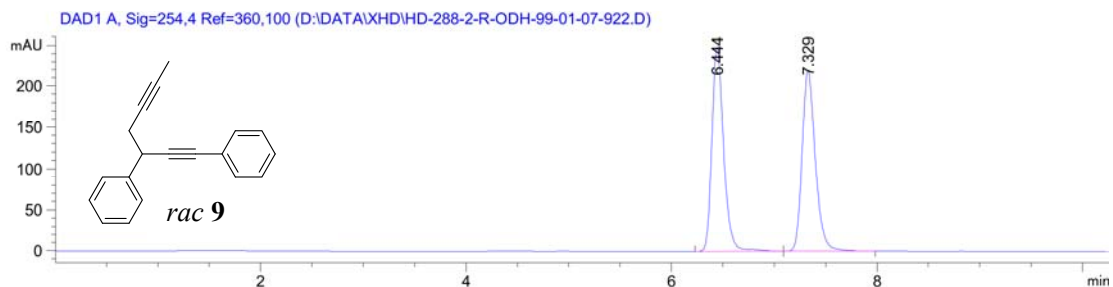
Totals : 3938.75720 316.50481



Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.005	BB	0.1480	2094.29419	214.41730	96.6518
2	12.958	BB	0.2546	72.55108	4.34861	3.3482

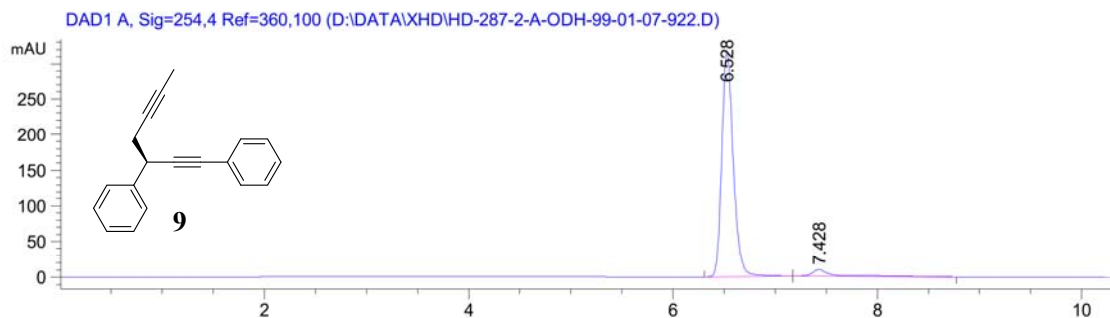
Totals : 2166.84527 218.76591



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.444	BB	0.1208	1981.47229	249.19753	50.0221
2	7.329	BB	0.1374	1979.72217	219.06058	49.9779

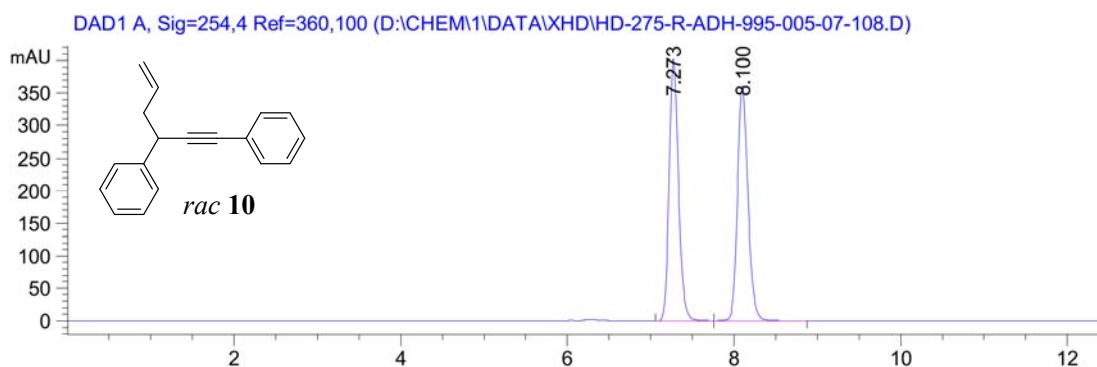
Totals : 3961.19446 468.25810



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.528	BB	0.1187	2471.37378	317.88901	95.2382
2	7.428	BB	0.1910	123.56570	8.95385	4.7618

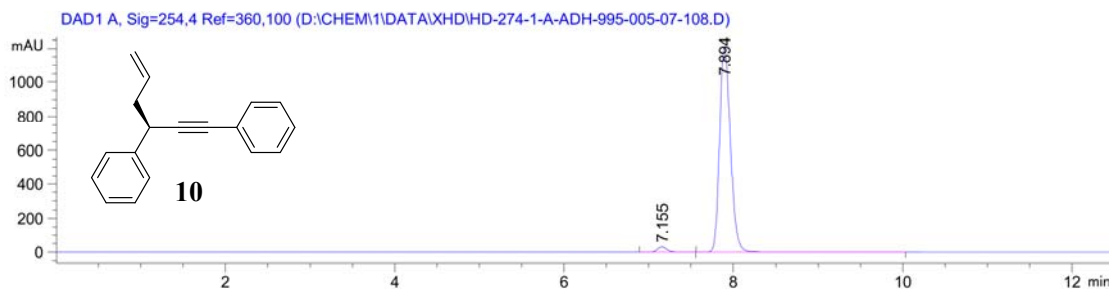
Totals : 2594.93948 326.84286



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.273	BB	0.1210	3137.54028	402.37854	49.8331
2	8.100	BB	0.1340	3158.55151	361.41068	50.1669

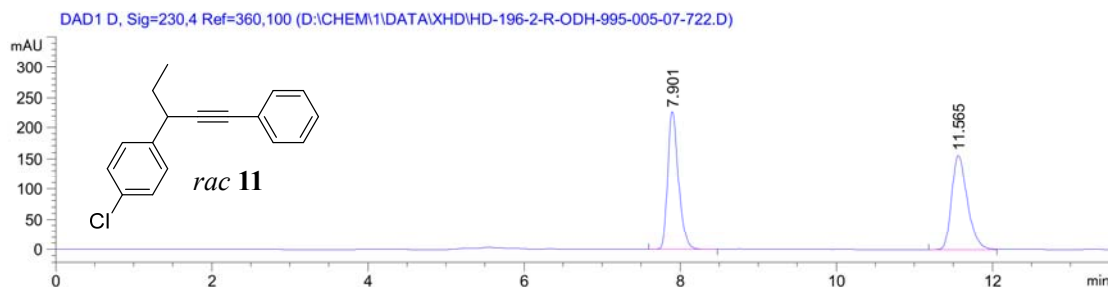
Totals : 6296.09180 763.78922



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.155	BB	0.1151	237.03439	31.75590	2.1532
2	7.894	BB	0.1380	1.07712e4	1207.91199	97.8468

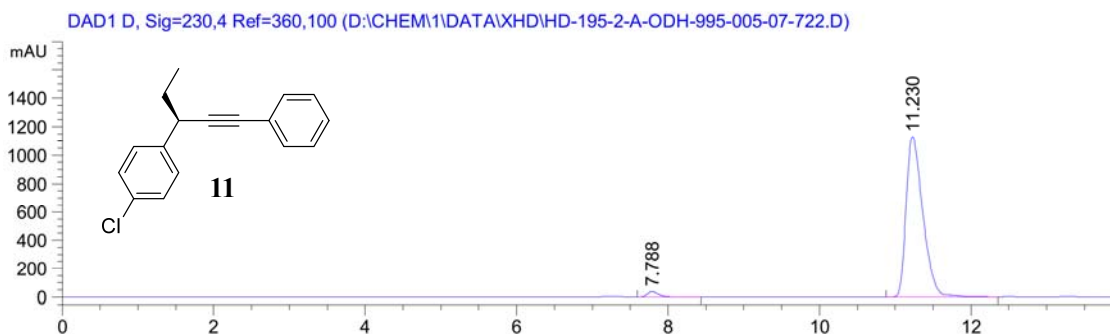
Totals : 1.10083e4 1239.66789



Signal 3: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.901	BB	0.1463	2182.33105	226.63695	50.0363
2	11.565	BV	0.2154	2179.16797	155.61476	49.9637

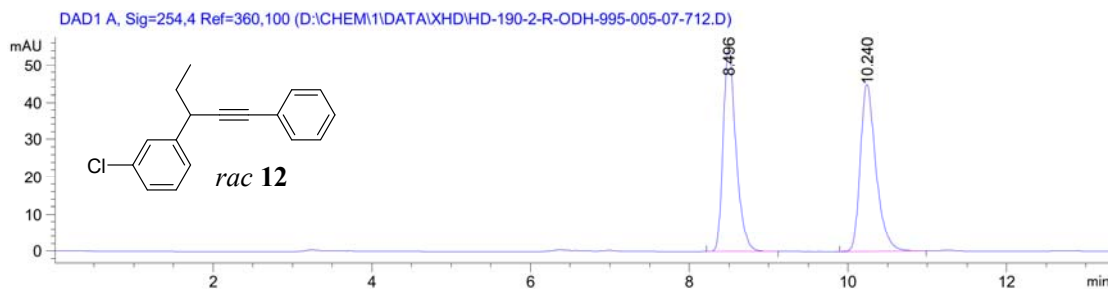
Totals : 4361.49902 382.25171



Signal 3: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.788	VB	0.1481	371.85010	38.00632	2.0977
2	11.230	BV	0.2371	1.73547e4	1130.20020	97.9023

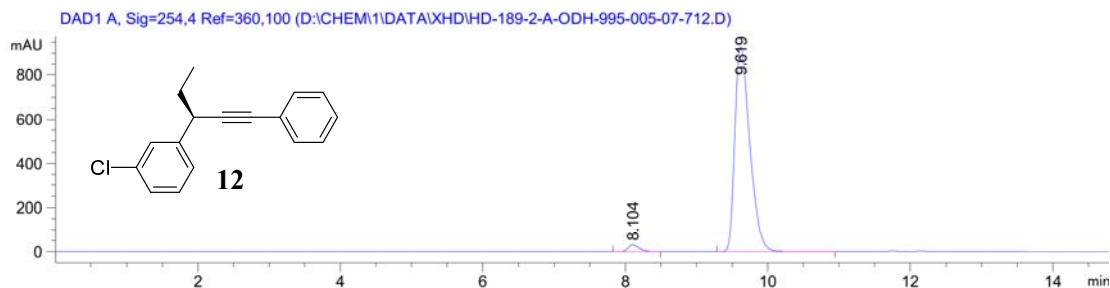
Totals : 1.77266e4 1168.20652



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.496	BB	0.1686	614.84637	54.93124	49.8986
2	10.240	BB	0.2067	617.34589	44.81832	50.1014

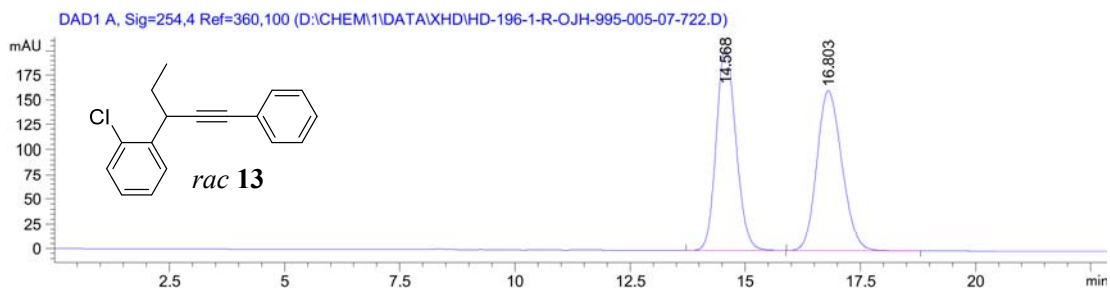
Totals : 1232.19226 99.74955



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.104	BB	0.1564	322.04242	31.20131	2.3435
2	9.619	BB	0.2227	1.34197e4	928.03546	97.6565

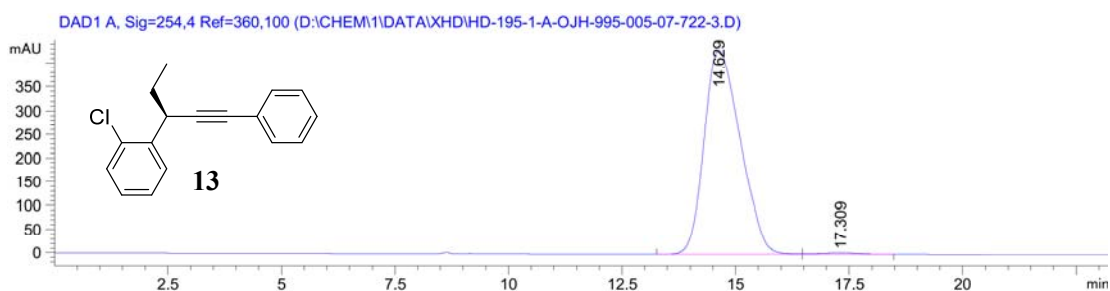
Totals : 1.37417e4 959.23677



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.568	BB	0.4621	6091.39063	205.31598	49.8681
2	16.803	BB	0.5917	6123.62305	161.02007	50.1319

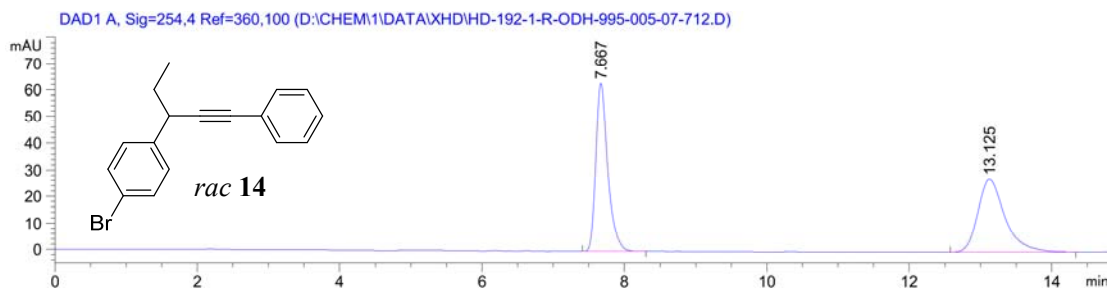
Totals : 1.22150e4 366.33604



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.629	BV	0.8784	2.38821e4	431.04367	99.0945
2	17.309	VB	0.9400	218.21996	3.50659	0.9055

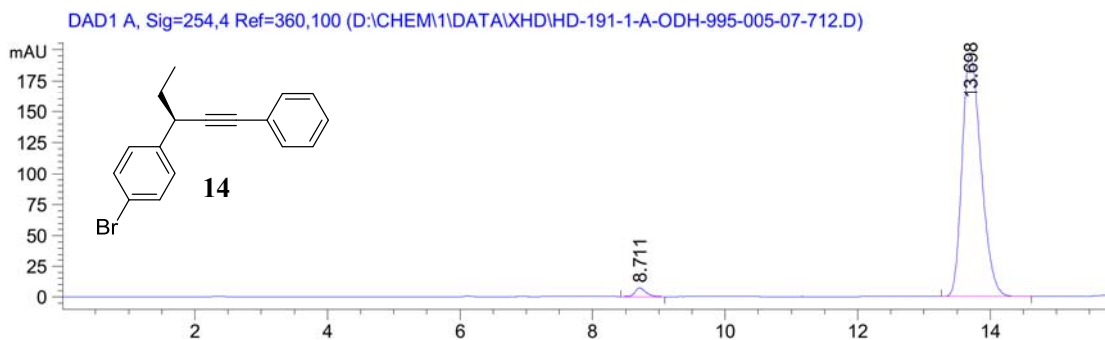
Totals : 2.41004e4 434.55026



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.667	BB	0.1732	732.37317	63.21516	49.9649
2	13.125	BB	0.4022	733.40210	27.58067	50.0351

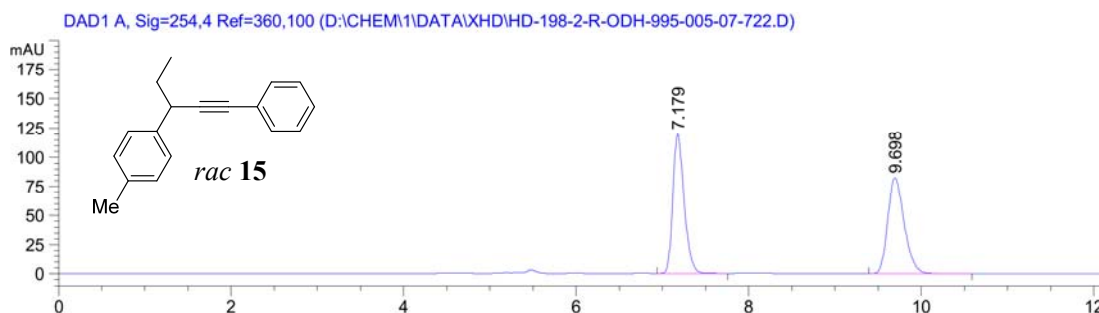
Totals : 1465.77527 90.79583



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.711	BB	0.1674	79.05783	7.12743	1.9850
2	13.698	BB	0.3102	3903.77417	196.14410	98.0150

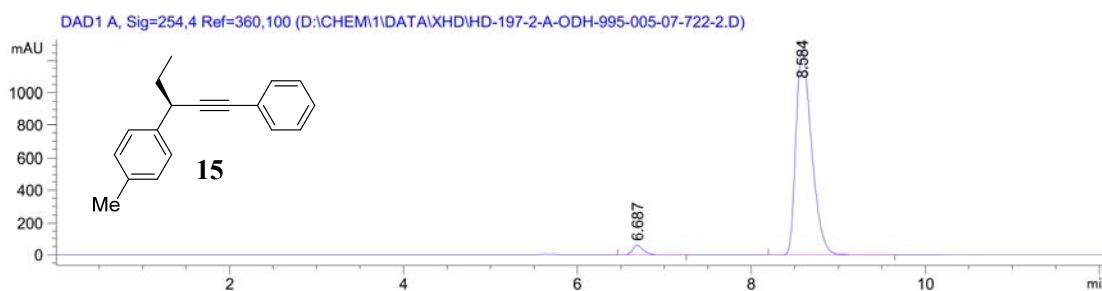
Totals : 3982.83200 203.27153



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.179	VV	0.1388	1097.91016	119.98072	50.2203
2	9.698	BB	0.2022	1088.27795	82.32094	49.7797

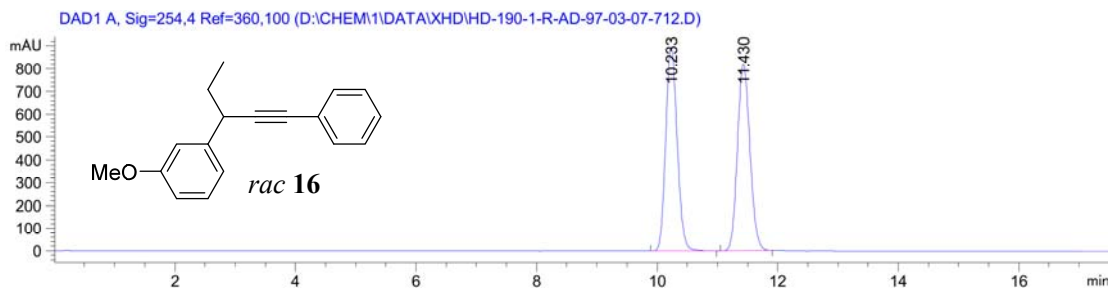
Totals : 2186.18811 202.30166



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.687	VB	0.1284	499.95093	59.25242	3.0369
2	8.584	BV	0.1929	1.59628e4	1267.41858	96.9631

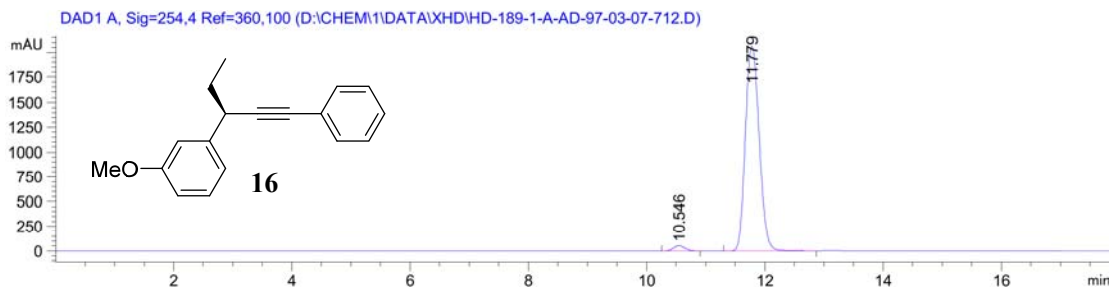
Totals : 1.64628e4 1326.67100



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.233	BB	0.2084	1.18611e4	896.11621	49.9979
2	11.430	BB	0.2264	1.18621e4	821.67535	50.0021

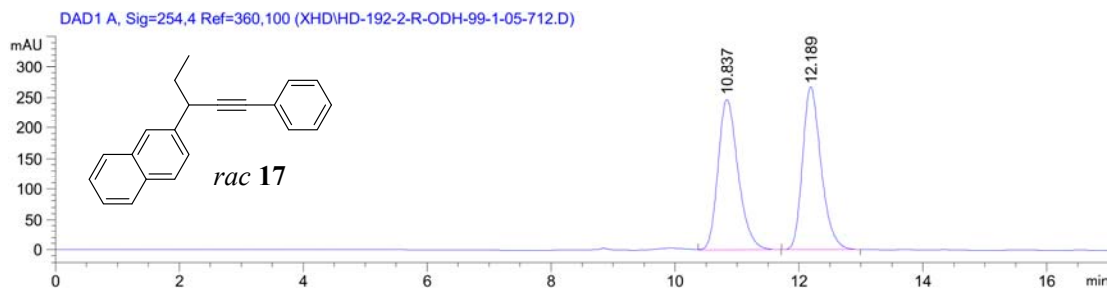
Totals : 2.37232e4 1717.79156



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.546	BB	0.2041	697.89124	53.52661	2.0315
2	11.779	BV	0.2583	3.36548e4	2063.56543	97.9685

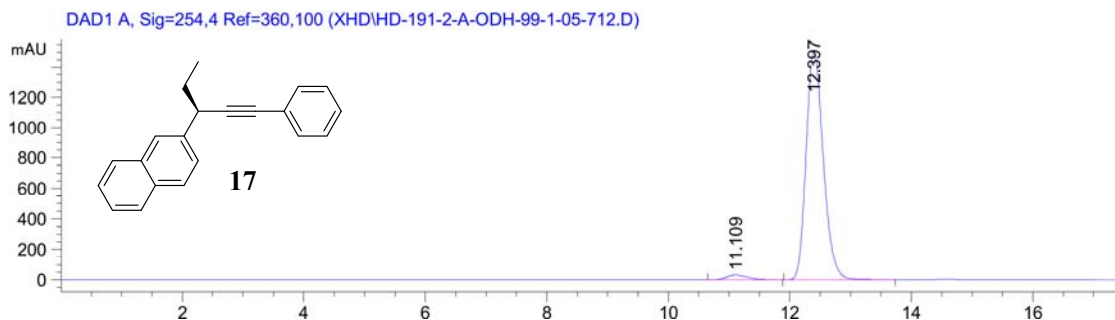
Totals : 3.43527e4 2117.09204



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.837	VB	0.3455	5585.55176	247.31053	50.1053
2	12.189	BB	0.3177	5562.08203	266.26132	49.8947

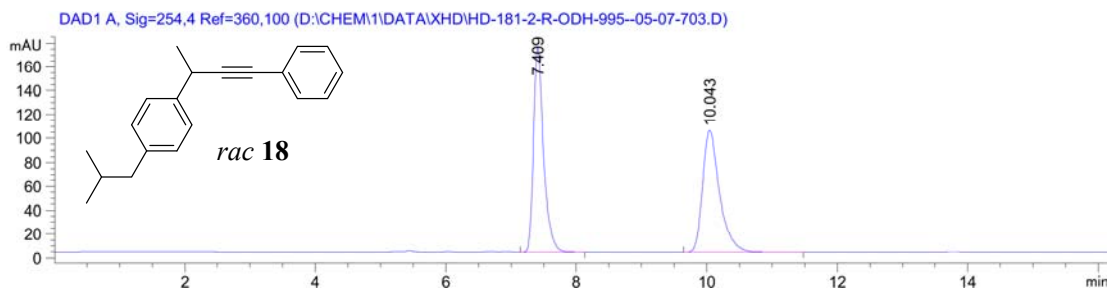
Totals : 1.11476e4 513.57185



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.109	BB	0.3675	747.47437	31.42877	2.4735
2	12.397	BB	0.3042	2.94720e4	1506.47449	97.5265

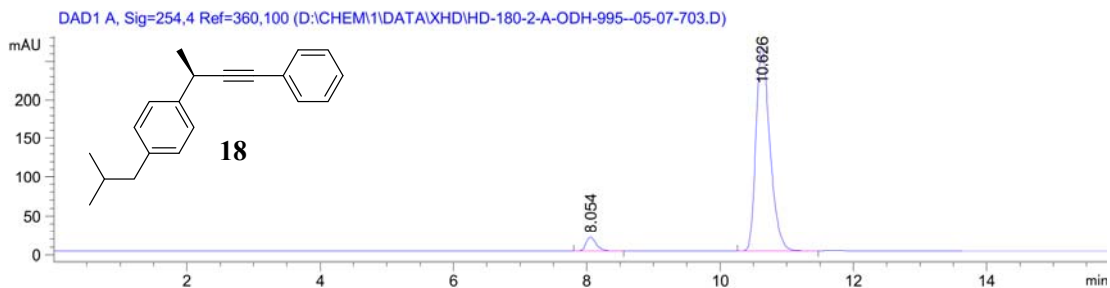
Totals : 3.02195e4 1537.90326



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.409	BB	0.1603	1831.47180	171.95747	49.8572
2	10.043	BB	0.2714	1841.96570	101.64752	50.1428

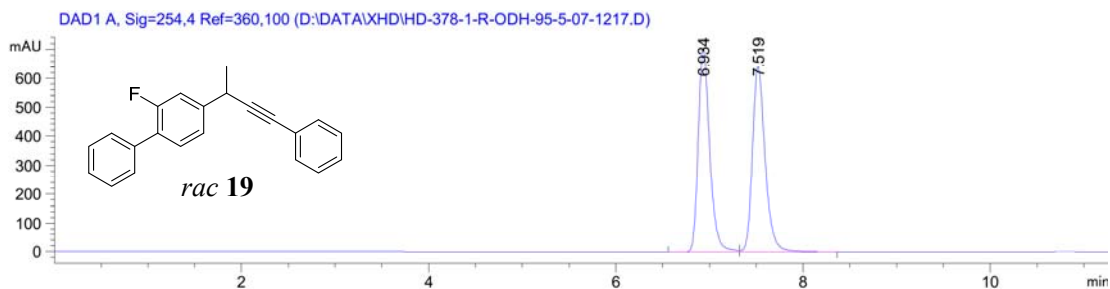
Totals : 3673.43750 273.60500



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.054	BB	0.1563	186.18045	18.05705	4.6331
2	10.626	BB	0.2214	3832.33521	263.98868	95.3669

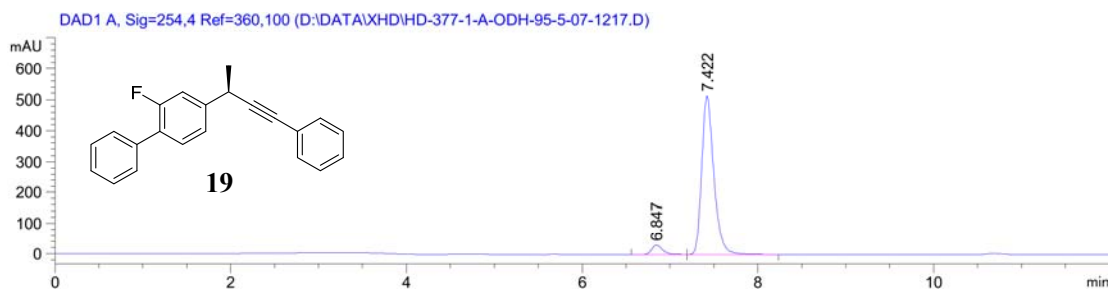
Totals : 4018.51566 282.04573



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.934	BV	0.1295	6033.58447	707.47205	49.7898
2	7.519	VV	0.1427	6084.52051	641.32257	50.2102

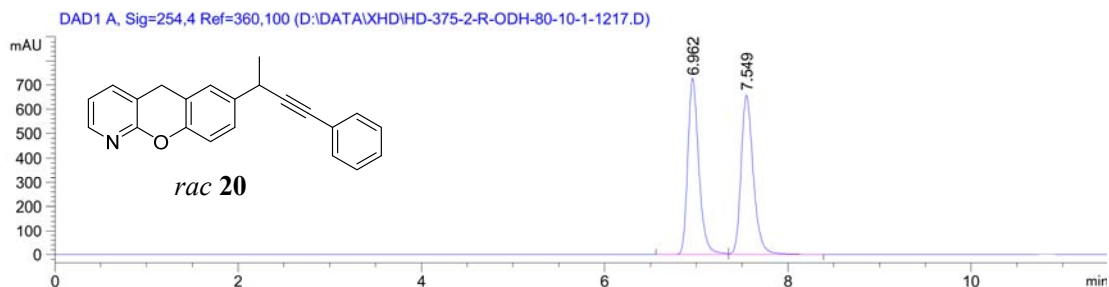
Totals : 1.21181e4 1348.79462



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.847	BV	0.1313	270.47659	31.14642	5.1869
2	7.422	VB	0.1463	4944.12695	513.62701	94.8131

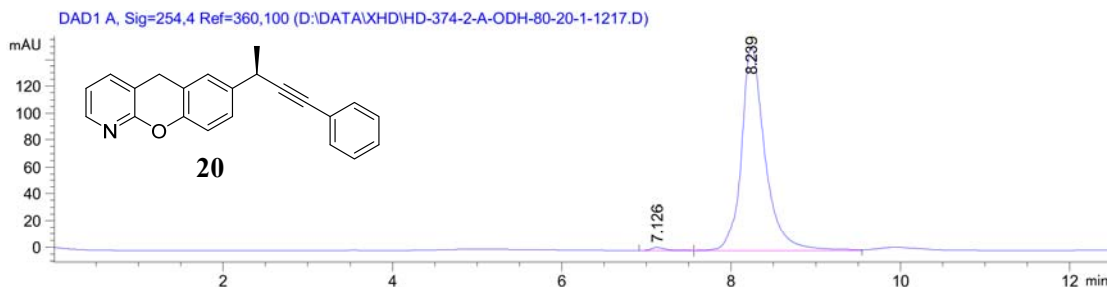
Totals : 5214.60355 544.77343



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.962	BV	0.1281	6102.81348	725.70984	49.7660
2	7.549	VV	0.1433	6160.20996	657.40430	50.2340

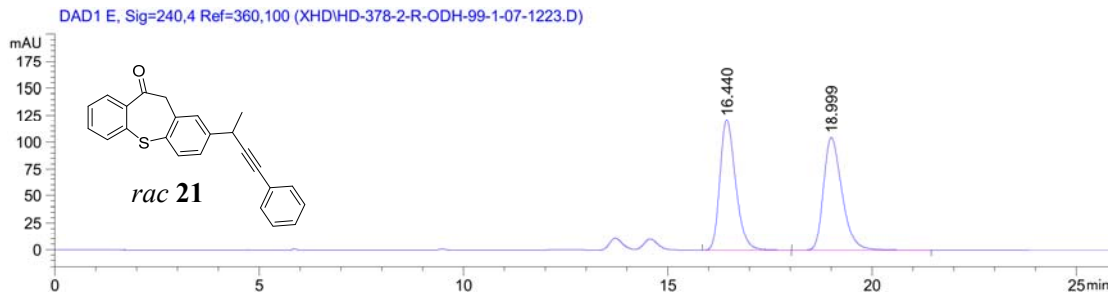
Totals : 1.22630e4 1383.11414



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.126	BB	0.1709	25.50886	2.23928	0.8617
2	8.239	BV	0.2824	2934.84790	152.55948	99.1383

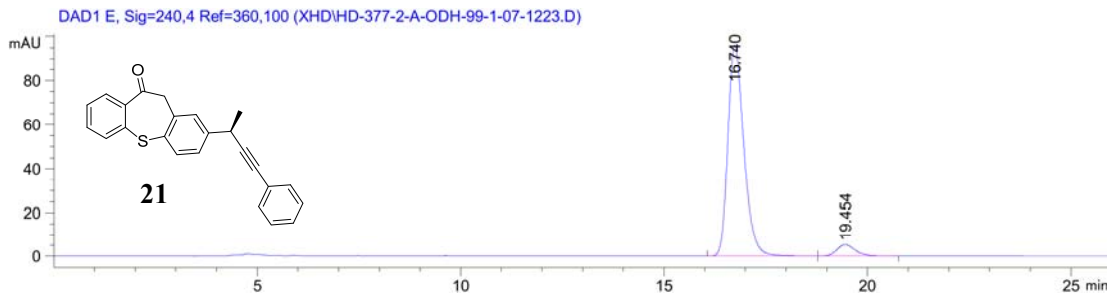
Totals : 2960.35676 154.79875



Signal 5: DAD1 E, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.440	BB	0.4122	3249.26440	121.41182	49.5960
2	18.999	BB	0.4843	3302.20605	104.60402	50.4040

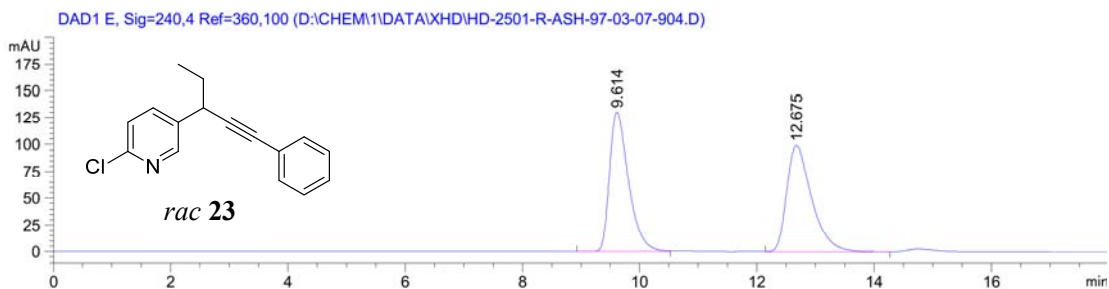
Totals : 6551.47046 226.01584



Signal 5: DAD1 E, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.740	BV	0.4194	2635.20630	96.24438	94.0122
2	19.454	VB	0.4826	167.84167	5.31247	5.9878

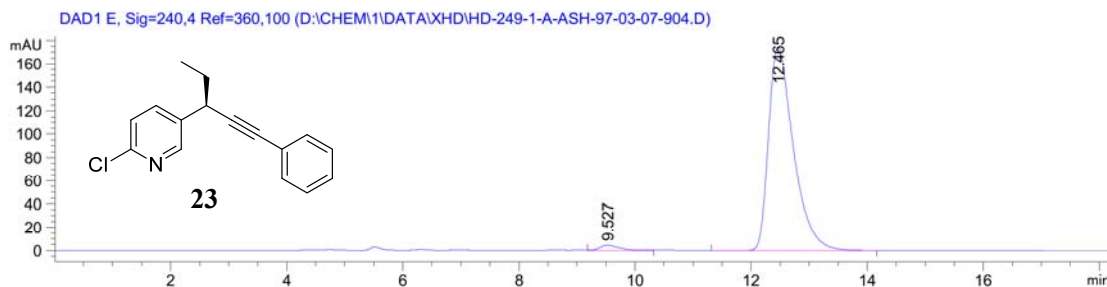
Totals : 2803.04797 101.55685



Signal 5: DAD1 E, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.614	BV	0.3440	2947.13159	130.22351	50.0039
2	12.675	VV	0.4508	2946.66797	99.12495	49.9961

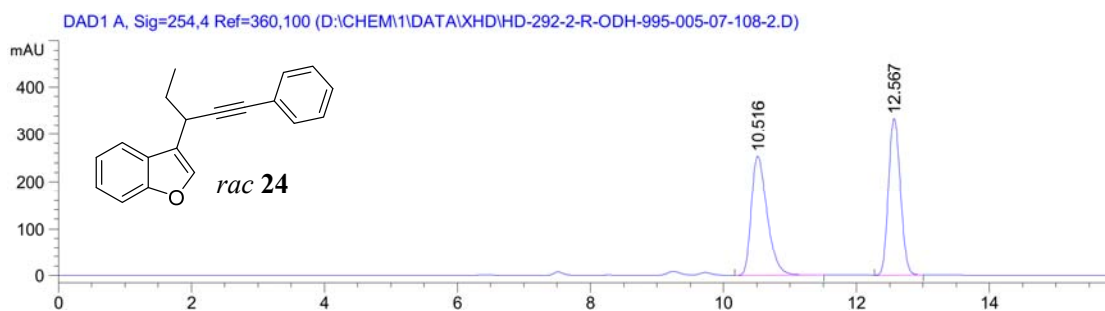
Totals : 5893.79956 229.34846



Signal 5: DAD1 E, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.527	VV	0.3428	102.88322	4.49958	1.9398
2	12.465	BV	0.4511	5201.03955	174.81360	98.0602

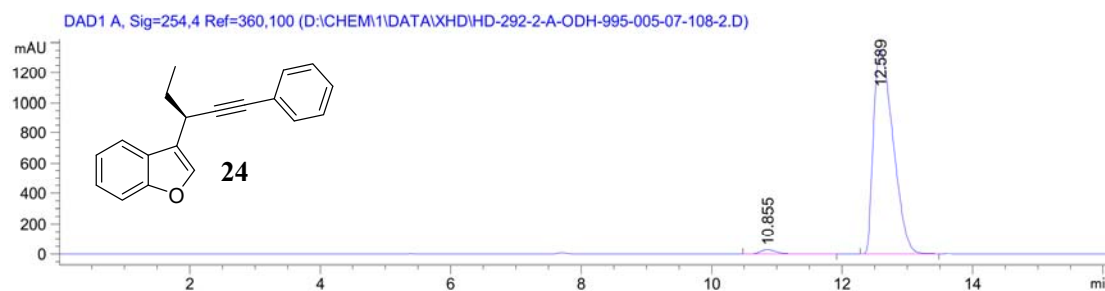
Totals : 5303.92278 179.31318



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.516	BB	0.2542	4212.61621	253.13699	50.2439
2	12.567	BB	0.1957	4171.71777	333.96585	49.7561

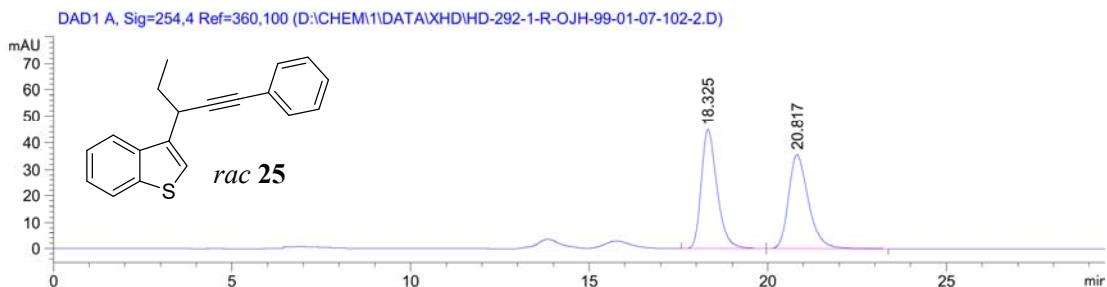
Totals : 8384.33398 587.10284



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.855	BB	0.2545	482.30731	29.23704	1.6910
2	12.589	BB	0.3340	2.80403e4	1352.04272	98.3090

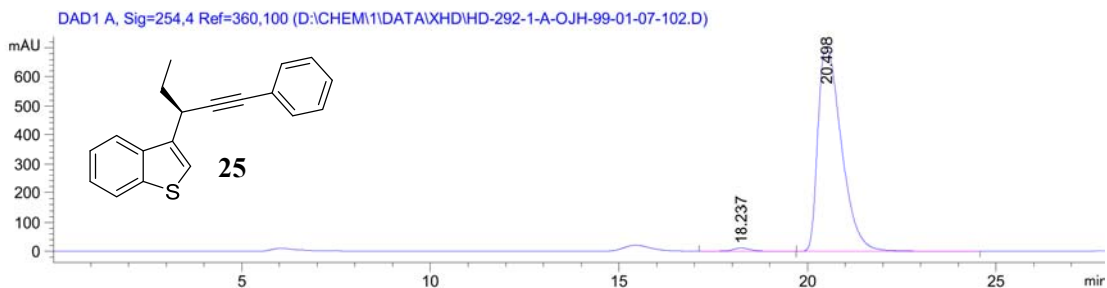
Totals : 2.85226e4 1381.27976



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.325	BB	0.4736	1391.97034	44.91646	50.1849
2	20.817	BB	0.5940	1381.71057	35.66548	49.8151

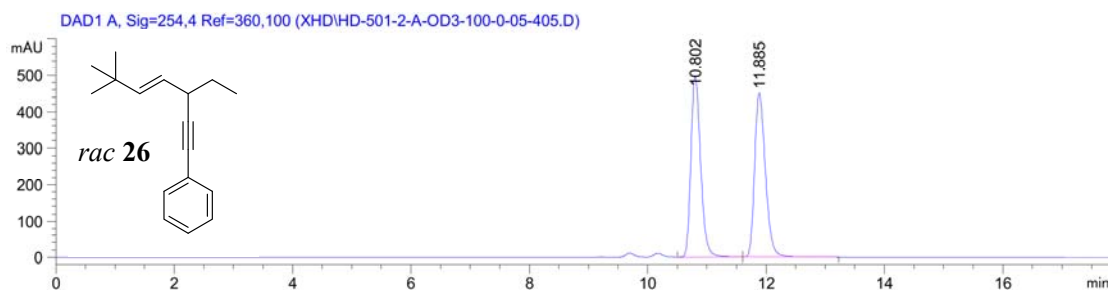
Totals : 2773.68091 80.58194



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.237	VB	0.5151	393.77368	11.39655	1.2798
2	20.498	BV	0.6687	3.03749e4	702.08319	98.7202

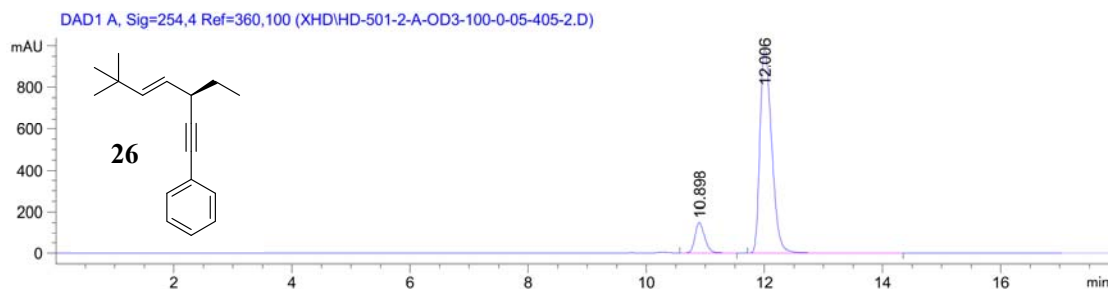
Totals : 3.07687e4 713.47974



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.802	BB	0.1771	5676.90234	497.68698	49.8766
2	11.885	BB	0.1959	5704.99756	449.96069	50.1234

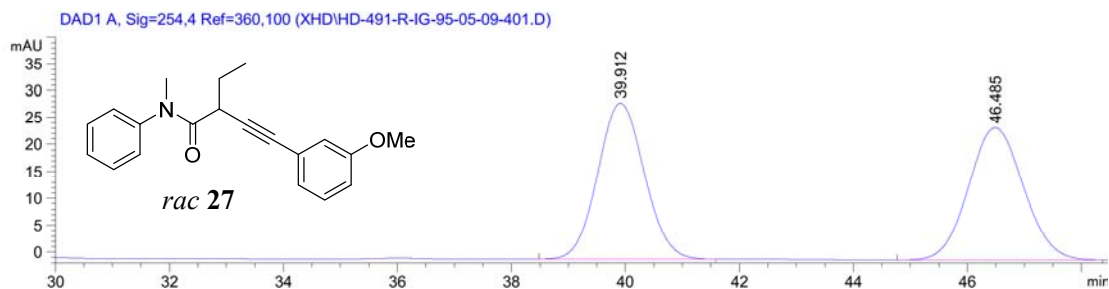
Totals : 1.13819e4 947.64767



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.898	VB	0.1778	1734.65515	151.28131	11.2730
2	12.006	BB	0.2154	1.36531e4	986.99860	88.7270

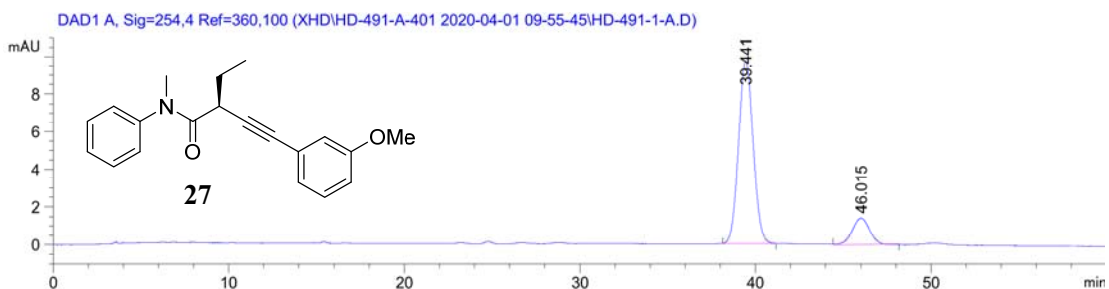
Totals : 1.53877e4 1138.27991



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	39.912	BB	0.8856	1648.83411	28.98965	49.9614
2	46.485	BB	1.0410	1651.38354	24.54158	50.0386

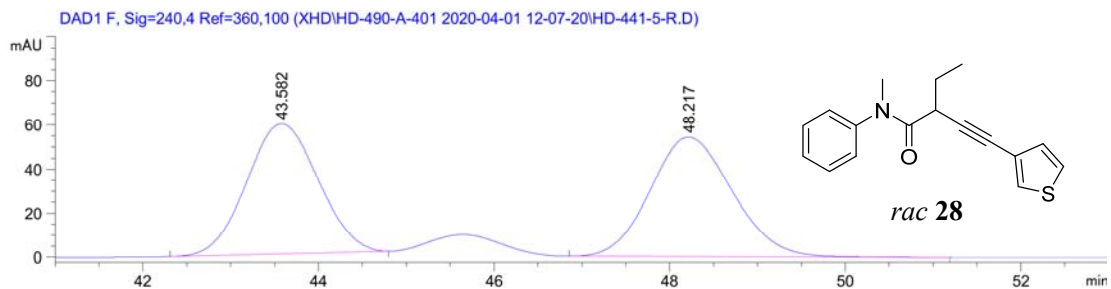
Totals : 3300.21765 53.53123



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	39.441	BB	0.8872	559.48083	9.69614	85.0907
2	46.015	MM R	1.1471	98.03036	1.42432	14.9093

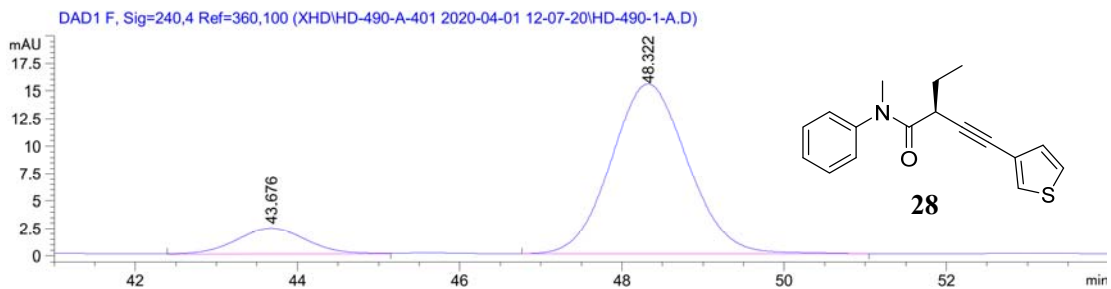
Totals : 657.51119 11.12046



Signal 6: DAD1 F, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	43.582	BB	0.8890	3350.97144	58.96780	48.6539
2	48.217	BB	1.0146	3536.38965	54.10306	51.3461

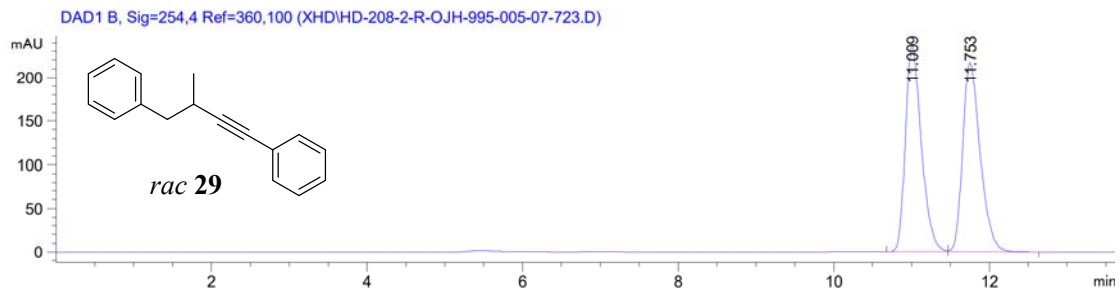
Totals : 6887.36108 113.07086



Signal 6: DAD1 F, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	43.676	MM R	1.0035	141.98506	2.35805	12.2424
2	48.322	BB	1.0011	1017.80072	15.43868	87.7576

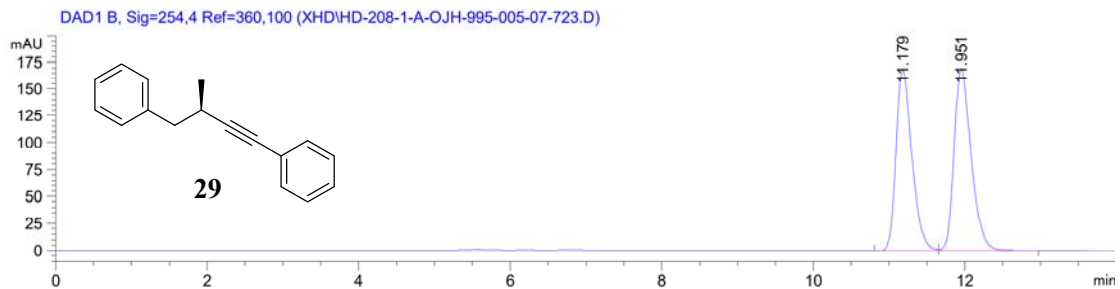
Totals : 1159.78578 17.79674



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.009	BV	0.2162	3363.47852	236.12163	49.8582
2	11.753	VB	0.2379	3382.61279	216.90707	50.1418

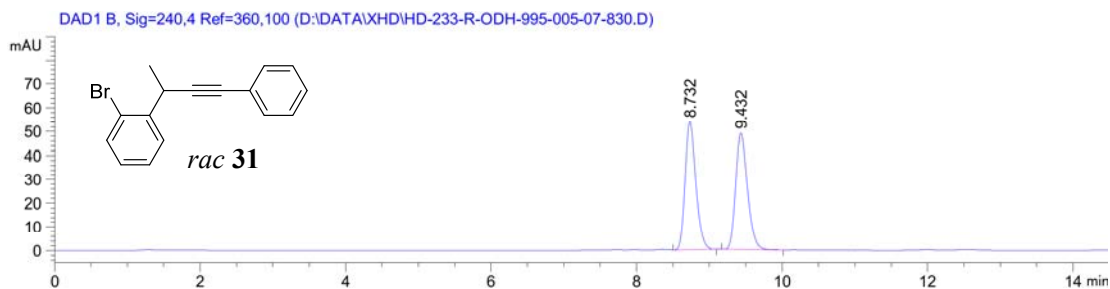
Totals : 6746.09131 453.02870



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.179	BV	0.2187	2431.72095	168.18225	48.0375
2	11.951	VB	0.2353	2630.40942	169.24474	51.9625

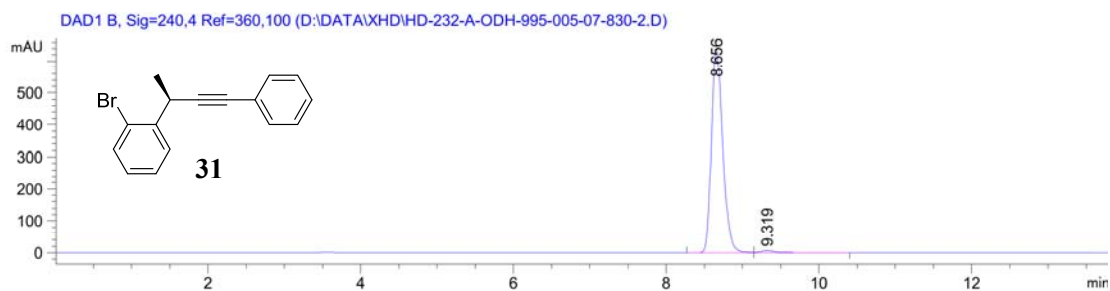
Totals : 5062.13037 337.42699



Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.732	BB	0.1591	556.27118	53.59978	49.9213
2	9.432	BB	0.1750	558.02576	48.93664	50.0787

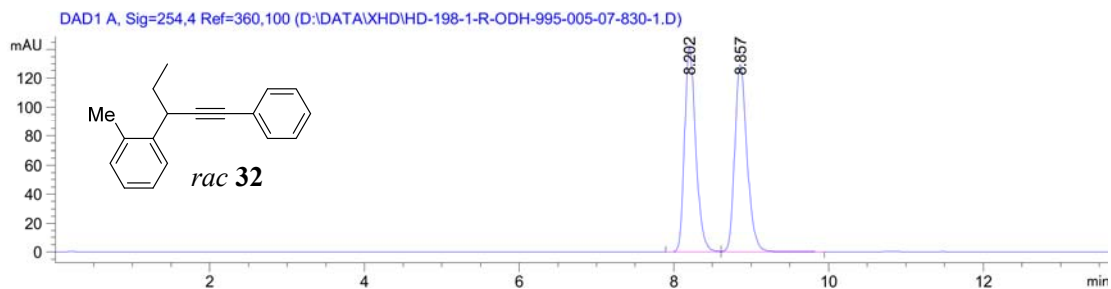
Totals : 1114.29694 102.53642



Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.656	BV	0.1558	6579.82178	640.59808	98.7241
2	9.319	VB	0.1976	85.03582	6.21492	1.2759

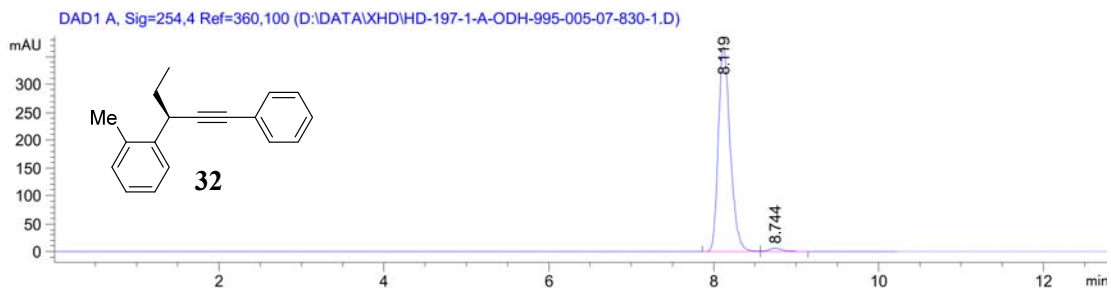
Totals : 6664.85760 646.81300



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.202	BV	0.1530	1421.47070	141.70264	49.7813
2	8.857	VB	0.1691	1433.95911	129.53770	50.2187

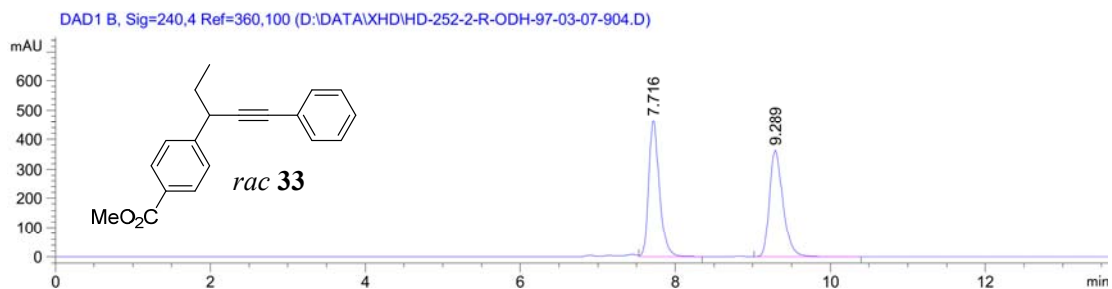
Totals : 2855.42981 271.24034



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.119	BV	0.1526	3680.53027	368.21539	98.2218
2	8.744	VB	0.1713	66.63286	5.82966	1.7782

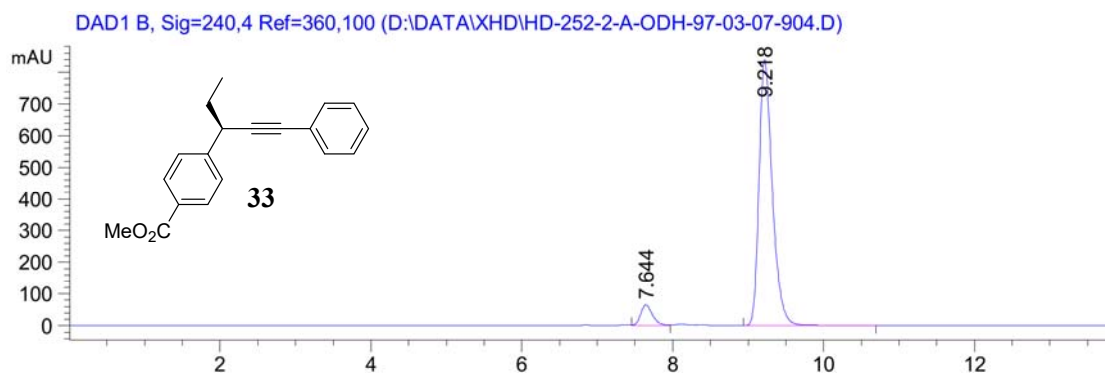
Totals : 3747.16313 374.04505



Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.716	VB	0.1437	4444.92188	464.44144	50.1005
2	9.289	VB	0.1843	4427.08203	362.96970	49.8995

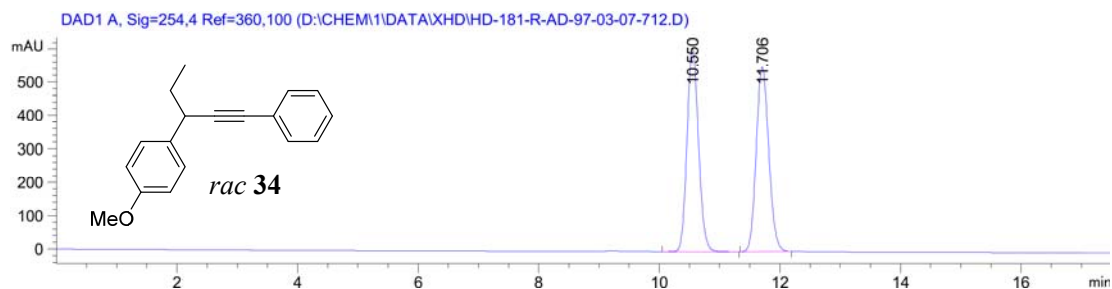
Totals : 8872.00391 827.41113



Signal 2: DAD1 B, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.644	VV	0.1639	700.30896	64.89249	6.3521
2	9.218	BB	0.1875	1.03246e4	839.47278	93.6479

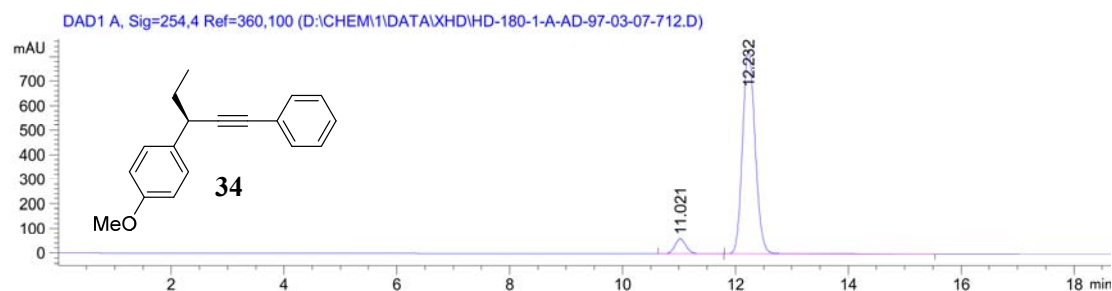
Totals : 1.10249e4 904.36527



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.550	BB	0.2061	8088.94043	612.23254	50.3510
2	11.706	BB	0.2243	7976.15820	552.92542	49.6490

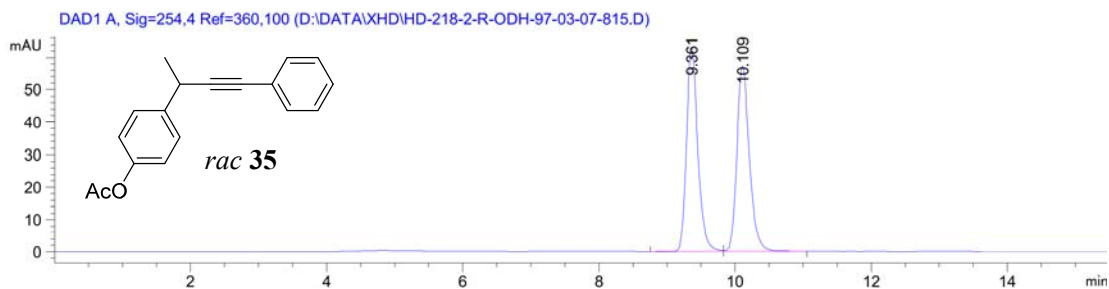
Totals : 1.60651e4 1165.15796



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.021	BB	0.2138	819.40558	59.82727	5.9997
2	12.232	BBA	0.2402	1.28379e4	831.09619	94.0003

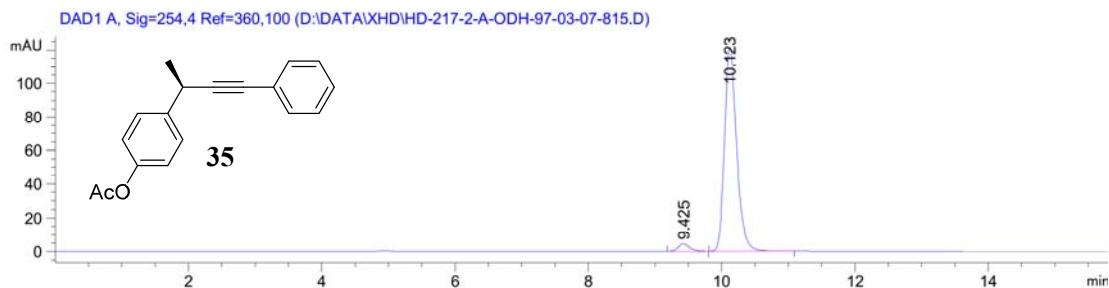
Totals : 1.36573e4 890.92346



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.361	BV	0.1748	731.42035	63.28177	49.9233
2	10.109	VB	0.1931	733.66797	57.40298	50.0767

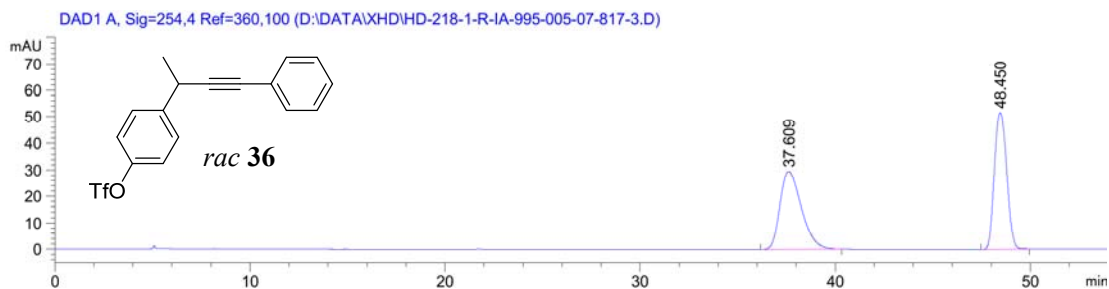
Totals : 1465.08832 120.68475



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.425	BB	0.1774	51.85070	4.46769	3.2652
2	10.123	BB	0.1914	1536.13745	121.59297	96.7348

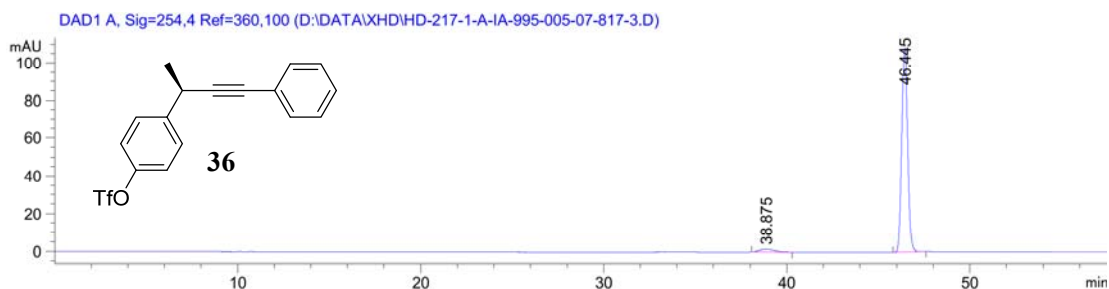
Totals : 1587.98815 126.06066



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	37.609	BB	1.1520	2264.83032	29.68773	49.9621
2	48.450	BB	0.7140	2268.26758	51.68632	50.0379

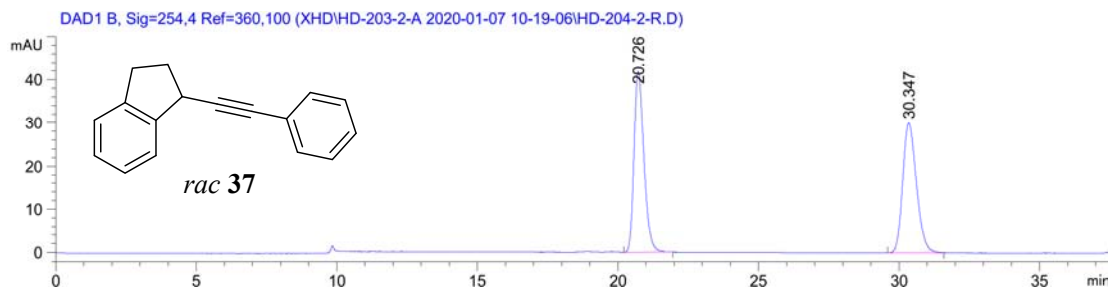
Totals : 4533.09790 81.37405



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.875	MM R	0.9846	90.04355	1.52427	3.5846
2	46.445	BB	0.3425	2421.88379	108.45319	96.4154

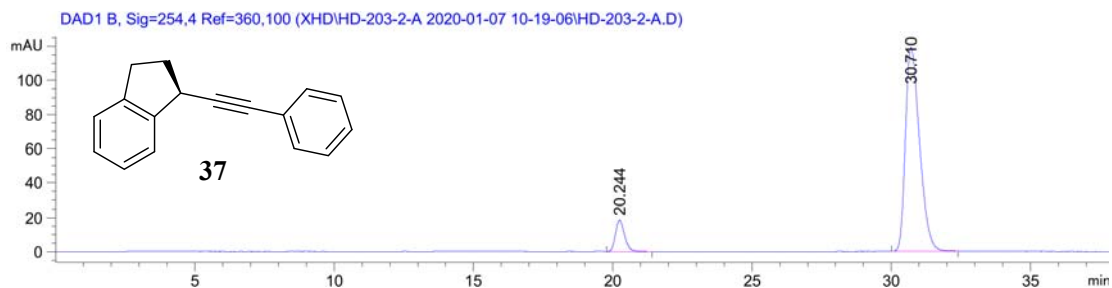
Totals : 2511.92734 109.97746



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.726	BB	0.3752	1020.36340	41.73885	50.1393
2	30.347	BB	0.5189	1014.69220	29.97383	49.8607

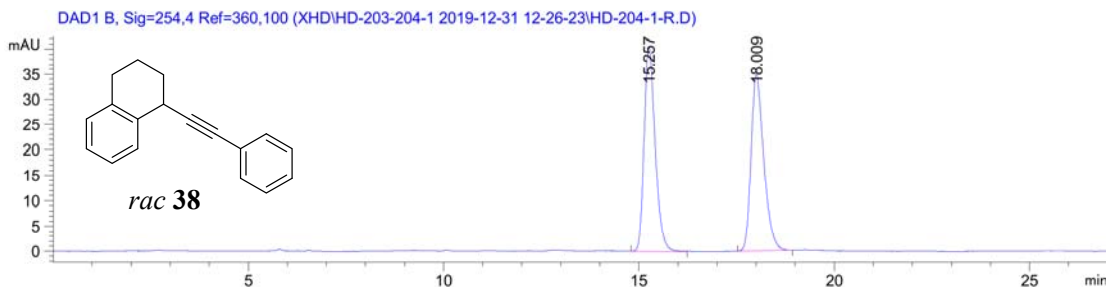
Totals : 2035.05560 71.71268



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.244	BB	0.3521	423.63586	18.57229	9.1671
2	30.710	BB	0.5344	4197.63770	119.32577	90.8329

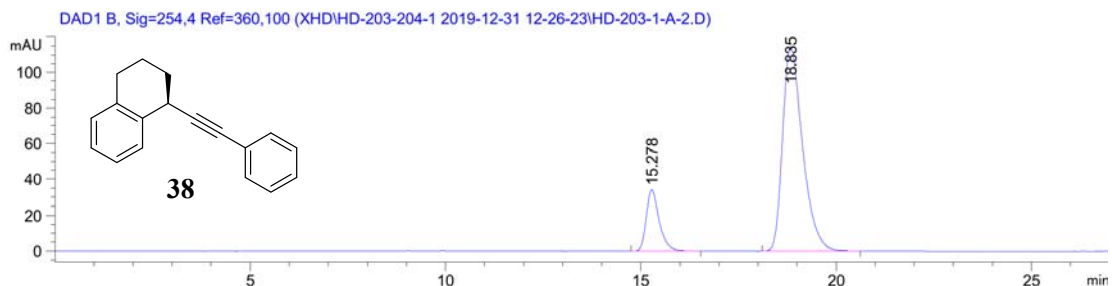
Totals : 4621.27356 137.89806



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.257	BB	0.3012	797.18793	40.58042	50.1597
2	18.009	BB	0.3380	792.11139	35.00698	49.8403

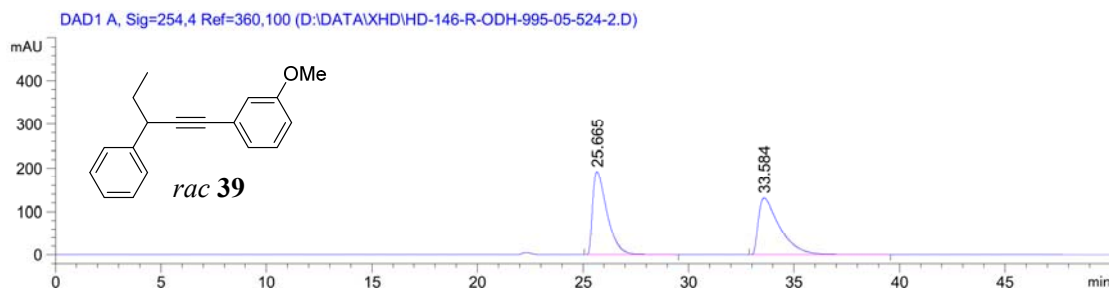
Totals : 1589.29932 75.58741



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.278	BB	0.3535	795.41681	34.17417	16.3506
2	18.835	BB	0.5384	4069.35205	114.57291	83.6494

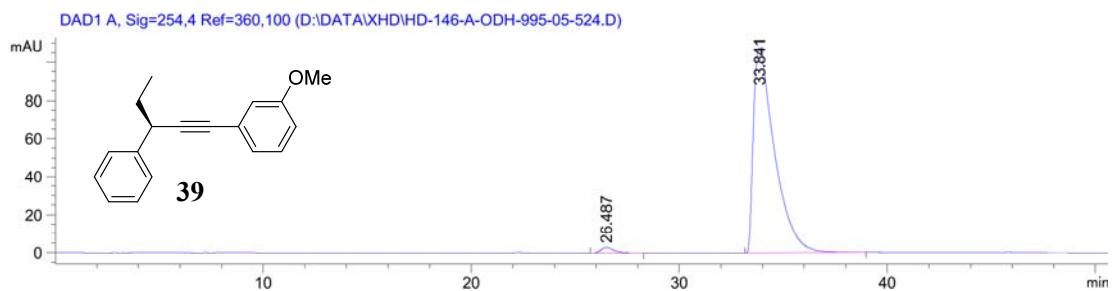
Totals : 4864.76886 148.74707



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.665	BB	0.7183	9137.25488	191.62433	49.7523
2	33.584	BB	1.0424	9228.22461	130.27808	50.2477

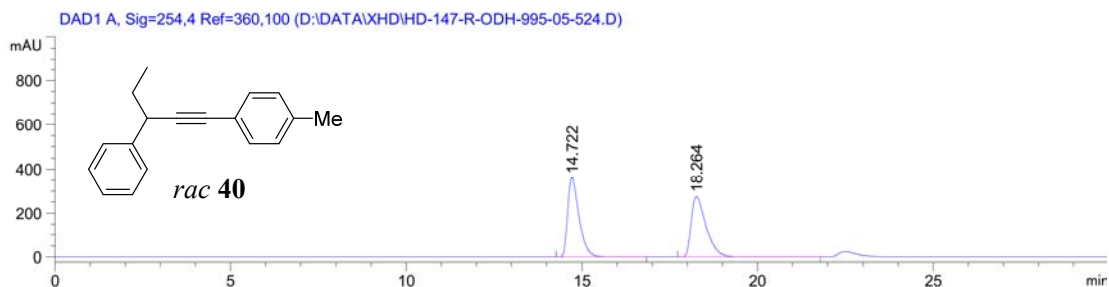
Totals : 1.83655e4 321.90240



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.487	BB	0.6300	124.05807	2.96561	1.6060
2	33.841	BB	1.0388	7600.55029	107.50506	98.3940

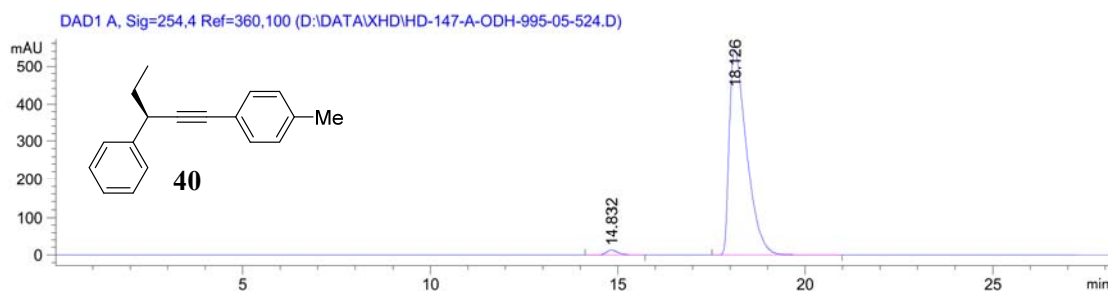
Totals : 7724.60837 110.47067



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.722	BV	0.3421	8105.09619	360.78421	49.9754
2	18.264	BB	0.4475	8113.06445	275.57626	50.0246

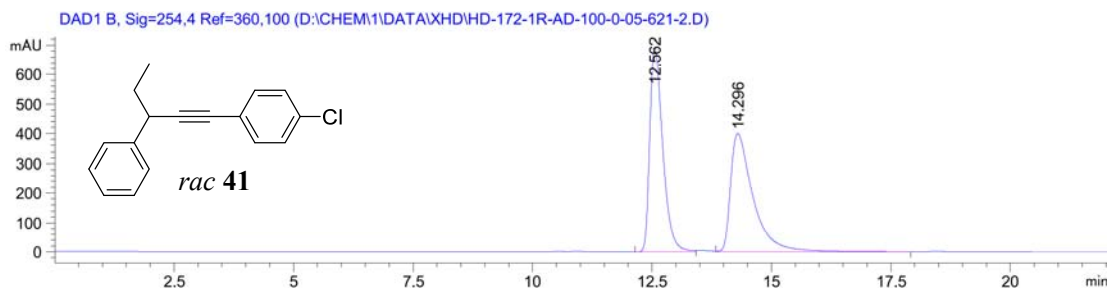
Totals : 1.62182e4 636.36047



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.832	BV	0.3298	274.93457	12.84259	1.5575
2	18.126	VV	0.4852	1.73777e4	543.23956	98.4425

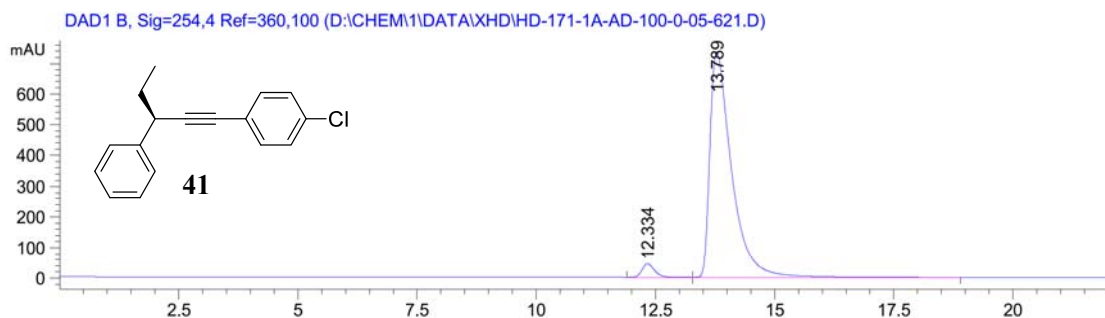
Totals : 1.76527e4 556.08215



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.562	BV	0.2882	1.30684e4	692.28748	49.7751
2	14.296	VB	0.4901	1.31865e4	398.49579	50.2249

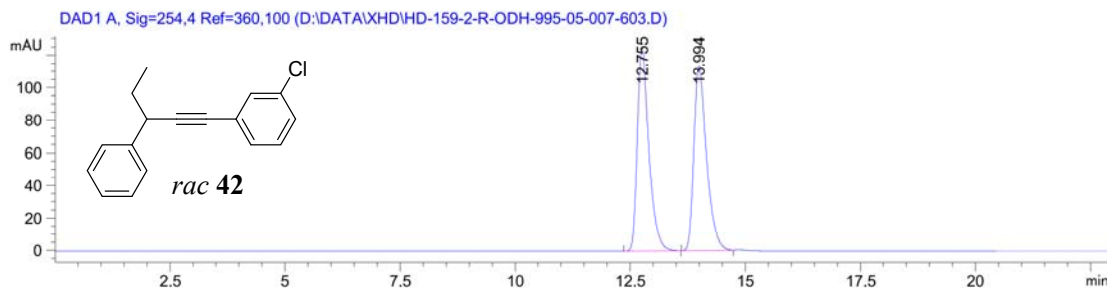
Totals : 2.62548e4 1090.78326



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.334	BB	0.2757	805.06354	44.36243	3.3349
2	13.789	BB	0.4736	2.33356e4	736.52789	96.6651

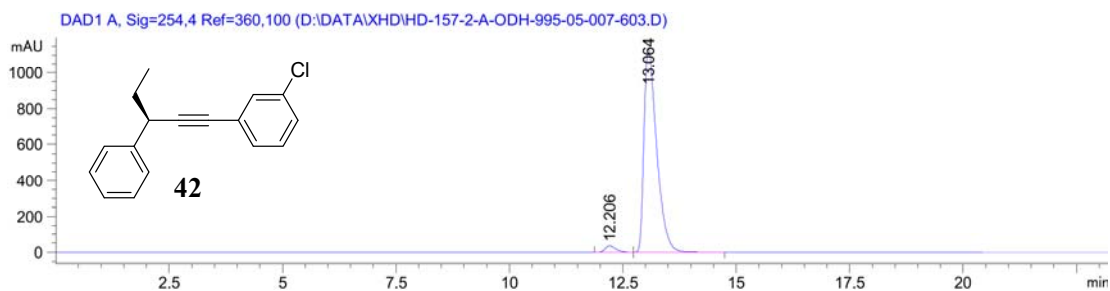
Totals : 2.41406e4 780.89032



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.755	BB	0.2609	2181.30762	125.39645	50.0811
2	13.994	BB	0.2874	2174.23975	113.54329	49.9189

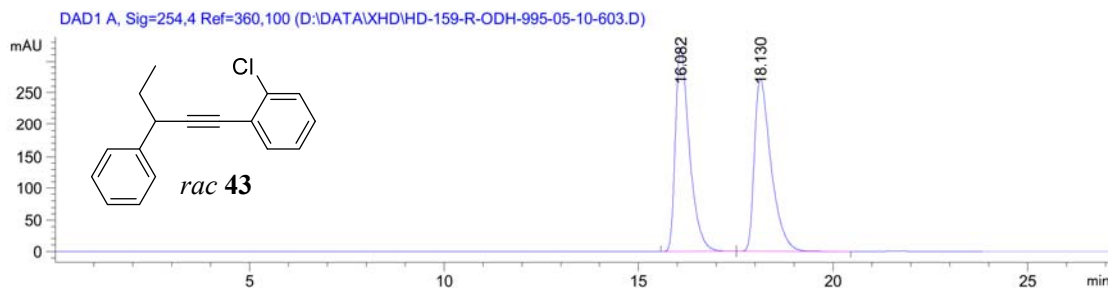
Totals : 4355.54736 238.93974



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.206	BV	0.2417	581.13727	36.12967	2.5660
2	13.064	VB	0.2920	2.20667e4	1139.16858	97.4340

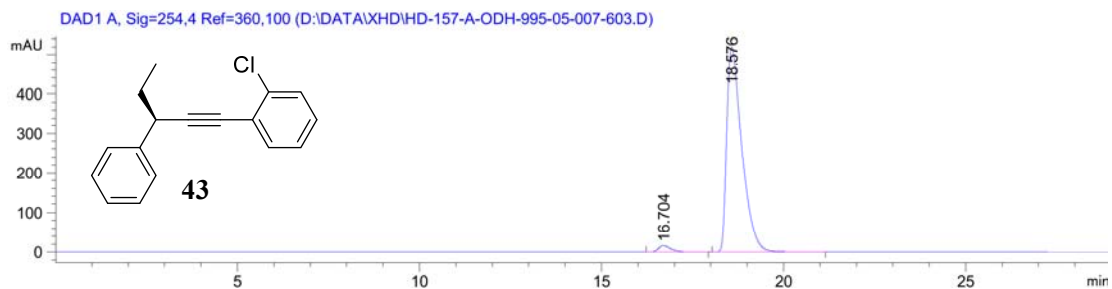
Totals : 2.26479e4 1175.29825



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.082	BB	0.3773	8091.87744	321.83011	49.9608
2	18.130	BB	0.4462	8104.58545	271.57565	50.0392

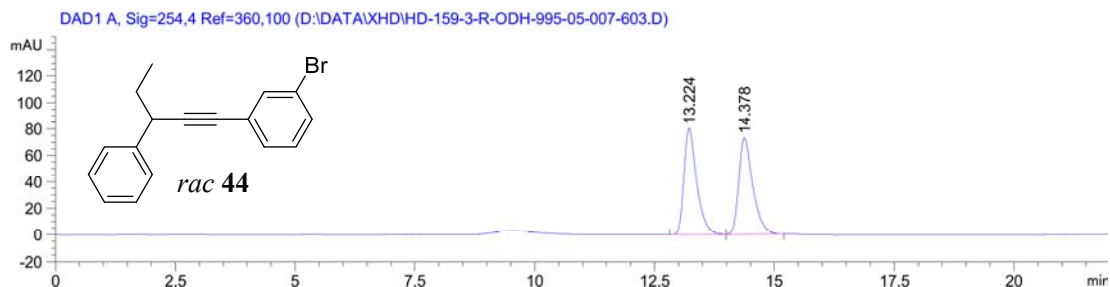
Totals : 1.61965e4 593.40576



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.704	BB	0.3408	378.40775	16.54974	2.4547
2	18.576	BB	0.4354	1.50371e4	519.91754	97.5453

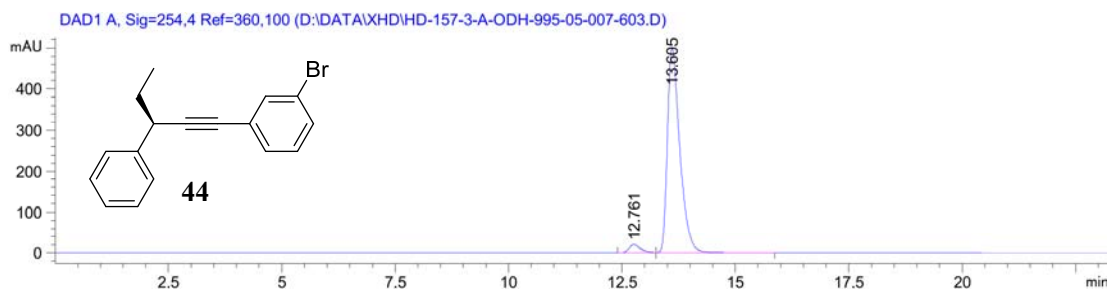
Totals : 1.54155e4 536.46728



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.224	BB	0.2751	1480.46533	80.30376	50.0093
2	14.378	BB	0.3028	1479.91309	72.89776	49.9907

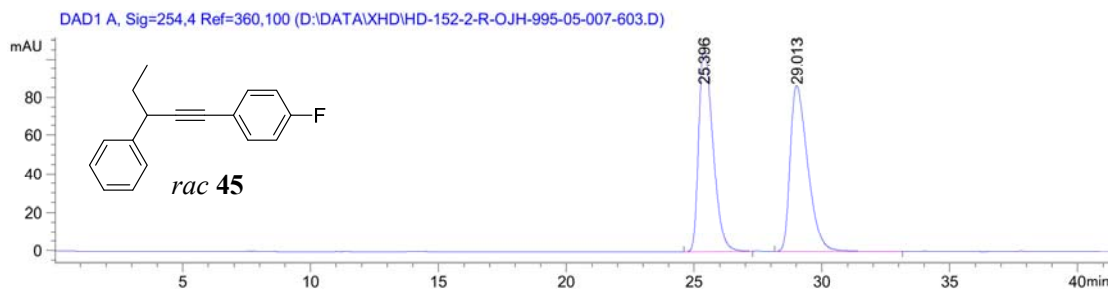
Totals : 2960.37842 153.20152



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.761	BV	0.2549	353.86914	20.75723	3.5725
2	13.605	VB	0.2869	9551.40332	499.84506	96.4275

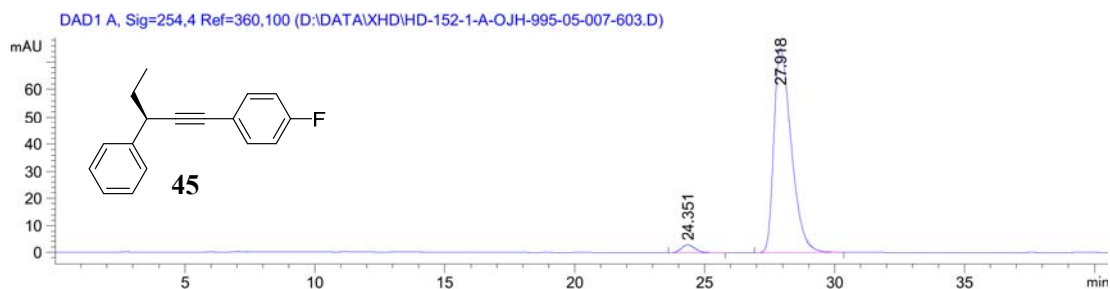
Totals : 9905.27246 520.60229



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.396	BB	0.5959	4111.97021	106.62293	49.8259
2	29.013	BB	0.7346	4140.70850	86.47428	50.1741

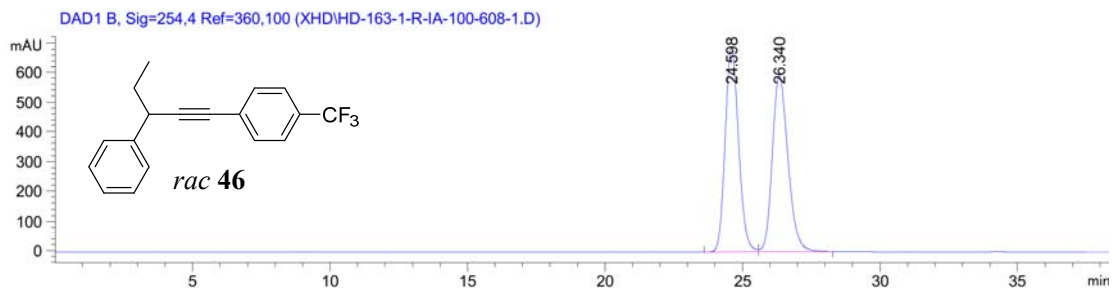
Totals : 8252.67871 193.09721



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.351	BB	0.5467	107.34194	2.93507	2.9629
2	27.918	BB	0.7145	3515.57813	75.32643	97.0371

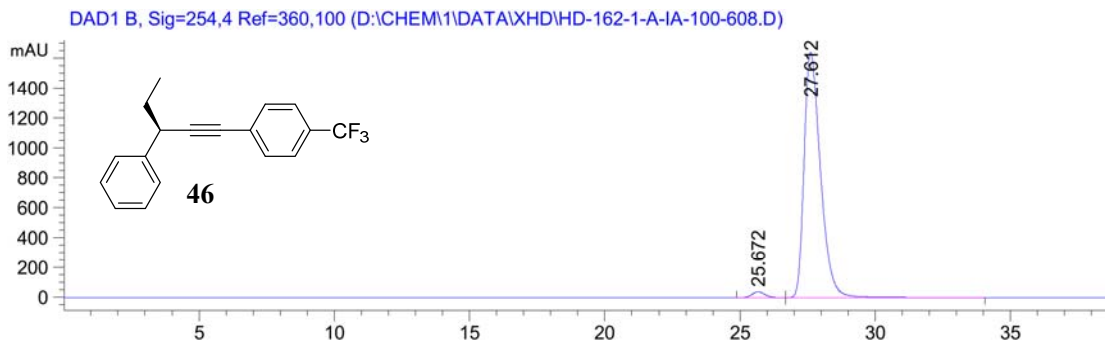
Totals : 3622.92007 78.26150



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.598	BV	0.5340	2.34461e4	690.73157	49.9323
2	26.340	VB	0.6091	2.35096e4	592.21149	50.0677

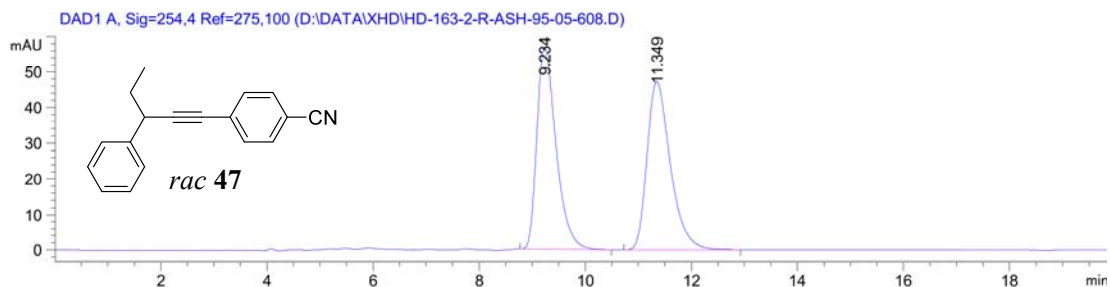
Totals : 4.69556e4 1282.94305



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.672	BV	0.5313	1348.76318	39.40878	1.9008
2	27.612	VV	0.6464	6.96081e4	1642.43896	98.0992

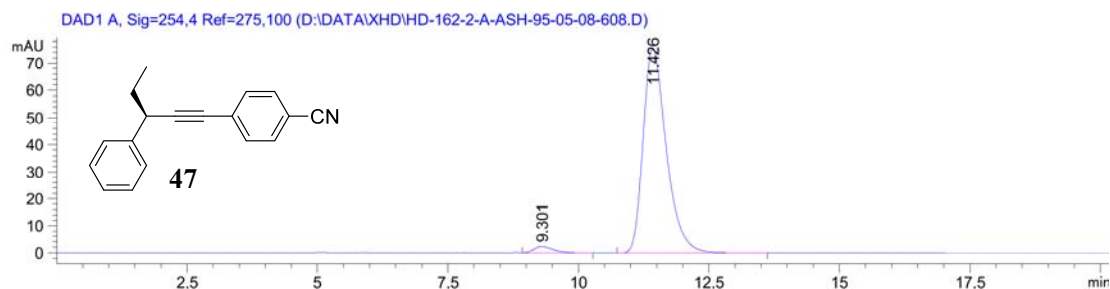
Totals : 7.09568e4 1681.84774



Signal 1: DAD1 A, Sig=254,4 Ref=275,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.234	BB	0.3826	1428.07458	56.93953	49.6868
2	11.349	BB	0.4671	1446.07800	47.25047	50.3132

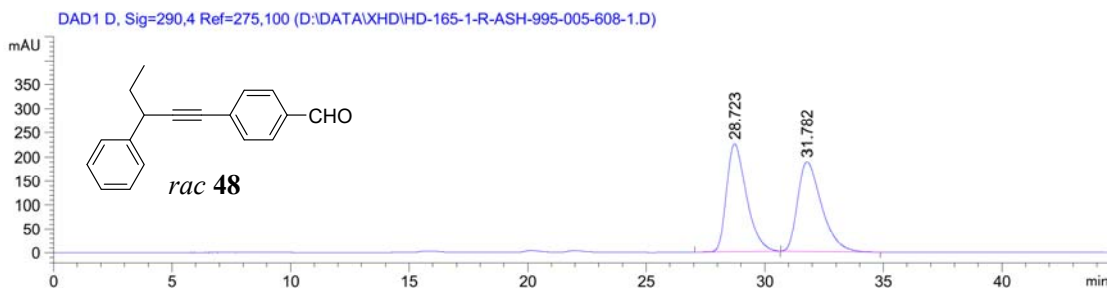
Totals : 2874.15259 104.18999



Signal 1: DAD1 A, Sig=254,4 Ref=275,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.301	BB	0.3709	57.14683	2.32362	2.3841
2	11.426	BB	0.4722	2339.80542	75.78040	97.6159

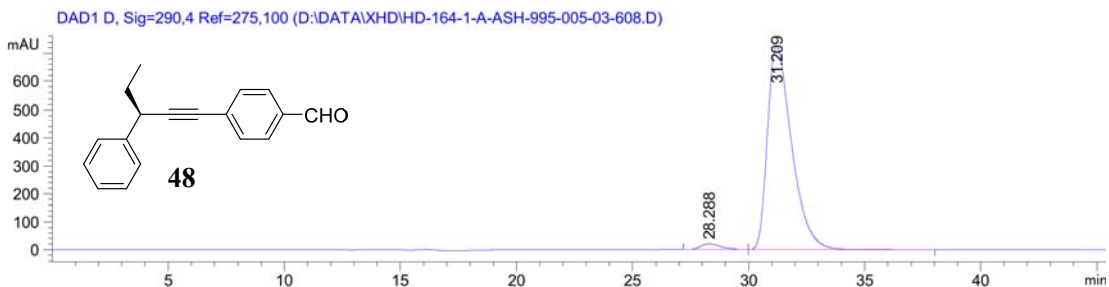
Totals : 2396.95225 78.10402



Signal 4: DAD1 D, Sig=290,4 Ref=275,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.723	BB	0.9040	1.31933e4	224.38988	50.0027
2	31.782	BB	1.0481	1.31918e4	187.66449	49.9973

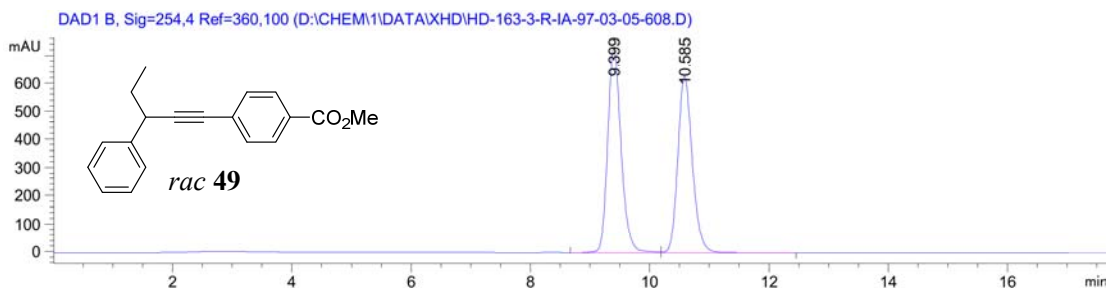
Totals : 2.63851e4 412.05437



Signal 4: DAD1 D, Sig=290,4 Ref=275,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.288	BB	0.8976	1208.90747	20.45207	2.2478
2	31.209	BB	1.0997	5.25739e4	725.23651	97.7522

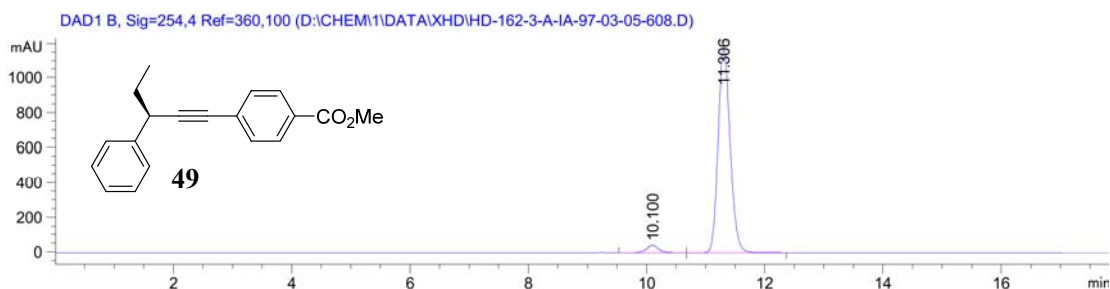
Totals : 5.37828e4 745.68858



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.399	BV	0.2430	1.14698e4	731.35083	51.8653
2	10.585	VB	0.2618	1.06448e4	627.75586	48.1347

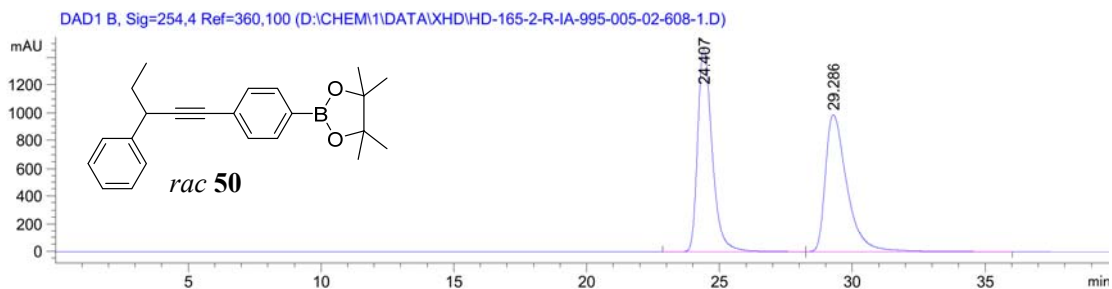
Totals : 2.21147e4 1359.10669



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.100	BB	0.2182	604.00775	42.40234	3.5015
2	11.306	BV	0.2215	1.66459e4	1173.22266	96.4985

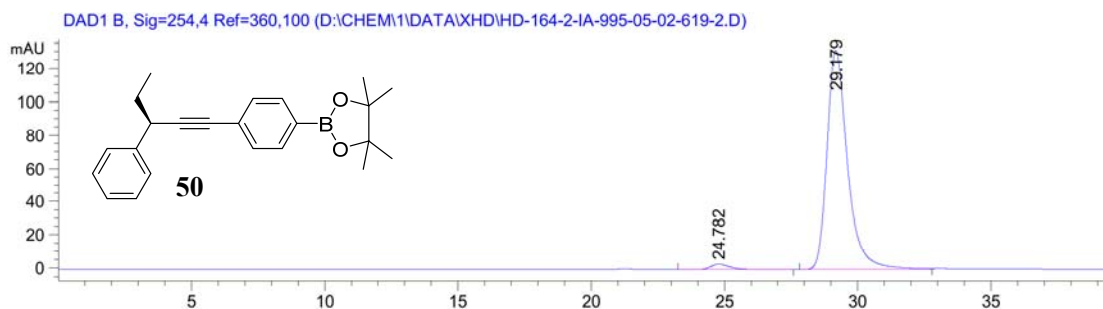
Totals : 1.72499e4 1215.62500



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.407	BB	0.5772	5.56960e4	1472.78284	49.9380
2	29.286	BV	0.8541	5.58343e4	989.84442	50.0620

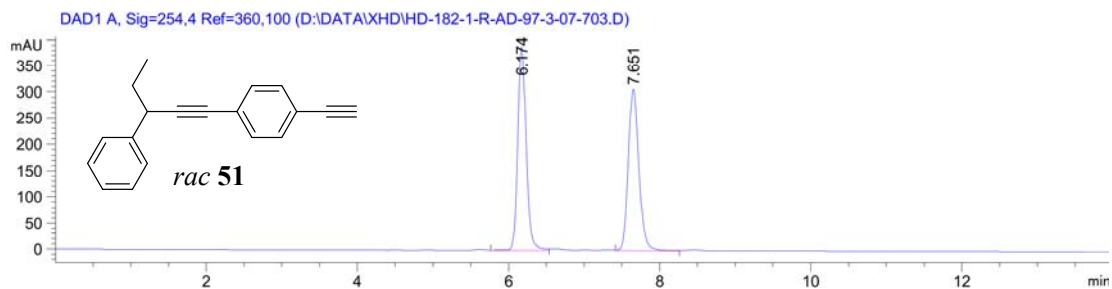
Totals : 1.11530e5 2462.62726



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.782	BB	0.7185	154.94252	3.19102	2.1503
2	29.179	BV	0.8095	7050.64648	131.50574	97.8497

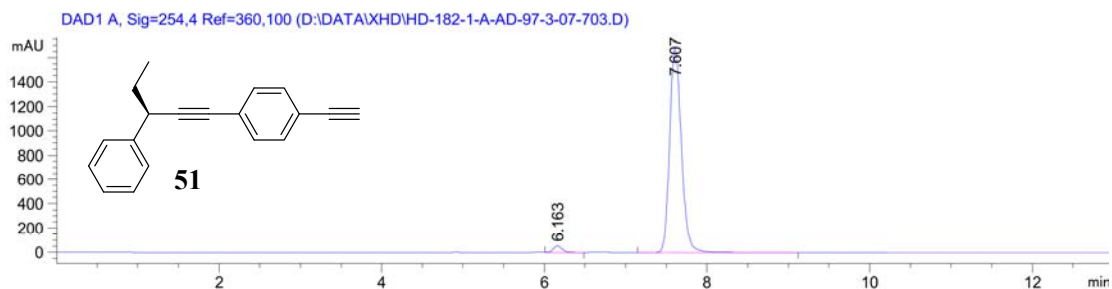
Totals : 7205.58900 134.69676



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.174	BV	0.1192	3041.91431	389.12375	50.1668
2	7.651	VV	0.1505	3021.68555	307.96439	49.8332

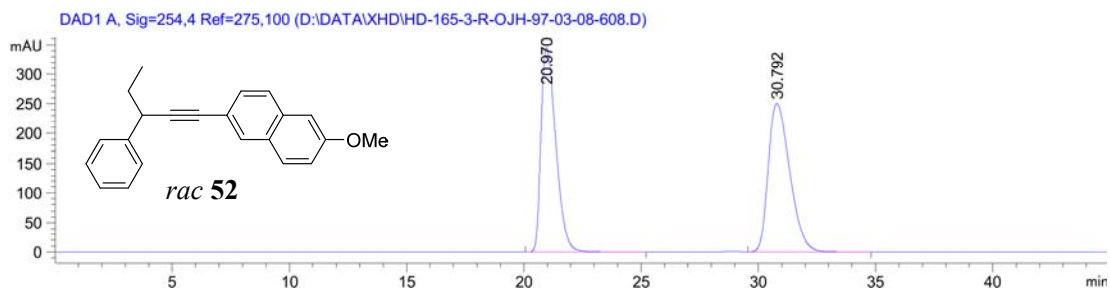
Totals : 6063.59985 697.08813



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.163	VV	0.1215	414.41406	52.85158	2.3954
2	7.607	BB	0.1549	1.68861e4	1685.51697	97.6046

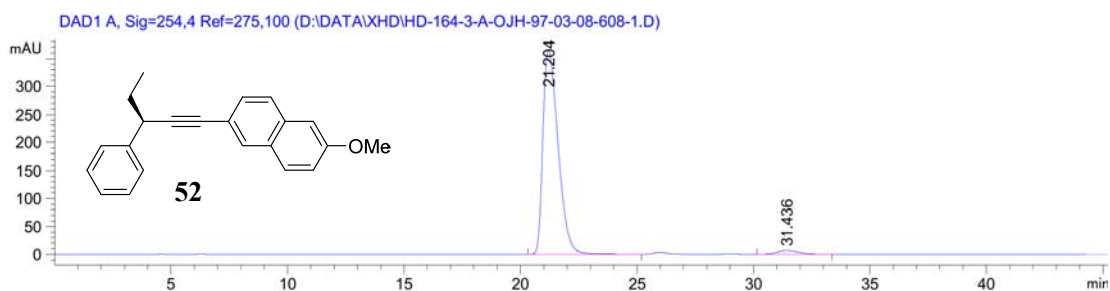
Totals : 1.73006e4 1738.36855



Signal 1: DAD1 A, Sig=254,4 Ref=275,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.970	BB	0.6864	1.51653e4	345.27274	48.6586
2	30.792	BB	0.9993	1.60014e4	250.49669	51.3414

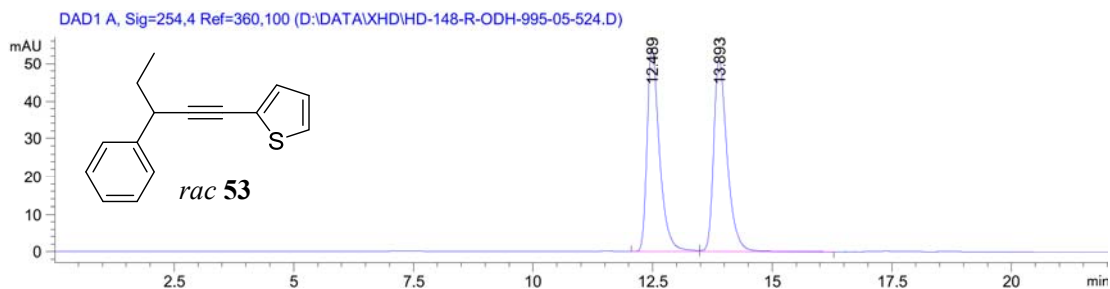
Totals : 3.11667e4 595.76942



Signal 1: DAD1 A, Sig=254,4 Ref=275,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.204	BB	0.7072	1.65025e4	365.19394	97.6171
2	31.436	BB	0.8765	402.83447	6.72829	2.3829

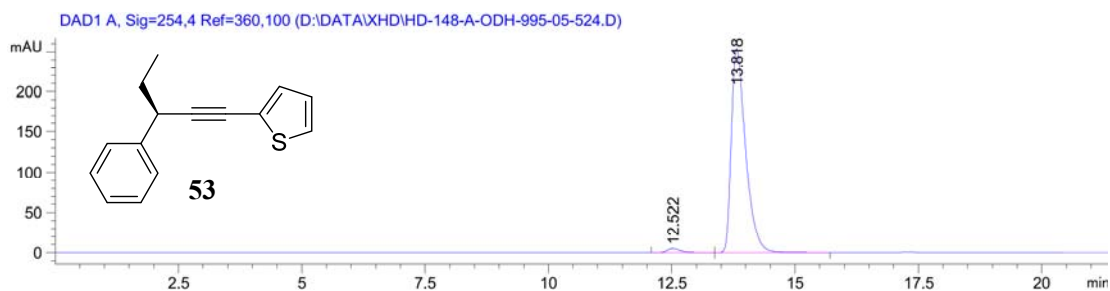
Totals : 1.69054e4 371.92223



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.489	VV	0.2757	980.67621	54.04567	49.3770
2	13.893	VB	0.2937	1005.42493	51.07379	50.6230

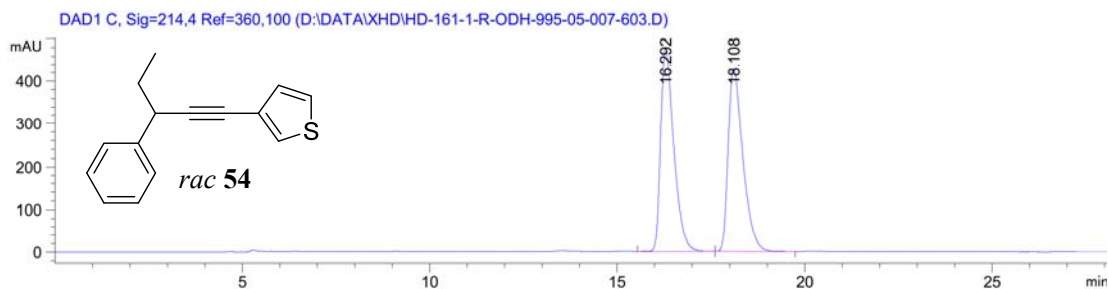
Totals : 1986.10114 105.11945



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.522	BV	0.2724	93.59621	5.04616	1.7931
2	13.818	VV	0.3081	5126.13574	253.28737	98.2069

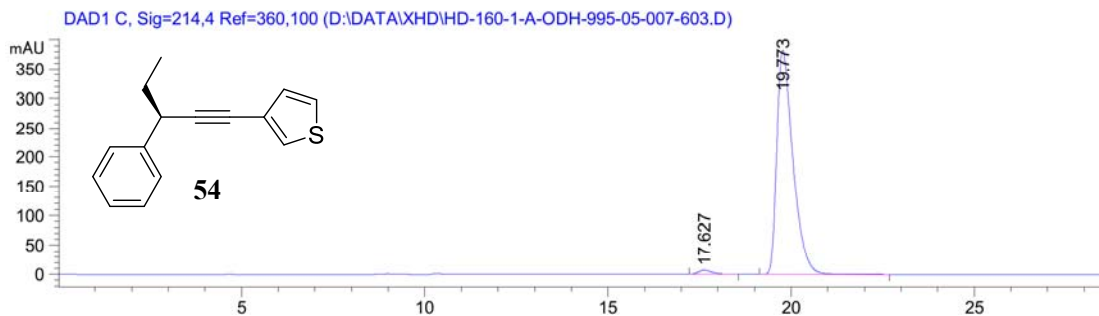
Totals : 5219.73196 258.33353



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.292	BB	0.3664	1.16311e4	477.15085	49.9073
2	18.108	BB	0.4143	1.16744e4	427.85065	50.0927

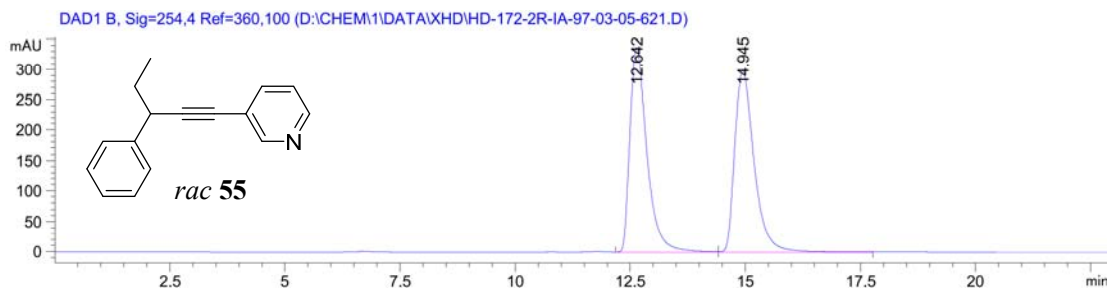
Totals : 2.33055e4 905.00150



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.627	BB	0.3775	169.18300	6.81889	1.4247
2	19.773	BB	0.4608	1.17062e4	382.78134	98.5753

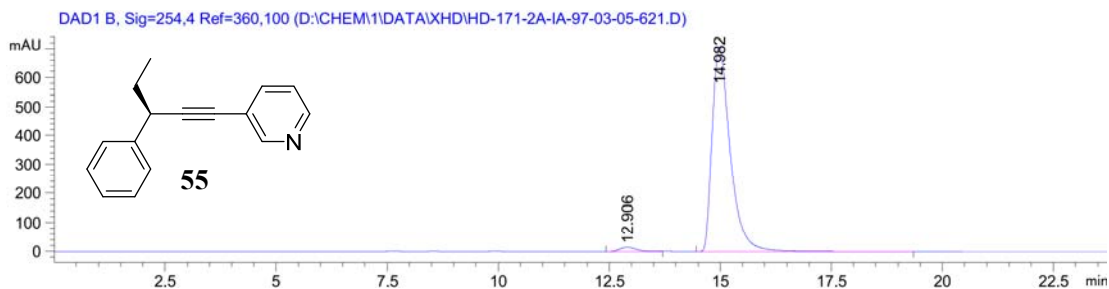
Totals : 1.18754e4 389.60023



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.642	BV	0.3935	8731.62305	337.82025	49.6548
2	14.945	VV	0.4568	8853.02148	299.53043	50.3452

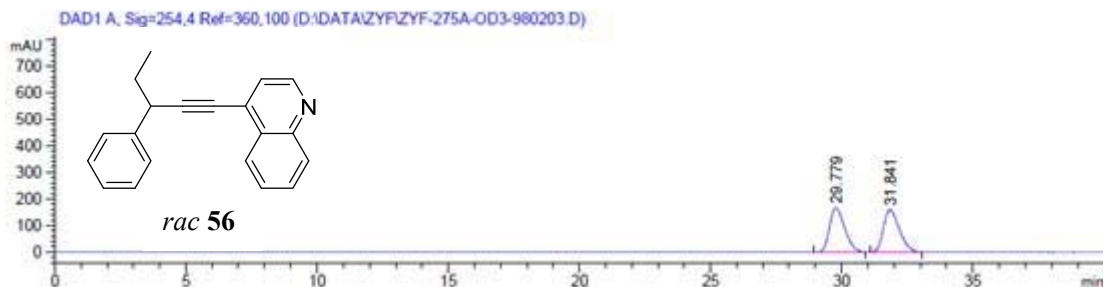
Totals : 1.75846e4 637.35068



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.906	BV	0.4175	406.92044	14.95312	1.9745
2	14.982	VV	0.4365	2.02020e4	708.83459	98.0255

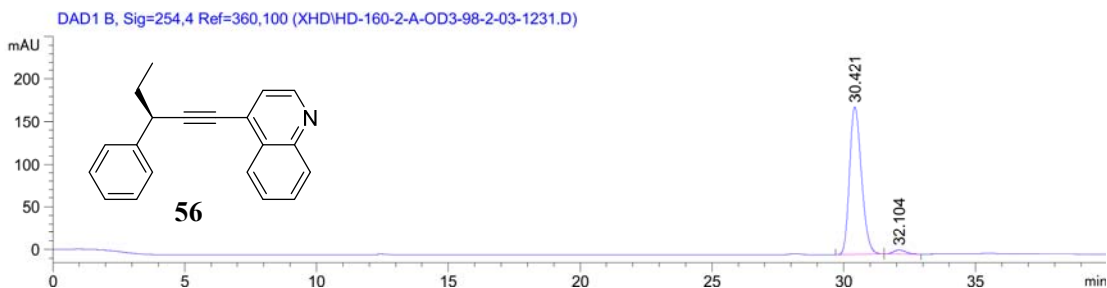
Totals : 2.06089e4 723.78771



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.779	BB	0.5944	6689.38721	167.34102	50.1421
2	31.841	BB	0.6395	6651.47559	158.54236	49.8579

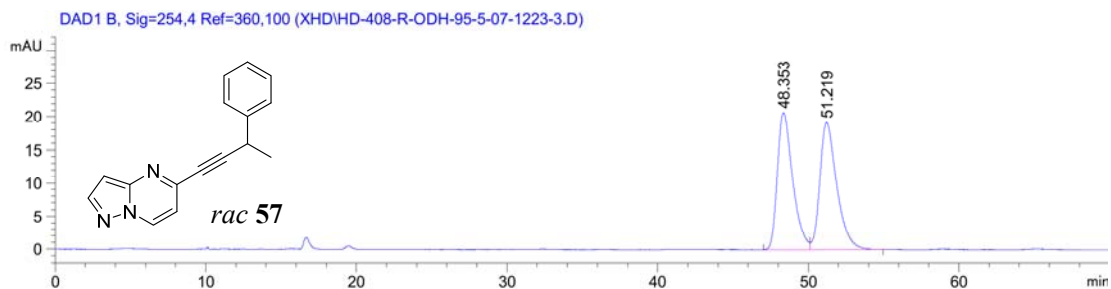
Totals : 1.33409e4 325.88338



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

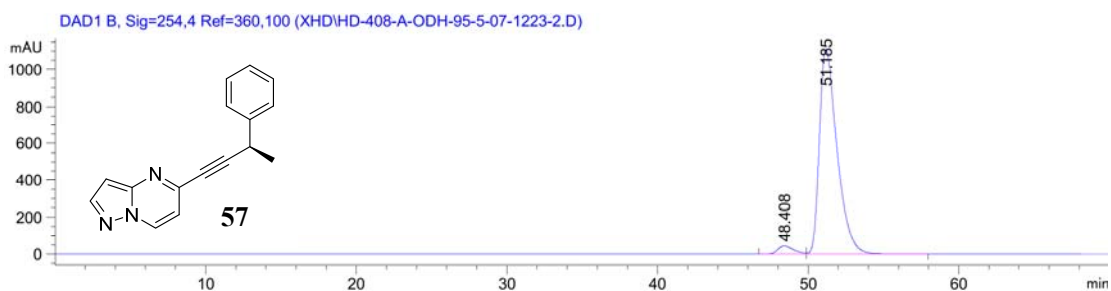
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.421	BB	0.4894	5460.33740	173.40587	97.3636
2	32.104	BB	0.4371	147.85175	4.67355	2.6364

Totals : 5608.18915 178.07942



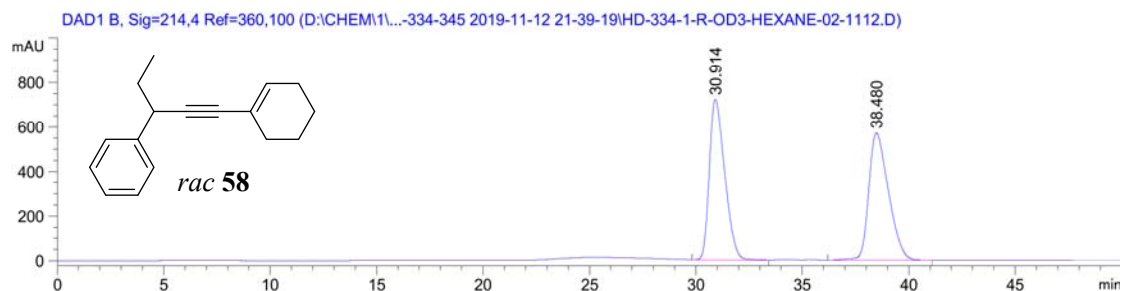
Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.353	BV	1.0605	1472.47644	20.73572	49.3653
2	51.219	VB	1.1745	1510.34290	19.18501	50.6347
Totals :				2982.81934	39.92073	



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

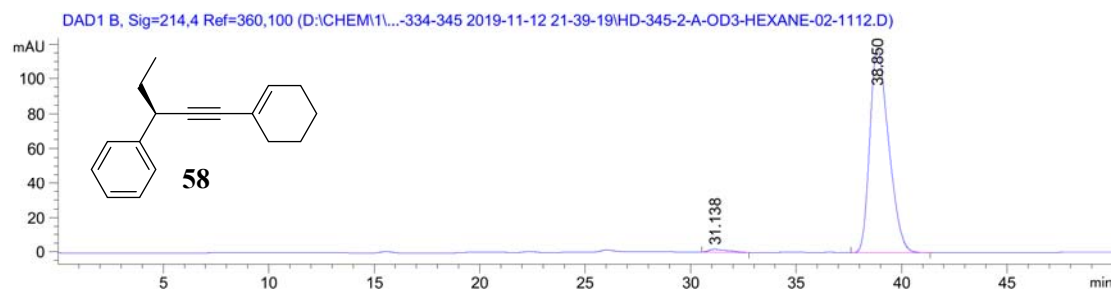
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	48.408	BV	1.0715	3143.25708	44.10449	3.3642
2	51.185	VB	1.2200	9.02886e4	1111.11963	96.6358
Totals :				9.34318e4	1155.22412	



Signal 2: DAD1 B, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.914	BB	0.7602	3.59539e4	718.05603	49.5527
2	38.480	BB	0.9762	3.66029e4	569.24237	50.4473

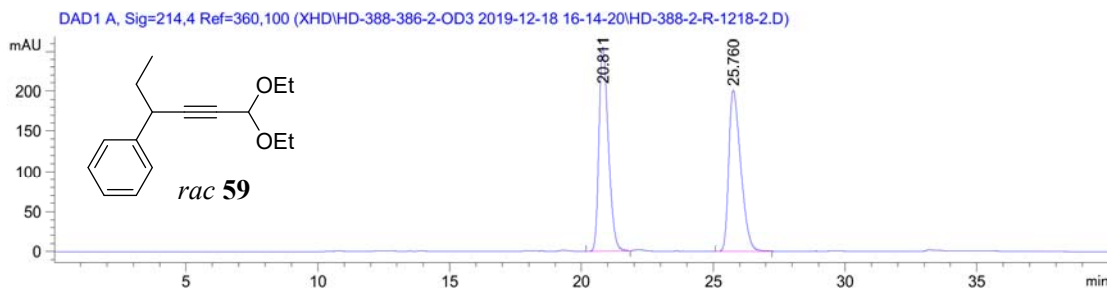
Totals : 7.25568e4 1287.29840



Signal 2: DAD1 B, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	31.138	MM R	1.0957	113.01952	1.71913	1.5175
2	38.850	BB	0.9452	7334.76318	117.99655	98.4825

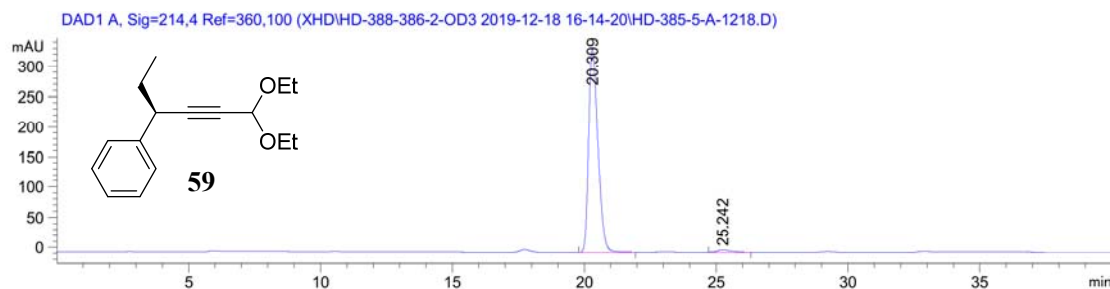
Totals : 7447.78270 119.71568



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.811	BB	0.3763	6306.20361	255.18086	49.9160
2	25.760	BB	0.4782	6327.41943	200.48511	50.0840

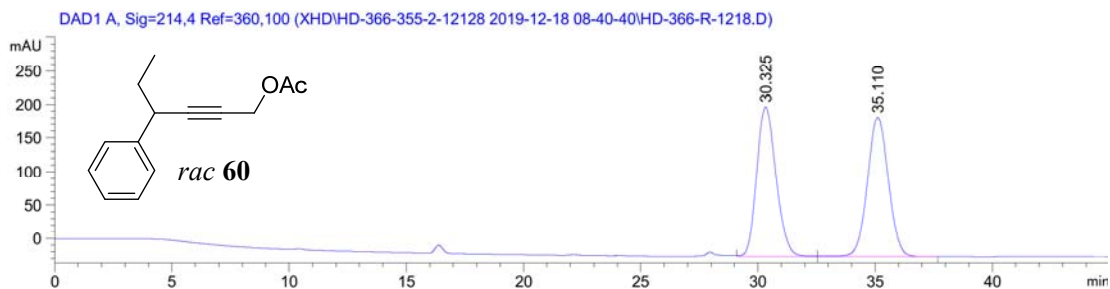
Totals : 1.26336e4 455.66597



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.309	BB	0.3757	8286.66211	338.37595	98.3438
2	25.242	BB	0.4244	139.55592	4.04602	1.6562

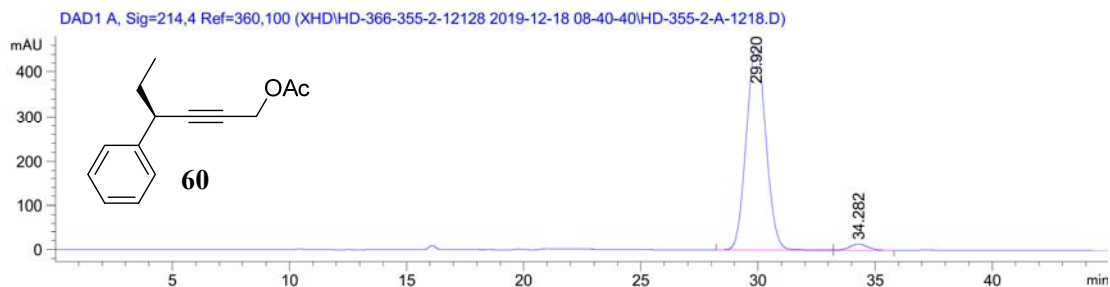
Totals : 8426.21803 342.42196



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.325	VV	0.8961	1.28594e4	223.21736	49.7953
2	35.110	VB	0.9605	1.29651e4	207.63132	50.2047

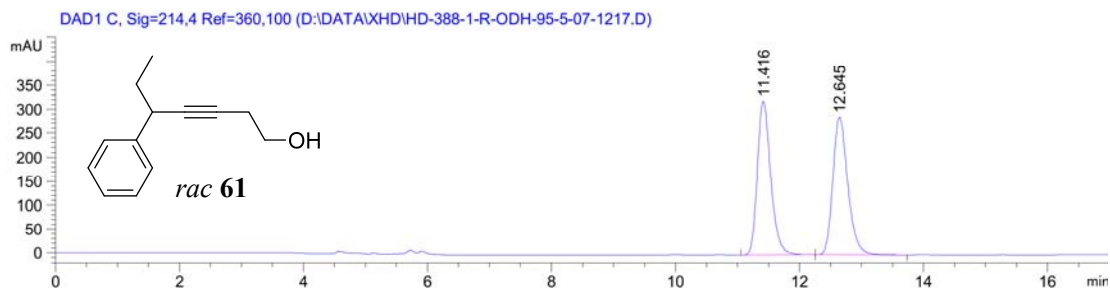
Totals : 2.58244e4 430.84868



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.920	BV	0.9021	2.66108e4	457.86212	97.2812
2	34.282	VB	0.8436	743.71826	13.65193	2.7188

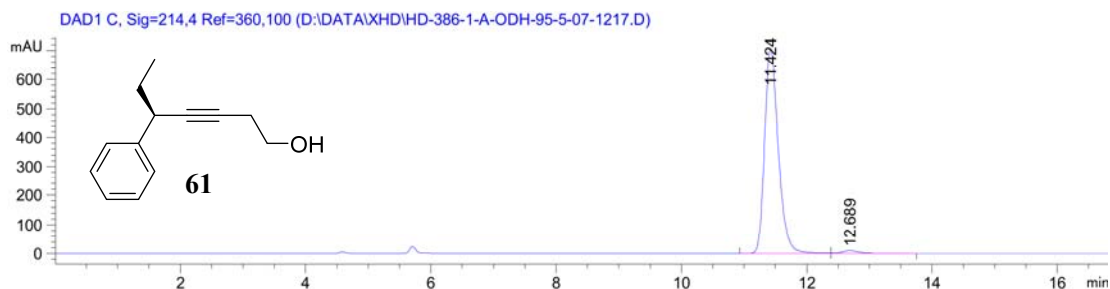
Totals : 2.73545e4 471.51405



Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.416	BB	0.2288	4828.20703	322.17624	49.8926
2	12.645	BV	0.2564	4849.00146	288.10391	50.1074

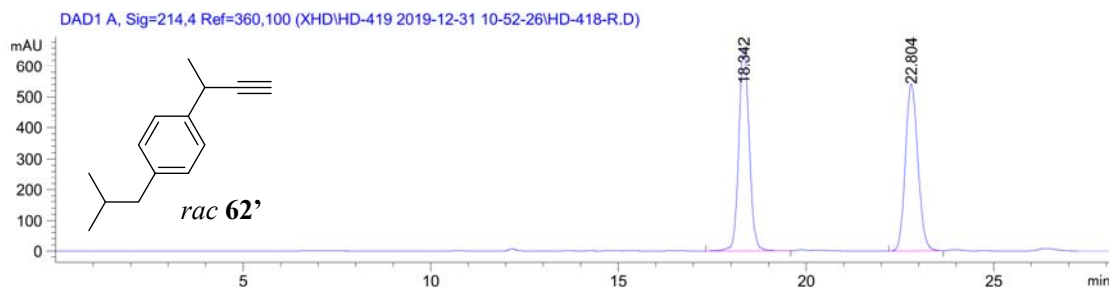
Totals : 9677.20850 610.28015



Signal 2: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.424	BV	0.2307	1.07228e4	708.12616	98.3463
2	12.689	VV	0.2840	180.30696	9.39141	1.6537

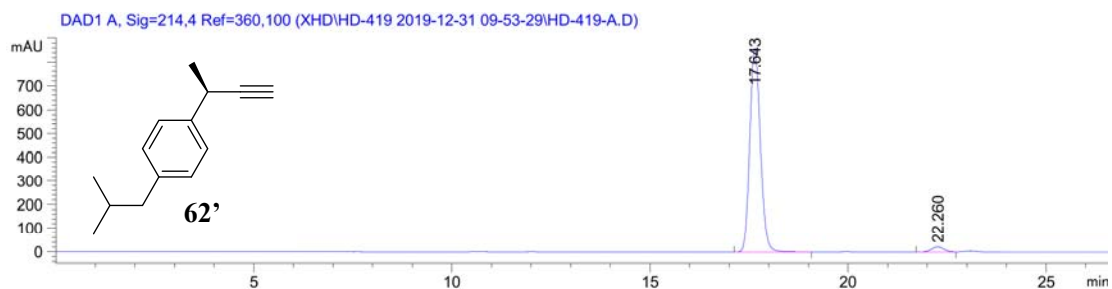
Totals : 1.09031e4 717.51757



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.342	BB	0.2998	1.28199e4	662.39044	50.5722
2	22.804	BB	0.3626	1.25298e4	540.27222	49.4278

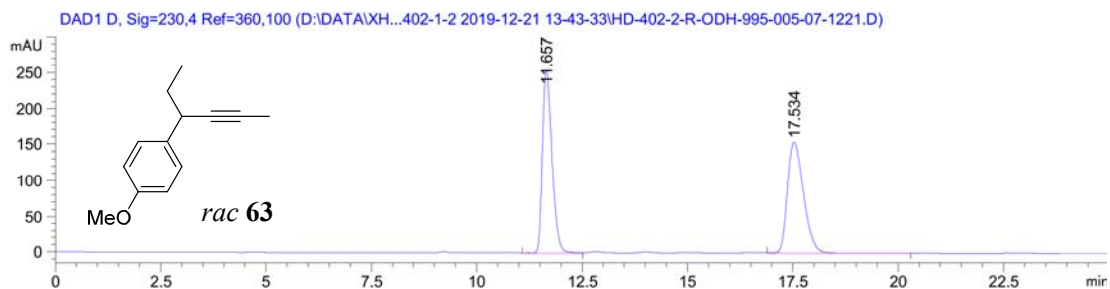
Totals : 2.53497e4 1202.66266



Signal 1: DAD1 A, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.643	BB	0.2896	1.60431e4	860.14917	97.1564
2	22.260	BB	0.3446	469.54657	21.51884	2.8436

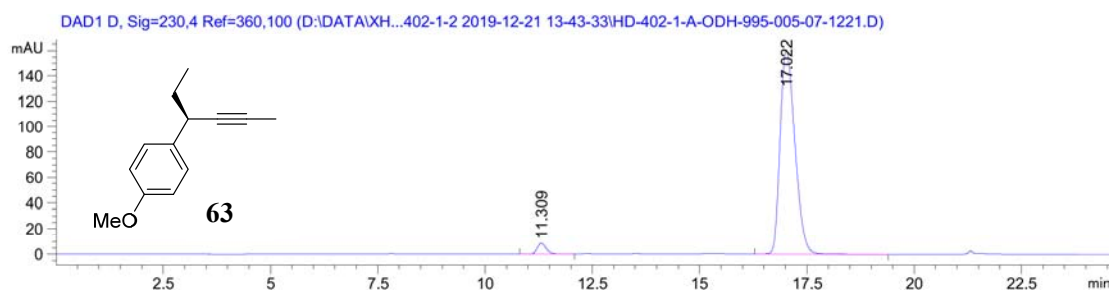
Totals : 1.65126e4 881.66801



Signal 3: DAD1 D, Sig=230,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.657	BV	0.2445	4020.3325	254.16200	49.3931
2	17.534	VB	0.4193	4119.12695	154.38954	50.6069

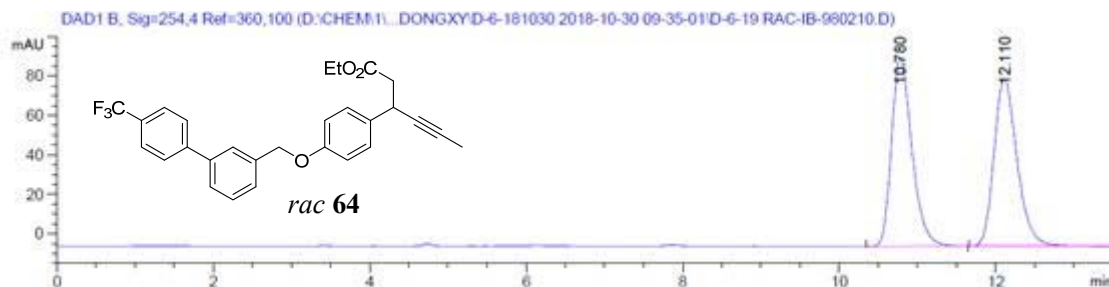
Totals : 8139.46021 408.55154



Signal 3: DAD1 D, Sig=230,4 Ref=360,100

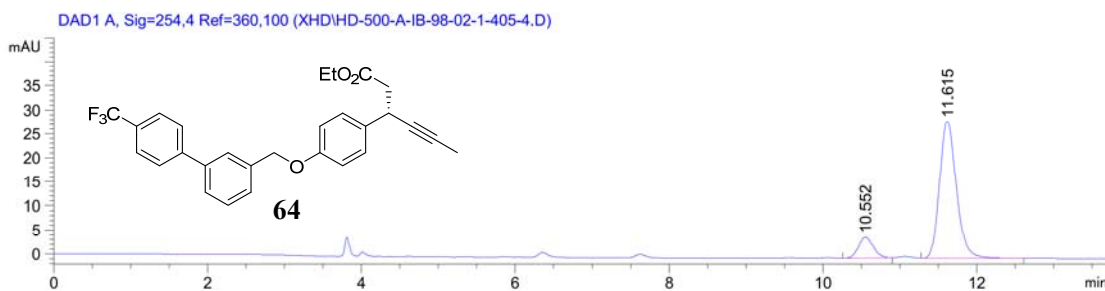
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.309	BV	0.2294	130.04753	8.74764	3.2647
2	17.022	VB	0.3756	3853.39160	160.80412	96.7353

Totals : 3983.43913 169.55177



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

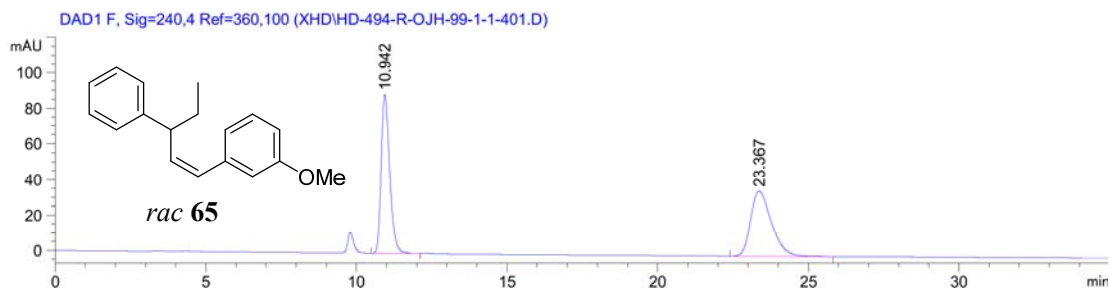
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.780	BB	0.2755	1732.62378	95.55478	49.8964
2	12.110	BB	0.3152	1739.81921	84.84905	50.1036



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.552	BB	0.2083	58.01698	4.33101	11.7734
2	11.615	BB	0.2333	434.76303	28.61264	88.2266

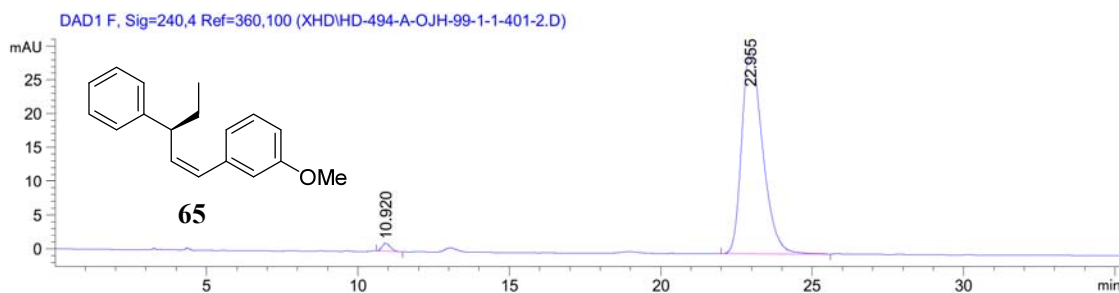
Totals : 492.78001 32.94365



Signal 6: DAD1 F, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.942	BB	0.3019	1755.87610	89.08886	49.8720
2	23.367	BB	0.7375	1764.88770	36.66800	50.1280

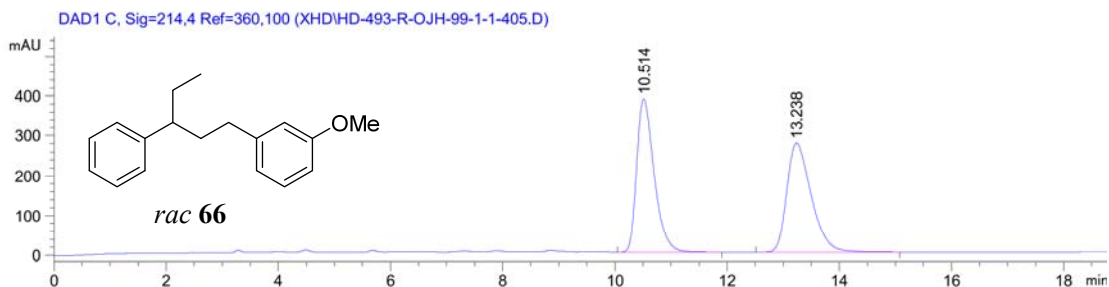
Totals : 3520.76379 125.75686



Signal 6: DAD1 F, Sig=240,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.920	BB	0.2947	21.54159	1.14911	1.4697
2	22.955	BB	0.7296	1444.14819	30.32329	98.5303

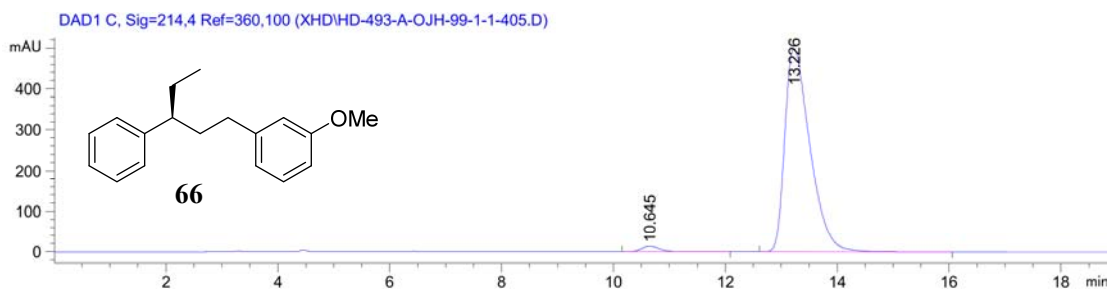
Totals : 1465.68979 31.47240



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.514	BB	0.3300	8320.85254	385.18195	50.0223
2	13.238	BB	0.4647	8313.43750	273.47012	49.9777

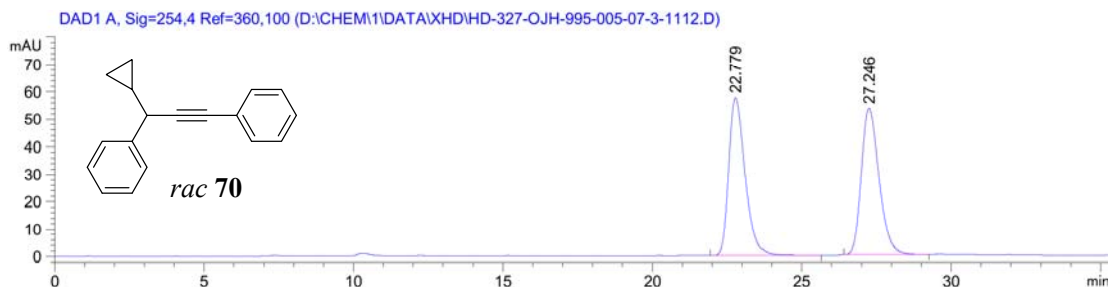
Totals : 1.66343e4 658.65207



Signal 3: DAD1 C, Sig=214,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.645	BB	0.3433	303.43787	13.54809	1.9046
2	13.226	BB	0.4765	1.56280e4	500.17468	98.0954

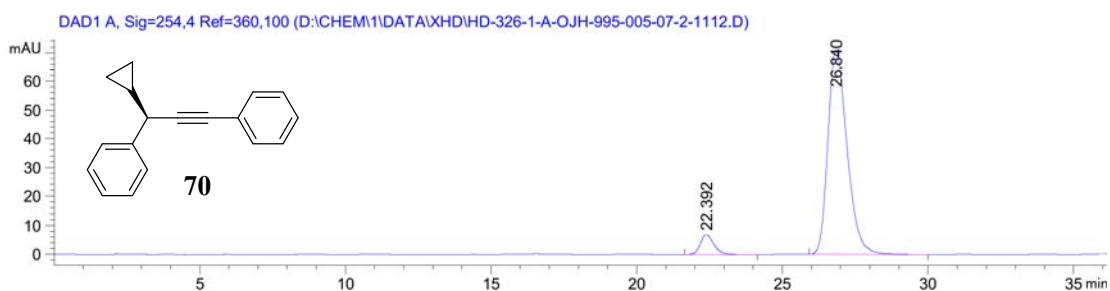
Totals : 1.59314e4 513.72277



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.779	BB	0.5804	2198.17407	57.70641	49.9465
2	27.246	BB	0.6348	2202.88062	53.44888	50.0535

Totals : 4401.05469 111.15529

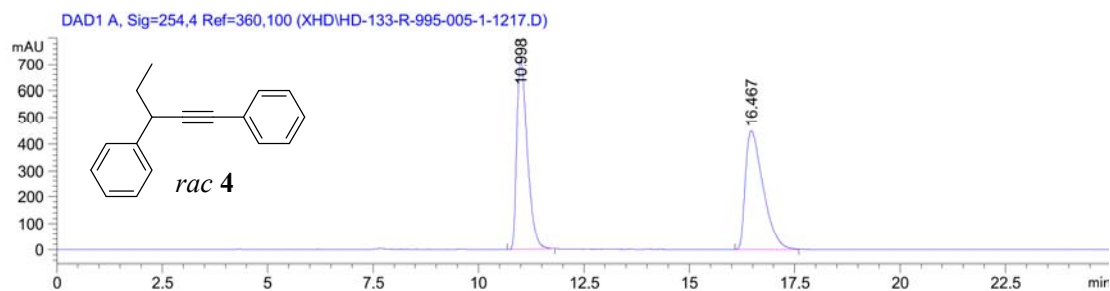


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.392	BB	0.4965	226.15912	6.97150	6.5256
2	26.840	BB	0.6994	3239.58228	71.13010	93.4744

Totals : 3465.74139 78.10159

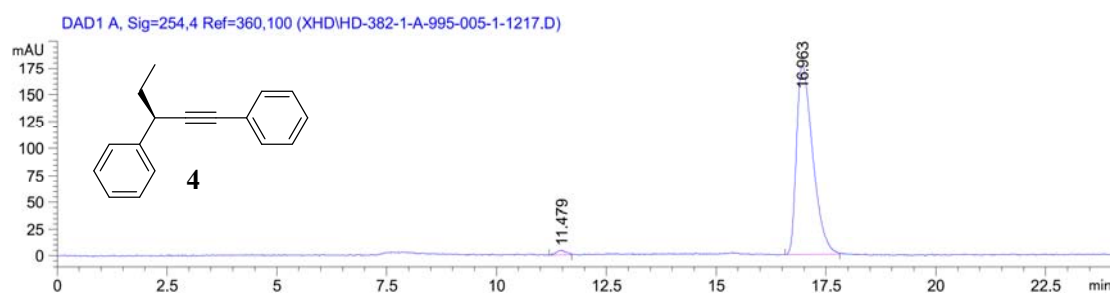
One-pot synthesis



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.998	VV	0.2766	1.25504e4	715.70178	48.7394
2	16.467	VV	0.4315	1.31996e4	448.29694	51.2606

Totals : 2.57501e4 1163.99872

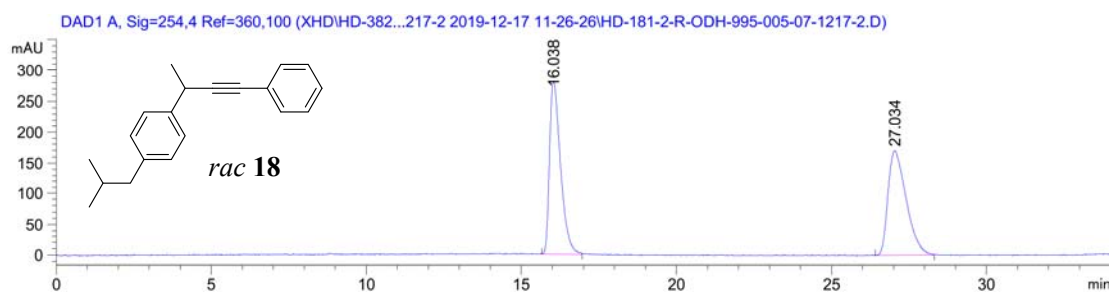


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.479	BV	0.1899	70.24247	4.50628	1.5123
2	16.963	BV	0.3832	4574.63037	177.19905	98.4877

Totals : 4644.87284 181.70533

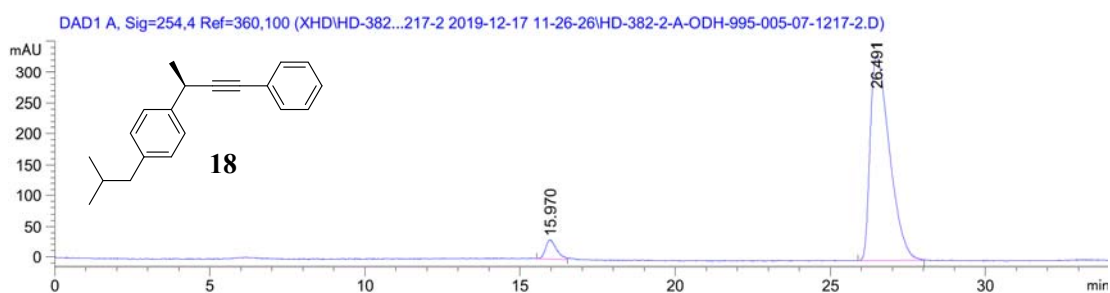
One-pot synthesis



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.038	VV	0.3759	6914.56250	280.15863	49.9724
2	27.034	VV	0.5605	6922.19775	169.48204	50.0276

Totals : 1.38368e4 449.64067

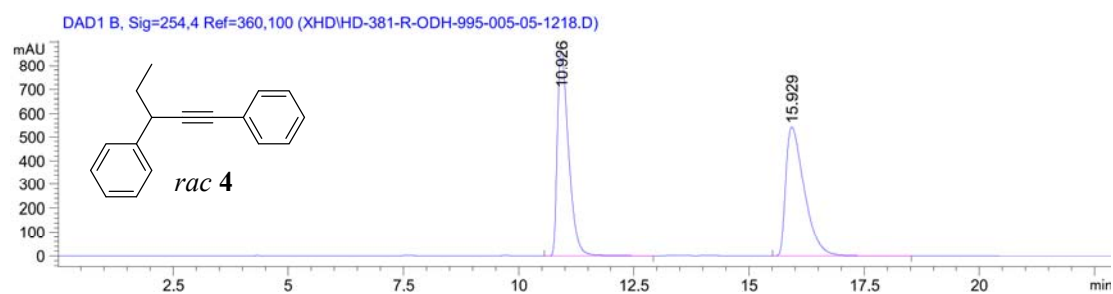


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.970	BV	0.3276	766.29358	30.86595	5.0577
2	26.491	BB	0.5862	1.43848e4	332.85953	94.9423

Totals : 1.51511e4 363.72547

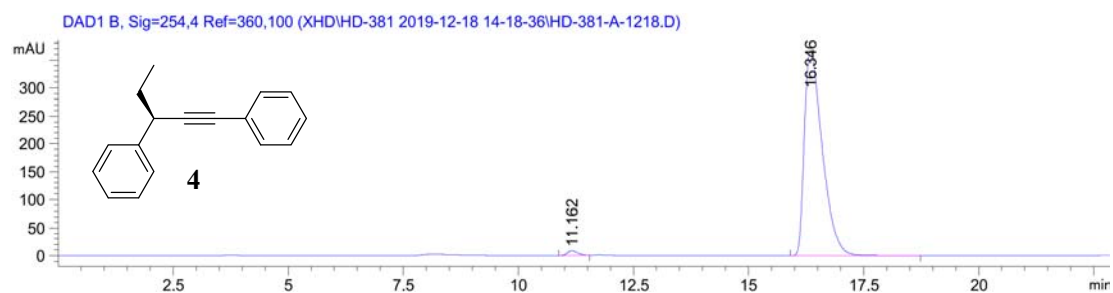
Large-scale synthesis



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.926	BB	0.2603	1.46767e4	863.26874	49.2075
2	15.929	BB	0.4240	1.51495e4	542.19293	50.7925

Totals : 2.98262e4 1405.46167



Signal 2: DAD1 B, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.162	BB	0.2345	124.32475	8.21722	1.2326
2	16.346	BB	0.4145	9962.19727	367.22672	98.7674

Totals : 1.00865e4 375.44394